

INCINERATED ASHES: CHARACTERISTICS AND POTENTIAL FOR USE IN CONCRETE-RELATED APPLICATIONS

by

Ciarán J. Lynn

Supervisors:

Dr. Gurmel S. Ghataora

Prof. Ravindra K. Dhir OBE

A thesis submitted to
the University of Birmingham
for the degree of
DOCTOR OF PHILOSOPHY

School of Engineering
College of Engineering and Physical Sciences
University of Birmingham
September 2017

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ABSTRACT

Municipal solid waste, described as household waste, and sewage sludge, arising from wastewater treatment, are produced in large quantities around the world. Incineration plays a key role in the management of these wastes, though the process leaves behind residual ashes, municipal incinerated bottom ash (MIBA) and sewage sludge ash (SSA), respectively. With increasing importance given to sustainability and aspirations to progress towards a zerowaste society, there is a strong interest in developing uses for these ashes. This study assesses the characteristics of MIBA and SSA, and their use in concrete-related applications. An original analytical systemisation methodology was developed which firstly involved sourcing of the global data, followed by the systematic coordination of these results into a collective form, for the analysis, evaluation and modelling work to be undertaken. After an initial overview of the ash production, the properties of the ashes were studied. On the use of MIBA/SSA, including as both aggregate and cement components, the applications explored included cement clinker production, pastes, mortars, concrete, blocks, lightweight aggregate concrete and aerated concrete. Models were developed for ash-concrete strength estimation. It was determined that MIBA and SSA can offer value in concrete products, though conditions on their use are required.

ACKNOWLEDGEMENTS

My upmost thanks and sincere gratitude goes to my supervisors, Dr. Gurmel S. Ghataora and Prof. Ravindra K. Dhir OBE for their outstanding guidance, support and inspiration throughout my PhD studies at the University of Birmingham.

I would like to gratefully acknowledge the beneficial friendly discussions and constructive feedback received from Dr. Michael Burrow and Prof. Jimmy Yang during the annual progress review meetings. I also thank the University of Birmingham for providing the opportunity for this work to be undertaken.

Special thanks to Chao-Qun Lye and Abdurrahman Elgalhud for their friendly advice, support and considerate nature throughout our shared struggles for these few years. My thanks also go to the many pleasant colleagues that I have crossed paths with at the University of Birmingham.

Finally, to my own family, in particular my Mam and Dad, I am extremely grateful and thankful for your encouragement, selflessness and love, which have supported me during these four years and throughout my whole life. *Go raibh míle maith agat*.

TABLE OF CONTENTS

CHAPTER 1 INTRODUCTION	1
1.1 Background	1
1.2 Scope of the Study	6
1.3 Outline of the Thesis	7
CHAPTER 2 PRODUCTION OF INCINERATED ASHES	9
2.1 Introduction	9
2.2 Generation of Municipal Solid Waste and Sewage Sludge	9
2.3 Composition of Municipal Solid Waste and Sewage Sludge	12
2.4 Management of Municipal Solid Waste and Sewage Sludge	16
2.5 Incineration Process	20
2.6 Post-Incineration Processing	22
2.7 Summary	26
CHAPTER 3 METHODOLOGY	28
3.1 Introduction	28
3.2 Analytical Systemisation	28
3.2.1 Identifying and Sourcing the Data	29
3.2.2 Building the Data Matrix	31
3.2.3 Analysis, Evaluation and Modelling	34
3.3 Peer-Review Feedback and Dissemination	36
3.4 Summary	37
CHAPTER 4 CHARACTERISTICS OF INCINERATED ASHES AND IMPLICATION	NS FOR USE IN
CONCRETE	39
4.1 Introduction	39
4.2 Physical Properties	40

	4.2.1 Particle Size Distribution	40
	4.2.2 Density	43
	4.2.3 Morphology	47
	4.2.4 Water Absorption	48
	4.2.5 Permeability	49
	4.3 Chemical Properties	50
	4.3.1 Oxide Composition	50
	4.3.2 Loss on Ignition	56
	4.3.3 Mineralogy	60
	4.3.4 Element Composition	63
	4.3.5 Organic Compounds	69
	4.4 Summary	72
Cŀ	HAPTER 5 USE OF MUNICIPAL INCINERATED BOTTOM ASH IN CONCRETE	75
	5.1 Introduction	75
	5.2 Use as Aggregate	76
	5.2.1 Mortar and Concrete	76
	5.2.2 Masonry Blocks	96
	5.2.3 Lightweight Aggregate Production	99
	5.2.4 Lightweight Aggregate Concrete	105
	5.3 Use as Cement Component	108
	5.3.1 Raw Feed in Cement Clinker Production	108
	5.3.2 Paste, Mortar and Concrete	111
	5.3.3 Aerated Concrete	122
	5.4 Environmental Assessment	125
	5.5.Case Studies	127

5.6 Summary	130
CHAPTER 6 USE OF SEWAGE SLUDGE ASH IN CONCRETE	133
6.1 Introduction	133
6.2 Use as a Cement Component	134
6.2.1 Raw Feed in Cement Clinker Production	134
6.2.2 Paste, Mortar and Concrete	137
6.2.3 Aerated Concrete	150
6.2.4 Blocks	152
6.3 Use as an Aggregate Component	154
6.3.1 Mortar and Concrete	154
6.3.2 Lightweight Aggregate Production	156
6.3.3 Lightweight Aggregate Concrete	158
6.3.4 Foamed Concrete	161
6.4 Environmental Assessment	162
6.4.1 SSA Leaching	162
6.4.2 Raw Feed in Cement Clinker Production	165
6.4.3 Lightweight Aggregate	166
6.4.4 Mortar and Concrete	166
6.4.5 Blocks	168
6.5 Case Studies	169
6.6 Summary	170
CHAPTER 7 MODELLING OF CONCRETE STRENGTH WITH INCINERATED ASHES	174
7.1 Introduction	174
7.2 Current Status of Modelling Work on Concrete using Incinerated Ashes	175
7.3 Strength Models for Normal Concrete Mixtures	177

7.4 Development of Strength Models with Incinerated Ashes	182
7.5 Cement Model	188
7.6 Aggregate Model	200
7.7 Model Application	210
7.8 Summary	214
CHAPTER 8 CONCLUSIONS AND RECOMMENDATIONS	217
8.1 Concluding Remarks	217
8.2 Recommendations for Further Work	220
REFERENCES	222

LIST OF FIGURES

Figure 1.1: Flow chart of the waste management process based on EU legislation2
Figure 2.1: Stages of the wastewater treatment process
Figure 2.2: Potential treatments of sewage sludge before utilization or disposal11
Figure 2.3: Management practices in recent years for (a) MSW (data from Eurostat, 2017c)
and (b) sewage sludge (data from Eurostat, 2017d)17
Figure 2.4: Main stages of the incineration process and the output residues produced20
Figure 3.1: Sample of the initial sorting process32
Figure 3.2: Sample of the data mining process on the particle size distribution of SSA, as part
of the data matrix formation33
Figure 3.3: Sample of the analysis and evaluation work on the effect of MIBA as aggregate on
the relationship between compression strength and elastic modulus of concrete35
Figure 4.1: Particle size distribution curves of unprocessed/screened, sieved and ground (a)
MIBA and (b) SSA samples41
Figure 4.2: Density of (a) MIBA and (b) SSA subjected to varying levels of processing44
Figure 4.3: Relationship between the density of SSA and the incineration temperature46
Figure 4.4: Permeability results for MIBA and SSA samples50
Figure 4.5: Box and whisker plots of the oxide contents of (a) MIBA and (b) SSA sample51
Figure 4.6: Ternary diagram of the contents of the main oxides SiO ₂ , Al ₂ O ₃ and CaO found in
(a) MIBA and (b) SSA samples53
Figure 4.7: Loss on ignition results, arranged in ascending order for MIBA and SSA samples 57
Figure 5.1: Effect of MIBA on concrete consistence (workability) as (a) fine aggregate and (b)
coarse aggregate replacement78
Figure 5.2: Effects of MIBA as aggregate on other fresh properties of mortar/concrete80
Figure 5.3: Effects of standard processed MIBA on 28-day compressive strength as fine
aggregate in mortar/concrete81
Figure 5.4: Compressive strength of mortar and concrete mixes using further processed MIBA
as (a) fine aggregate and (b) coarse aggregate86
Figure 5.5: Effects of MIBA as aggregate in mortar/concrete on the relationship between
compressive strength and (a) flexural strength and (b) tensile strength88

Figure 5.6: Effects of MIBA as aggregate in concrete on (a) the elastic-modulus-compressive
strength ratio and (b) effect of aggregate replacement level on elastic modulus90
Figure 5.7: Ranking of the effects of MIBA as aggregate on concrete durability93
Figure 5.8: Particle density results for (a) MIBA lightweight aggregates and (b) the effect of
sintering temperature102
Figure 5.9: Water absorption results for MIBA lightweight aggregates103
Figure 5.10: Consistence of lightweight MIBA aggregate concrete106
Figure 5.11: Compressive strengths (28 days) for lightweight MIBA aggregate concrete107
Figure 5.12: Effects of MIBA on the setting of pastes, mortar and concrete mixes114
Figure 5.13: Effects on the strength development of cement pastes using (a) standard
processed MIBA and (b) further chemically or thermally treated MIBA115
Figure 5.14: Compressive strength of mortars using MIBA subjected to (a) standard processing
and (b) thermal treatment
Figure 5.15: Compressive strength of concrete using MIBA subjected to (a) standard
processing and (b) additional thermal treatment
Figure 6.1: Effects of SSA as a cement component on (a) mortar flow and (b) concrete slump
Figure 6.2: Consistence of mortars containing a combination of SSA and coal fly ash as cement
components, with respect to the control Portland cement mix
Figure 6.3: Effects of SSA as a cement component on the setting behaviour of pastes, mortar
and concrete mixes
Figure 6.4: Strength activity index (SAI) results with SSA as a cement component141
Figure 6.5: Combined data on the effect of SSA content on mortar/concrete strength 143
Figure 6.6: Comparison of the relationship between compressive and flexural strength for
mixes with SSA and the control Portland cement mixes146
Figure 6.7: Compressive strength performance of aerated concrete mixes with SSA151
Figure 6.8: Specific bulk density of SSA lightweight aggregates157
Figure 6.9: Effect of SSA as a fine aggregate replacement on foamed concrete properties. 161
Figure 7.1: Leverage values for the cement model building data195
Figure 7.2: Cook's distance data for the cement model building data 195

Figure 7.3: Standardised residuals for the cement model building data196
Figure 7.4: Assessment of the raw residuals for the cement model198
Figure 7.5: Predicted strength vs measured strength for cement model (a) building data and
(b) validation data199
Figure 7.6: Leverage values for the aggregate model building data206
Figure 7.7: Cook's distance values for the aggregate model building data206
Figure 7.8: Standardised residuals for the aggregate model building data207
Figure 7.9: Raw residuals for the aggregate model building data208
Figure 7.10: Predicted strength vs measured strength for aggregate model (a) building data
and (b) validation data209
Figure 7.11: Compressive strength vs w/c for concrete using incinerated ash as (a) cement and
(b) aggregate replacements

LIST OF TABLES

Table 1.1: Production and description of waste materials subjected to thermal treatments4
Table 2.1: MSW composition for selected countries (Zhang et al., 2010)13
Table 2.2: Basic physical and chemical characteristics of sewage sludge14
Table 2.3: Data on the heavy metal contents of sewage sludge16
Table 3.1: Metadata for publications on MIBA/SSA and their use in concrete30
Table 4.1: Data on the fineness of SSA (adapted from Lynn et al., 2015)43
Table 4.2: Accompanying loss on ignition data analysis for MIBA and SSA samples58
Table 4.3: Additional minerals identified in MIBA and SSA61
Table 4.4: Description of the information on the glass contents of MIBA and SSA62
Table 4.5: Analysis of the element composition of MIBA and SSA samples64
Table 4.6: Element variability of Italian MIBA samples (previous data for worldwide MIBA
samples given in parentheses for comparison)67
Table 4.7: PCDD/F concentration limits outlined in the literature70
Table 4.8: PCDD/F contents in (a) MIBA and (b) SSA71
Table 5.1: Consistence results for mortars using MIBA as a fine aggregate replacement77
Table 5.2: Effects of further processing on the compressive strength of mortar and concrete
mixes containing MIBA as fine or coarse aggregate components84
Table 5.3: Effects of MIBA as fine/coarse aggregate on the concrete drying shrinkage91
Table 5.4: Work undertaken on the use of MIBA as aggregate in concrete blocks96
Table 5.5: Lightweight aggregate mix series produced using MIBA100
Table 5.6: Chemical composition of cement clinker mixes that used MIBA in the raw feed 110
Table 5.7: Effects of MIBA as a cement component on mortar/concrete consistence113
Table 5.8: Effects of MIBA on the durability of paste, mortar and concrete mixes121
Table 5.9: Mix designs and density and strength results using MIBA in aerated concrete124
Table 5.10: Test methods, mix designs and leaching results for pastes, mortars and concrete
127
Table 6.1: Constituent contents of blends using SSA in cement clinker production134
Table 6.2: Chemical composition of cement clinkers produced using SSA in the raw feed136
Table 6.3: Effect of SSA on 28 day mortar/concrete compressive strength143

Table 6.4: Effects of SSA on mortar/concrete strength development with age	144
Table 6.5: Results on the shrinkage of mortar and concrete mixes containing SSA	147
Table 6.6: Effect of SSA on the durability of mortar and concrete mixes	148
Table 6.7: Performance of concrete blocks incorporating SSA as a cement component	153
Table 6.8: Results on the performance of mortar/concrete with SSA as aggregate	155
Table 6.9: Properties of concrete containing SSA lightweight aggregate	159
Table 6.10: Leaching behaviour of SSA using the TCLP	163
Table 6.11: Leaching behaviour of SSA using the EN 12457 (2002) method	164
Table 6.12: Leaching results (TCLP) of cement clinkers incorporation SSA in the raw feed	165
Table 6.13: Leaching results for concrete using SSA as a partial cement component	167
Table 6.14: Case studies undertaken using SSA in concrete-related applications	170
Table 7.1: Modelling work undertaken with SSA/MIBA in concrete-related applications	176
Table 7.2: Selection of machine learning modelling work on concrete strength	182
Table 7.3: Summary of initial data for the incinerated ash aggregate and cement models	185
Table 7.4: Coefficient estimates, standard error and p-values for six iterations with the cen	nent
model	192
Table 7.5: RMSE and R ² values for cement model and validation data RMSE	192
Table 7.6: Potential outlier data points in the ash-concrete cement model	197
Table 7.7: Coefficient estimates, standard error and p-values for six iterations with	the
aggregate model	203
Table 7.8: RMSE and R^2 values for the aggregate model and validation data RMSE	204
Table 7.9: Potential outlier data points in the ash-concrete aggregate model	207
Table 7.10: Input parameter ranges for the incinerated ash models source data	211
Table 7.11: Typical ash properties used for the strength design demonstration	212

LIST OF ABBREVIATIONS AND DEFINITIONS

A, A₁₋₇, B, m Model Coefficients

a/b Aluminium powder/binder ratio

agg Aggregate

ANOVA Analysis of Variance
APC Air pollution control

AR Alumina ratio

Ave Average

BET Brunauer-Emmett-Teller c/b Cement/binder ratio

C_{10mins} Capillary water absorption coefficient after a time period of 10mins

C_{final} Capillary water absorption coefficient at the test end time

CFBC Circulating fluidised bed combustion

CFCs Chlorofluorocarbons

Char Characteristic
CKD Cement kiln dust

Coef Coefficient
Comp Compressive

CV Coefficient of variation

D Mean aggregate diameter

d.l. Detection limit

 D_{10} 10% of the sample's mass is comprised of particles < this diameter D_{30} 30% of the sample's mass is comprised of particles < this diameter D_{50} 50% of the sample's mass is comprised of particles < this diameter D_{60} 60% of the sample's mass is comprised of particles < this diameter D_{90} 90% of the sample's mass is comprised of particles < this diameter

D_{max} Maximum aggregate size

E Activation energy
EC European Community

EIA Environmental Impact Assessment
EPA Environmental Protection Agency

F Blaine fineness
FA Coal fly ash

f_c Ash mix strength (28 days) in MPa, f_c' Control mix strength (28 days) in MPa

F-CaO Free CaO

F_{cuk} Target concrete cube strength at 28 days

F_{High} Ash classified as high fineness

GGBS Ground granulated blastfurnace slag

Gwell Ash categorised as well graded

HM Hydraulic modulus

I_{corr} Instantaneous corrosion rate

I_{DAL} Ash with an inhibiting factor (low density/high absorption/high LOI)

I-TEQ International toxic equivalency
IWSA Industrial wastewater sludge ash

K Paste to aggregate bond strength constant

K_{eff} Efficiency factor L/S Liquid/solid ratio

Laga Länderarbeitsgemeinschaft Abfall Leca Lightweight expanded clay aggregate

LOI Loss on ignition
LS Limestone

LW Lightweight

LWA Lightweight Aggregate

MIBA Municipal incinerated bottom ash MIFA Municipal incinerated fly ash

MK Metakaolin

MPT Maximum paste thickness

MR Metal removal

MSW Municipal solid waste n Number of samples

nd Not detected
NP Natural pozzolan

OECD Organisation for Economic Co-operation and Development

PAHs Polycyclic aromatic hydrocarbons

PC Portland cement

 p_{C3A} Weight ratio of C_3A per total cement p_{C3S} Weight ratio of C_3S per total cement p_{FA} Weight ratio of fly ash per total cement

p_{FA-CaO} Weight ratio of fly ash-CaO per total cement

p_{SLAG} Weight ratio of slag per total cement p_{SO3} Weight ratio of SO₃ per total cement

PCBs Polychlorinated biphenyls

PCDDs Polychlorinated dibenzo-p-dioxins
PCDFs Polychlorinated dibenzofurans

Pen Penetration

P_{Fur} Further processed ash (washed, chemically treated, thermally treated)

Proc Proceedings

R Universal gas constant

r Cement replacement level (as a fraction)

R_{c28} Standard cement 28 day strength

RH Relative humidity

RMSE Root mean square error

ROC Republic of China

RSM Recycled and secondary materials

S/S Solidification/stabilisation
SAI Strength activity index

SEA Strategic Environmental Assessment

SF Silica fume
Sg Specific gravity
SP Superplasticiser

SPC Sulphate resistant cement

SR Silica ratio
SS Sewage sludge
SSA Sewage sludge ash
SSE Sum of squared errors

SSR Regression sum of squares

SST Total sum of squares
St Dev Standard deviation
T Concrete temperature

t Age in days

TCDD Tetrachlorodibenzodioxins

TCLP Toxicity characteristic leaching procedure

t_e Equivalent ageUN United Nations

UNFCCC United Nations Framework Convention on Climate Change

 $\begin{array}{ll} \text{Unspec.} & \text{Unspecified} \\ \text{UW}_t & \text{Unwashed} \\ \text{V}_a & \text{Volume of air} \end{array}$

 V_c Volume of cement V_w Volume of water

W_t Washed

w/b Water/binder ratio w/c Water/cement

w/c' Water/cement ratio of the control mix

W_{Control} Ash mixes for which the water content is controlled

WHO World Health Organisation
WPSA Water purification sludge ash

Wt Weight

X Gel space ratio

α Degree of cement hydration

 α_{cr} Critical degree of cement hydration α_u Ultimate degree of cement hydration

β Hydration shape parameter

 $\lambda_{10,dry}$ (p= 50%) Thermal conductivity in dry blocks, 50th percentile $\lambda_{10,dry}$ (p= 90%) Thermal conductivity in dry blocks, 90th percentile

τ Hydration time parameter

 $\phi \qquad \qquad \text{Volumetric fraction of aggregate} \\ \phi^* \qquad \qquad \text{Maximum aggregate packing density}$

LIST OF PUBLICATIONS

1. Directly Related to the Thesis

- Lynn C J, Dhir R K, Ghataora G S, and West R P. (2015) Sewage sludge ash characteristics and potential for use in concrete. *Construction and Building Materials*, 98, 767-779.
 DOI: 10.1016/j.conbuildmat.2015.08.122.
- Lynn C J, Dhir R K and Ghataora G S. (2016c) Municipal incinerated bottom ash characteristics and potential for use as aggregate in concrete. *Construction and Building Materials*, 127, 504-517. DOI: 10.1016/j.conbuildmat.2016.09.132.
- Lynn C J, Dhir R K and Ghataora G S. (2017b) Municipal incinerated bottom ash use as a cement component in concrete. *Magazine of Concrete Research*, 69(10), 512-525.
 DOI: 10.1680/jmacr.16.00432.

2. Related to Other Uses of the Incinerated Ashes

- Lynn C J, Ghataora G S and Dhir R K. (2016a) Environmental Impacts of MIBA in geotechnics and road applications. *Institution of Civil Engineers (ICE) Environmental Geotechnics*. DOI: http://dx.doi.org/10.1680/jenge.15.00029.
- Lynn C J, Dhir R K and Ghataora G S. (2016b) Sewage sludge ash characteristics and potential for use in bricks, tiles and glass ceramics. Water Science & Technology, 74(1), 17-29. DOI: 10.2166/wst.2016.040.
- Lynn C J, Ghataora G S and Dhir R K. (2017a) Municipal Incinerated Bottom Ash (MIBA)
 Characteristics and Potential for Use in Road Pavements. *International Journal of Pavement Research and Technology*, 10(2), 185-201. DOI: 10.1016/j.ijprt.2016.12.003.

CHAPTER 1

INTRODUCTION

1.1 Background

The concept of sustainability and the challenge of protecting the environment in the midst of prosperous economic development was initially discussed 45 years ago, at the 1972 United Nations Conference on the Human Environment in Stockholm. This issue gained further recognition with the introduction of the term 'sustainable development' in the 1987 United Nations' World Commission on Environment and the developed Brundtland Report (United Nations, 1987) and indeed has been incorporated as a core principle of subsequent United Nations Conferences. The United Nations Conference on Environment and Development held in Rio de Janeiro in 1992 was another major milestone that introduced an action plan to promote sustainable development and opened for signature the United Nations Framework Convention on Climate Change (UNFCCC) treaty. Annual UN conferences have subsequently been held for member states to discuss the progress of managing climate change. Alongside the UNFCCC framework, specific treaties have been adopted, such as the Kyoto Protocol in 1997, setting out legal requirements for states to reduce greenhouse gas emissions and improve efficiency, conserve natural resources and promote renewable energy sources. The most recent conference, held at the end of 2015, yielded the Paris Climate Change Agreement, At the time of writing this thesis, this agreement had been signed by 174 independent nations, and aims to limit the temperature increases associated with global warming, which, in lay terms, makes the sustainability objectives easier to appreciate and, it is hoped, more effective in achieving the desired end results.

Transitioning from broader sustainability goals to the topic of waste management, which is of relevance for this project, it is clear that management of waste is a problem common to all countries. The conversion of past waste treatment practices towards more environmentally friendly options continues to be a key objective for moving towards a more sustainable society. In Europe, waste management is directed by Waste Directive 2008/98/EC (European Community, 2008), in which Article 4 outlines the desired order of treatment options. An outline of the legislation relating to this waste management process is presented in Figure 1.1.

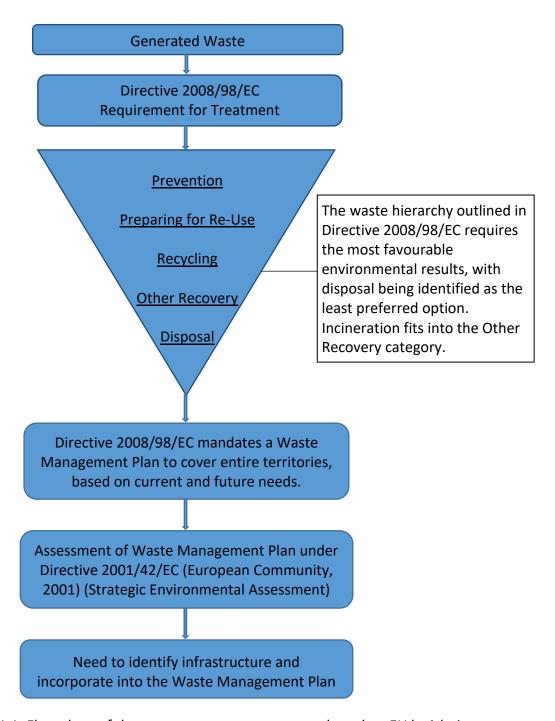


Figure 1.1: Flow chart of the waste management process based on EU legislation

The first option of prevention seeks to reduce waste at the source, which can done by encouraging manufacturers to produce more environmentally friendly products, increasing the life cycle duration of resources and enticing households to curtail the quantities they put out for collection. Next in the waste hierarchy is preparing for reuse, which can involve

cleaning, repairing or refurbishing to give the material another use to avoid it becoming 'waste'. Recycling involves processing of the material to be exploited in another use. Incineration falls into the penultimate 'other recovery' section, whereby energy can be recovered in the form of electricity generation or space heating from waste combustion, whilst valuable metals may also be recoverable from the residues. However, as is the focus of this work, the incinerated ashes generated may also be developed as valuable construction materials.

Disposal, quite rightly, is the least favourable option. Measures have been introduced to discourage this treatment, with ever-reducing targets set for the allowable landfilled biodegradable fraction (European Community, 1999), whilst the disposal of sludge at sea, which has been common in the past, has been banned.

To support the objectives and provisions of the Waste Directive 2008/98/EC (European Community, 2008), including compliance with the waste hierarchy, requirement are also outlined for Plans and Programmes to analyse the current waste management systems and set out the waste management strategies and infrastructure required. There may also be the need for a Strategic Environmental Assessment (SEA) and Environmental Impact Assessment (EIA) under Directive 2001/42/EC (European Community, 2001) to be included. The impact of this legislation and policies has been to shift waste treatment away from landfilling and lead to an increase in its recycling and incineration.

Focusing on incineration, this treatment utilises the waste as a fuel for energy recovery, though this is not a complete treatment as residual ashes are left behind. The combustion treatment is also, in some cases, used solely to reduce the size of the material to manage, without energy recovery. A variety of materials are subjected to this thermal treatment, including municipal solid waste, sewage sludge, wood waste, rice husk, medical waste and other hazardous wastes. Reported data on the materials' production and a brief description of each are presented in Table 1.1.

Table 1.1: Production and description of waste materials subjected to thermal treatments

MATERIAL	PRODUCTION	DESCRIPTION
Municipal Solid Waste	242 MT in EU, 663 MT in OECD, 1840 MT worldwide per annum	Consists of mainly organics, paper/cardboard, plastics, glass and metals.
Sewage Sludge	5 MT in EU	From primary, secondary, and tertiary treatment of sewage wastewater treatment. High organics & volatile matter, low solids.
Wood Waste	98.2 x 10 ⁷ m ³ generated worldwide per year, equating to approximately 200 MT.	Variable composition depending on wood source, grade, growing environment. Has a high carbon content.
Medical Waste	Waste generation (kg/bed-day): USA 11, Jordan 6, Spain 4, Norway 4, UK 3, France 3, Brazil 3, Taiwan 3, Pakistan 2, Turkey 1, Tanzania 0.1.	85% general waste (paper, cardboard, plastics). 15% hazardous (sharps, infectious, pathological, chemical, radioactive)
Rice Husk	97 MT of rice husk per year (20% of 484 MT of rice paddy production)	High organics, carbon and ash contents. Mostly used as fuel in power plants
Other Hazardous Waste	22 MT produced per year in EU-15 countries. 12% incineration rate.	A variety of wastes from industries and merchant plants.

Data from: Eurostat (2017a); Eurostat (2017b); European Commission (2006); FAO, cited in James et al. (2012); Windfeld and Brooks (2015); WHO (2015); OECD (2017); Statista (2017) and Waste Atlas (2013).

Of the listed materials, the hazardous and medical wastes, because of their chemical characteristics, pose the highest risks in terms of harm to health and the environment. Indeed, for these reasons, the incineration of medical waste is on the decline in developed countries (United Nations Development Programme and Global Environment Facility, 2016). On the other end of the scale, rice husk and wood waste contain less environmentally harmful constituents. The ashes produced from the thermal treatment of these materials have shown good potential for use in concrete, though their fineness indicates that they would be used as cementitious components. The production of rice and management of the resultant husk ash, though very large, are also mainly limited to Asia, which accounts for around 90% of the global rice generation.

Sewage sludge and, in particular, municipal solid waste are two major waste streams that are produced all around the world and are incinerated in large quantities. The nature of these

materials and the presence of heavy metals raise some questions around their use and the associated environmental impacts. However, there has been significant interest and subsequent research carried out on these ashes and indeed they appear to show good potential for development as construction materials. Municipal incinerated bottom ash (MIBA) and sewage sludge ash (SSA) were selected as the focus of this study.

The construction industry is expected to play a major role in the progression towards zero waste. Countries such as Belgium, Denmark, Germany and The Netherlands have been amongst the leaders in taking advantage of the ashes as a valuable resource, using 100%, 98%, 86% and 80% of the MIBA generated per annum, respectively, as construction materials (An et al., 2014; Qing and Yu, 2013). This has largely been as fill material and in road pavement layers. However, in the UK, past practice with MIBA has included its use as aggregate in concrete blocks.

The use of MIBA and SSA in concrete related applications appears to be a promising outlet. Concrete is known to be one of the most widely used construction materials, though it has a high carbon footprint due to the emissions released during cement manufacture as a result of the high energy requirements and the conversion of the calcium carbonate in limestone to calcium oxide and carbon dioxide. Indeed, cement manufacture has been reported to contribute 8% of the global CO₂ emissions (Olivier et al., 2015). Concrete also carries a high demand for natural materials, of which there is a finite supply. As such, in recent times, with the growing importance given to sustainability, strong efforts are being made to reduce CO₂ emissions and conserve natural resources through the incorporation of secondary and recycled materials as aggregate or cementitious components, including coal fly ash (FA), ground granulated blastfurnace slag (GGBS) and construction and demolition waste (for example Lye et al., 2015; Lye et al., 2016; Pacheco-Torgal et al., 2013).

Despite the significant experimental research undertaken on the use of MIBA and SSA in concrete-related applications, the practical application of the materials in these products has not yet developed to the same extent as their use in road construction. The information in its

current state is fragmented, coming from all parts of the world and presented in various forms. However, with this sourced literature acting essentially as a global laboratory source, the abundance of experimental data obtained provides a great opportunity for robust and critical analysis, evaluation and modelling of the collective work, well beyond what is possible for the individual source authors. This innovative approach, in determining the findings emerging from decades of experimental work, can be very useful and timely, given the potential for growth in the use of incinerated ashes in concrete.

As is examined in the succeeding chapters, the characteristics of these incinerated ashes suggest that they can be potentially used as valuable components of both aggregates and cement. Concrete offers a higher value use of MIBA and SSA, though it typically has more onerous material requirements compared to geotechnic and road pavement applications. As such, this outlet, though promising, cannot be seen as just a dumping ground and it must be shown that the materials can deliver suitable performance and be incorporated within the concrete mix design procedures.

1.2 Scope of the Study

The main aim of this work has been to:

 Study the characteristics of incinerated ashes, MIBA and SSA, and their potential for sustainable use in concrete-related applications, through the analysis and evaluation of the globally published experimental data and the development of statistical models for the prediction of concrete strength performance with these materials.

To achieve this aim, the following objectives were set:

- Source the globally published research on MIBA and SSA and their use as construction materials.
- Analyse and evaluate the mined experimental results to robustly determine the characteristics of the ashes and the findings on their use in construction applications, with a focus on the critical assessment of performance in concrete.

- Undertake statistical modelling of the data, specifically focusing on the strength performance of MIBA and SSA in concrete.
- Disseminate the work to industry, practitioners and researchers, in reputable peerreviewed journals continually during the course of the PhD studies. This also allowed useful feedback to be received from esteemed colleagues, as part of the peer-review process.

As part of the full study, the analysis and evaluation covered the use of MIBA and SSA in a variety of construction applications including geotechnics, road pavements, ceramics and concrete. The focus subsequently honed in specifically on concrete applications, which were identified as an outlet with great potential for developing the use of the incinerated ashes. As such, the work presented in this final thesis is limited to the material characteristics and their use in concrete, though the remaining work in the other areas has still been published in multiple reputable peer-reviewed journals: *Environmental Geotechnics* (Lynn et al., 2016a), *Water Science & Technology* (Lynn et al., 2016b) and the *International Journal of Pavement Research and Technology* (Lynn et al., 2017a).

The overall approach adopted in this study is very different to the norm, which typically involves a literature review and some form of experimental work in the laboratory. The uniqueness and innovation in this study lies in the process of analysis, evaluation and modelling of the experimental data published globally. The world literature, when coordinated, provided an enormously strong and wide-ranging pool of experimental results, compared to what could be achieved by an individual researcher. The focus was then on critically assessing this vast resource of knowledge to determine the important findings regarding the use of incinerated ashes in concrete.

1.3 Outline of the Thesis

Chapter 2 provides the background information on the production of incinerated ashes, starting with the original municipal solid waste and sewage sludge materials and their treatment, with a specific focus on the incineration process. The subsequent processing and management of the incinerated ashes are also covered.

Chapter 3 details the methodology developed for sourcing the published work, mining the data and building the data matrix, undertaking analysis and evaluation of the collective experimental results and modelling the concrete strength performance.

Chapter 4 presents the analysis and evaluation of the characteristics of MIBA and SSA, dealing with the physical and chemical properties, and their potential suitability for future use in concrete applications.

Chapters 5 and 6 deal with the analysis and evaluation of global data on the use of MIBA and SSA, respectively, in a range of concrete related applications including cement clinker production, cement pastes, mortar, concrete, masonry blocks, lightweight aggregate production, aerated concrete and foamed concrete.

Chapter 7 covers the development of statistical models to estimate the compressive strength performance with MIBA and SSA as both aggregate and cement components in concrete applications.

Chapter 8 presents the salient conclusions emerging from this study and offers recommendations that can be beneficial in further progressing the sustainable use of incinerated ashes in concrete.

CHAPTER 2

PRODUCTION OF INCINERATED ASHES

2.1 Introduction

Prior to the analysis and evaluation work on the characteristics of MIBA and SSA and their use in concrete, an overview of the information on the production of the materials is presented as a background chapter. Although, this area is not the main focus of this project, the production and treatment of the source municipal solid waste (MSW) and sewage sludge (SS) have a determining effect on the properties of the resultant MIBA and SSA. It is also important to have an awareness of the variations in waste management policies that are implemented worldwide, how these may change in the future and the potential implications for the use of these incinerated ashes as construction materials.

This chapter begins with a description of the production of MSW and SS, followed by an appraisal of the composition of the materials. The management practices adopted for MSW and SS in recent years are also covered. The next section deals specifically with the process of incineration. Post-incineration processing treatments of MIBA and SSA are also dealt with.

2.2 Generation of Municipal Solid Waste and Sewage Sludge

Municipal incinerated bottom ash and sewage sludge ash are the main residues produced from the incineration of MSW and SS. Municipal waste is defined in the Landfill Directive (European Community, 1999) as 'waste from households, as well as other waste which, because of its nature or composition, is similar to waste from households' and consists mainly of organic matter, paper, cardboard, plastics, glass and metals. Management of this material is a challenge common to every country in the world, though waste collection rates tends to be more limited in lower income countries, falling below 50%, with open dumping still common (World Bank, 2012).

Sewage sludge is generated during the wastewater treatment process as the collected wastewater undergoes a series of treatments to remove harmful contaminants before it is discharged back into the water cycle. The typical stages involved in the wastewater treatment

process are outlined in Figure 2.1, though the specific nature of treatment varies from plant to plant, depending on the composition of the incoming sludge, the environmental sensitivity at the discharge location and the governing regulations.

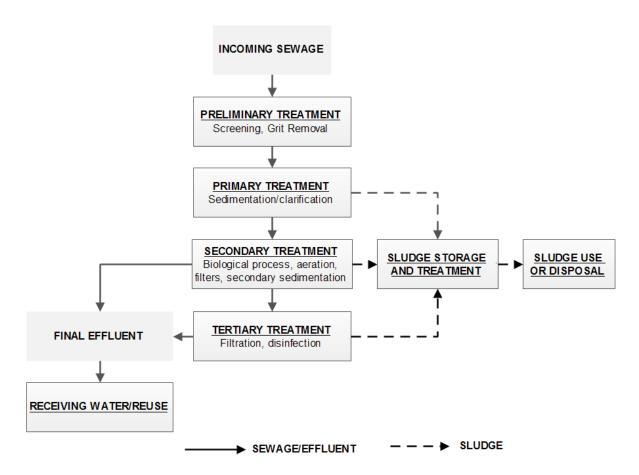


Figure 2.1: Stages of the wastewater treatment process

The preliminary treatment stage involves screening to take away large components and grit removal using a channel or chamber. During primary treatment, sludge particles settle to the bottom in sedimentation tanks/clarifiers and are subsequently removed, whilst the scum surface layer is also skimmed off. The secondary stage consists of a biological treatment using micro-organisms under oxidised conditions to further remove the organics and suspended solids and may also include secondary sedimentation. Tertiary treatment may also be included with more onerous wastewater treatment requirements and can consist of additional filtration, disinfection and nitrogen and phosphorus removal.

As outlined in Figure 2.1, after the sludge has been removed during wastewater treatment, the material is stored and requires additional processing to improve its suitability for future transportation, further treatments such as incineration, or its end use. The common processing treatments adopted with sewage sludge are shown in Figure 2.2, though typically only a selection from the full list is used, depending on the end use of the material.

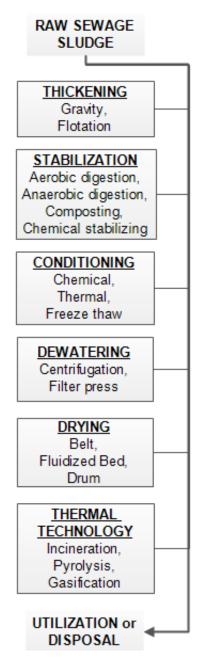


Figure 2.2: Potential treatments of sewage sludge before utilization or disposal

Thickening is commonly one of the first treatments adopted and it results in a reduction in the sludge water content and therefore the total volume of material. The solids contents can be increased to around 10% with this treatment.

Dewatering and drying treatments, using mechanical (centrifugation and filter press) and thermal (belt drying, fluidised bed drying and drum drying) techniques respectively, are also used to decrease the moisture content and resultant volume of the sewage sludge.

In addition, conditioning may be implemented beforehand to improve the effectiveness of dewatering through charge neutralisation and flocculation of the sewage sludge particles to promote water depletion.

Stabilisation aims to reduce organic matter and harmful micro-organisms, using biological (aerobic digestion, anaerobic digestion and composting) and chemical treatments (lime and chlorine stabilisation).

Thermal treatments, including incineration and other technologies such as gasification and pyrolysis, are used to reduce the volume of the sewage sludge and also allow for energy to be recovered.

2.3 Composition of Municipal Solid Waste and Sewage Sludge

In addition to the main constituents of organic matter, paper and cardboard, plastics, glass and metals, smaller quantities of textiles, hazardous waste, electrical and electronic goods, soil, ceramics, wood, bamboo, straw, coconut shells, sanitary products, rubber, garden wastes and slag have also been found in MSW (Chang et al., 2009; Chattopadhyay et al., 2009; Jung et al., 2006; Lo and Liao, 2007; Shen et al., 2010; Yin et al., 2007; Zhang et al., 2010).

The specific composition of MSW is influenced by many factors including economic and cultural issues, as well as waste management policies. Recordings of the average MSW composition in a selection of countries (Zhang et al., 2010) are presented in Table 2.1. The

data suggest that higher income countries tend to contain higher percentages of paper and cardboard and lower organics. The content of metals in MSW, which can have a significant impact on its potential environmental suitability as a construction material, also appears to be higher in higher income countries. It should be noted that the 'Others' heading in Table 2.1 consisted of components such as ash, textiles, wood and construction debris.

Table 2.1: MSW composition for selected countries (Zhang et al., 2010)

COUNTRY YEAR		COMPOSITION OF MSW, %					
COUNTRY	TEAK	Organic Garbage	Paper/Cardboard	Plastics	Glass	Metals	Others
UK	2000	40.0	18.0	8.0	7.0	8.0	19.0
France	2005	32.0	20.0	9.0	10.0	3.0	26.0
Germany	2005	14.0	34.0	22.0	12.0	5.0	12.0
USA	2005	25.0	34.0	12.0	5.0	8.0	16.0
Japan	2000	34.0	33.0	13.0	5.0	3.0	12.0
Mexico	2005	51.0	15.0	6.0	6.0	3.0	18.0
China	2003	52.6	6.9	7.3	1.6	0.6	30.8

Significant variance has also been evident when examining the composition of the collected MSW within individual countries. Indeed, when the composition of MSW samples collected from 12 cities across a vast country like China was recorded, the organics content were found to range from 16% to 68% (Liu et al., 2006). The contents of the specific components of MSW can also change over time. Results of MSW composition in Germany from 1990 to 2005, for example, indicate trends of reducing organic material, glass and paper/cardboard and increasing plastics and other components, whereas the metals contents remained consistent (OECD, 2009). It is likely that the change over from glass to plastics for beverage containers made a significant contribution to the proportions of these components, whilst increasing emphasis on recycling may have contributed to the decreasing paper/cardboard content.

This changeable composition means that robust quality control systems are needed to regulate the material, as it is being fed into the incinerator and potentially, when the resultant bottom ash is to be used in construction. Reductions in the material variability through waste sorting procedures, along with regulations on the composition of material that the

incinerators are licensed to treat, can be beneficial in improving the prospects of MIBA as a reliable construction material.

The composition of SS is dependent on factors such as the population's density and habits, the contributions from industrial sources and the specific treatments that the wastewater and the sludge residue have undergone. An analysis of the characteristics of SS is presented in Table 2.2, based on samples generated around the world.

Table 2.2: Basic physical and chemical characteristics of sewage sludge

RANGE	MEAN
4.3-8.5	7.0
0.3-12.0	3.9
10-66	54.1
0.3-9.4	6.4
5-56	29.1
17-56	37.4
11000-20096	15474
20-95	58.0
0.8-9.2	3.5
0.4-11.0	2.0
0.3-1.1	0.4
	4.3-8.5 0.3-12.0 10-66 0.3-9.4 5-56 17-56 11000-20096 20-95 0.8-9.2 0.4-11.0

Data from: Ates and Pehlivan (2014), Bianchini et al. (2015), Bonfiglioli et al. (2015), Botha et al. (2011), Carbonell et al. (2009), Cui et al. (2006), Escudey et al. (2007), Fuentes et al. (2004), Fytianos et al. (1998), Galvez et al. (2007), Green and Tibbett (2001), Hospido et al. (2005), Houdkova et al. (2008), Houillon and Jolliet (2005), Hu et al. (2012a), Husillos-Rodriguez et al. (2013), Jackobsen and Willett (1986), Kikuchi (1998), Lederer and Rechberger (2010), Liew et al. (2004), Lim et al. (2006), Lin et al. (2006a), Luan et al. (2013), Malliou et al. (2007), Mingwei et al. (2011), Mun (2007), Niu et al. (2015), Page (1974), Paramasivam et al. (2005), Petersen (2001), Sajwan et al. (2003), Scott (1980), Smith (2007), Sort and Alcaniz (1996), Tay and Show (1992b), Tirado-Montiel et al. (2003), Triner et al. (2001), Tuan et al. (2013), Valls and Vazquez (2001), Wang et al. (2009), Weng et al. (2003), Xu et al. (2008) and Zabaniotou and Theofilou (2008).

As is revealed by Table 2.2, SS contains high amounts of organics and volatiles, along with a very low solids content. The material has a significant ash fraction and high loss on ignition, which, combined with the previous properties, is generally not conducive to producing a

robust construction material. However, the pH of the SS, combined with its nitrogen, phosphorus and potassium contents indicates that the material has strong potential for use as a fertiliser on agricultural lands. Indeed, this has been one of the most common uses for SS in the past, though a more cautious approach is being adopted in recent times because of health concerns relating to the build-up of heavy metals.

An assessment of the oxides composition revealed SiO_2 (average 31.7%), Al_2O_3 (13.8%), P_2O_5 (9.4%), Fe_2O_3 (6.4%) and CaO (4.3%) as the main constituents present in SS. These silica, calcium, alumina and iron oxides suggest that the sludge can be a suitable material to contribute to the formation of the main clinker compounds in cement manufacture, whilst its calorific value can also reduce the fuel requirements.

The potential use of SS and the residues arising from its incineration is acutely dependent on avoiding any damaging environmental impacts associated with the release of harmful constituents into the surrounding environment. Data on the heavy metals contents for SS samples have been compiled and are presented in Table 2.3. Accompanying heavy metal limits for fertiliser use in Germany (German Fertiliser Ordinances from Kruger and Adam, 2015), Austria (Austrian Fertiliser Ordinances from Adam et al., 2007), Switzerland (Swiss Fertiliser Regulations from Franz, 2008) and The Netherlands (Decree of the Ministry of Agriculture and Forestry on Fertiliser Products 24/11 from Havukainen et al., 2012) are also provided to give context to the values.

It is evident that the average contents of many of the more toxic heavy metals exceeded the outlined limits; however, it should be noted that only a fraction of the total metal contents would be in the form in which they could leach out. The leaching behaviour is strongly dependent on the site conditions and factors such as the pH, and as such, the potential implications associated with the use sewage sludge or SSA in individual construction applications must be precisely evaluated.

Table 2.3: Data on the heavy metal contents of sewage sludge

PARAMETER	ELEMENT CONTENT, mg/kg												
	Ca	Mg	Na	Al	Fe	Mn	Cd	Cr	Cu	Ni	Pb	Zn	Hg
No. of samples	15	16	7	13	14	11	42	49	48	39	47	40	6
Mean	35099	7913	2937	17969	15519	447	87	287	383	278	198	1001	1.7
Min	2308	688	1538	221	619	3	0.1	0.2	3	1	1	22	0.5
Max	173874	30721	5600	66600	43000	1700	1713	3809	1491	4140	1090	4130	3.3
St Dev	45363	7721	1583	17040	13872	525	274	605	337	733	291	939	1.1
CV, %	129	98	54	95	89	118	315	211	88	264	147	94	65
Fertiliser Limiting	Require	ments											
Germany	-	-	-	-	-	-	50	-	_	80	150	1000	1
Austria	-	_	-	-	-	-	15	667	778	100	100	3333	1
Switzerland	-	_	-	-	_	-	3	200	400	50	200	1300	-
The Netherlands	-	_	-	_	_	-	1.5	300	600	100	100	1500	1

St Dev - Standard Deviation, CV - Coefficient of Variation

Data from: Ates and Pehlivan (2014), Bacon et al. (2001), Bonfiglioli et al. (2015), Carbonell et al. (2009), Fuentes et al. (2004), Fytianos et al. (1998), Fytili and Zabaniotou (2008), Green and Tibbett (2001), Hospido et al. (2005), Houdkova et al. (2008), Husillos-Rodriguez et al. (2013), Huybrechts (2001), Lederer and Rechberger (2010), Lim et al. (2006), Lin et al. (2006a), Malliou et al. (2007), Mun (2007), Paramasivam et al. (2005), Petersen (2001), Sajwan et al. (2003), Sort and Alcaniz (1996), Tirado-Montiel et al. (2001), Valls and Vazquez (2001), Weng et al. (2003), Werther and Ogada (1999) and Xu et al. (2008).

2.4 Management of Municipal Solid Waste and Sewage Sludge

In line with the sustainable waste management policies outlined in Chapter 1, the treatment of materials such as MSW and SS is shifting towards more environmentally favourable treatment options. Data on the treatment type breakdown in the EU countries in recent years, as given in Eurostat (2017c and 2017d) have been graphically illustrated in Figure 2.3 (a) for MSW and 2.3 (b) for SS. It should be noted that the recorded data for MSW are more comprehensive compared to sewage sludge. For the latter, data were not reported in some countries for certain years, which led to a degree of choppiness in Figure 2.3 (b).

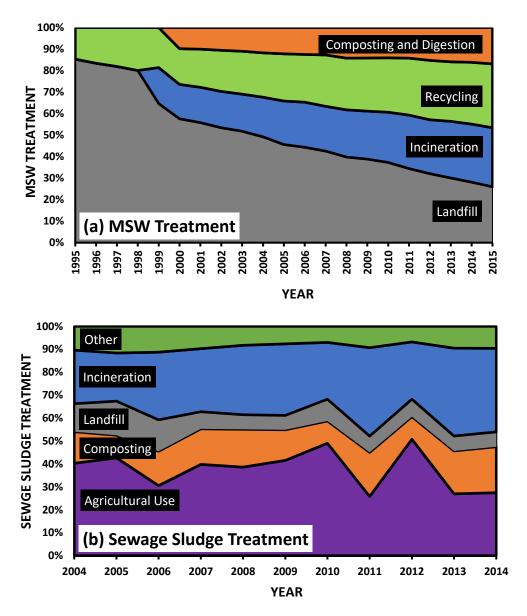


Figure 2.3: Management practices in recent years for (a) MSW (data from Eurostat, 2017c) and (b) sewage sludge (data from Eurostat, 2017d)

Despite the impacts of more environmentally conscientious legislation and policies, landfilling remains amongst the most common treatment options for MSW. However, management practices vary drastically from country to country, particularly when considering developing regions outside the European Union. In countries such as Sweden, Switzerland and The Netherlands the focus is on moving towards sustainable treatment, discouraging landfill to a great extent and seeking to minimise waste production and promote re-use and recycling of the residual materials (Arm, 2003; Bourtsalas et al., 2013). This is also the case in other EU

member states, though countries such as the UK still have more reliance on landfilling, which it is seeking to phase out gradually over the coming years.

In contrast, in developing countries such as China and India, there are major inadequacies in the systems, beginning with the collection and sorting processes (Chen et al., 2010; Chattopadhyay et al, 2009). There are large fractions not collected and not treated in accordance with the regulations, leading to health and environmental problems. The emphasis in these areas is on increasing the quantity of safely disposed waste, and there is not the same focus on the sustainability of the treatment technologies. It is very difficult to overcome these problems, given that the treatment quantities increase as collection rates increase and also because of the effects of population growth. This puts huge demands on the management systems and, in a lot of cases, the controlling bodies simply do not have the financial resources to cope. These issues inevitably lead to human health hazards and environmental damage due to inadequate collection and treatments.

The management of SS has relied more on its use as a fertiliser on agricultural lands and related applications such as composting, whilst landfilling has played a smaller role. However, because of the health risks associated with the build-up of heavy metals from the sludge, countries have begun to adopt a more cautious approach towards the spreading of SS on soils. Utilisation of the material in construction applications such as cement clinker production, lightweight aggregate production, ceramics and soil stabilisation has also been explored somewhat, along with other miscellaneous uses in biotechnology products, carbon products, animal feed and the removal of metals from wastewater.

Incineration plays an important role in the treatment of both MSW and SS. The high initial setup cost of the plants makes them more suited to large-scale operations and this is also a contributing factor towards the limited number of incinerators in developing countries. The incineration process results in an approximately 70% reduction by mass and 90% reduction by volume of the material. Bottom ash makes up 80%–90% of the residual material, with the rest being fly ash and air pollution control residues depending on the plant's cleaning system. As

outlined in Table 1.1, MSW production rates of 242 MT in the European Union (Eurostat, 2017a), 663 MT in the OECD countries (OECD, 2017) and 1840 MT worldwide (Waste Atlas, 2013) per annum have been reported. Incineration rates of 27% in the EU (Eurostat, 2017c) and 24% in the OECD (OECD, 2017) have been recorded. Complete data on the worldwide incineration rate have not been documented, though it is assumed that it would be quite low in the majority of developing African and Asian countries because of the high initial capital costs associated with the plant setup and the lower priority given to waste management.

As for sewage sludge, 36% of the material has been incinerated in the 28 European countries per annum (Eurostat, 2017d). Countries such as The Netherlands, Switzerland and Belgium, with limited space for landfilling, have the highest rates of sewage sludge incineration at 100%, 97% and 83%, respectively. Within the EU, Germany incinerated the largest overall quantity of sludge, totalling 1.1 MT of dry matter annually (Eurostat, 2017d).

The rate of incineration is strongly influenced by the waste management approach of each government, manifested in the policies implemented and the associated economic implications. For example, since a landfill tax was introduced in the UK in 1996, the standard rate has continually increased from an initial £7 per tonne up to £84.40 per tonne in 2017 (British Government, 2017), which has contributed to reduced landfilling and has further incentivised incineration. However, despite the overarching European legislation that emphasises sustainable waste management practices, the specific tax rates adopted in different countries vary substantially. Indeed, incineration taxes have also been adopted in certain countries such as Belgium and France. As discussed by Dubois (2013), with rising levels of trade taking place across the European market, greater harmonisation of the taxation policies in neighbouring countries needs to be encouraged to avoid large cost discrepancies between competing waste management companies that could indirectly discourage sustainable practices in certain locations.

Based on the previously outlined records, MIBA production per annum is estimated at around 17 and 41 MT in the EU and OECD countries, respectively, whilst SSA production is estimated

at upwards of 0.5 MT annually in the EU. Though the quantity of SSA to manage is considerably less than that of MIBA, both materials present a major management challenge in many regions across the world. Indeed, with the aspirations of moving towards a truly sustainable society, there must be a desire to develop appropriate uses for all materials as resources.

2.5 Incineration Process

The process of incineration can be split into three main parts: (i) combustion, (ii) energy recovery and (iii) the air pollution control system. A flow diagram outlining an example of the typical components involved in each stage of MSW incineration is presented in Figure 2.4, in which the output residues are also identified. The majority of the residue, that is, the bottom ash, is produced at the combustion stage, whilst the additional fly ash and air pollution control residues are collected in the process of cleaning the flue gases before they are emitted through the stack.

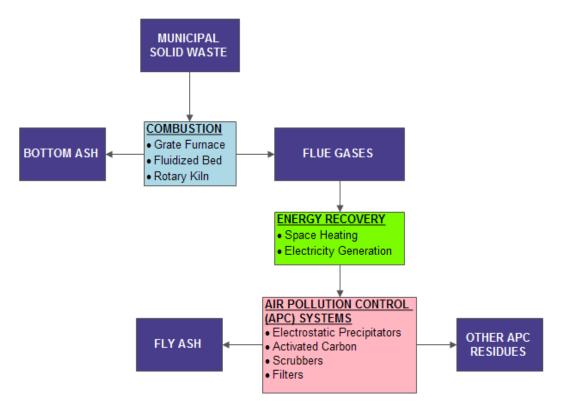


Figure 2.4: Main stages of the incineration process and the output residues produced

Prior to being fed into the furnace, the MSW and SS materials undergo varying levels of pretreatment. Pre-incineration treatment of MSW can be broadly broken down into two categories:

- Mass burn systems the as-received material is fed into the plant with very minimal processing
- Refuse-derived fuel systems pre-processing such as shredding and sorting are incorporated to improve the homogeneity of the MSW

The treatment of SS typically involves thickening, dewatering and/or drying to increase the solids content and the calorific value of the material prior to combustion.

At the combustion stage, grate furnace and fluidised bed combustor are the most commonly used set-ups. The fluidised bed is the newer system in which the input waste during combustion is suspended in a bed of inert material behaving like a fluid, as opposed to burning the material on a grate. Rotary kilns were also used, though less commonly. Each system has its advantages and disadvantages but generally the modern fluidised bed combustor is considered easier to operate, provides more flexibility and causes less wear on the equipment because there are no mechanical moving parts, whilst the rotating action of the kiln promotes better mixing of the fuel, though leads to greater entrainment of the particulate matter and fly ash residues.

During heating at temperatures typically around 850°C, the volatile and organic components are burnt off as CO₂ and other gases and are conveyed out of the furnace chamber, along with the fine particulate inorganic matter. The inorganic fine particulates and exhaust gases then pass through a heat exchanger, where the energy can be reused to heat the combustion air or as part of a pre-drying treatment of the waste, or recovered in the form of electricity. The flue gases are subsequently conveyed through the cleaning system to remove the particulates and harmful gases, before being released out of the stack. Air pollution control systems can contain various cleaning components, including scrubber units, electrostatic precipitators and filters, which leave behind the residual fly ash and other air pollution control residues.

The levels of emissions present in the flue gases are affected by many factors such as the composition of the waste input, temperature, moisture, residence time for the flue gases, turbulence, oxygen concentration, refuse loading and chlorine behaviour, as well as the type of combustor and air pollution control devices. The emissions of concern include:

- Heavy metals
- Particulate emissions
- Sulphur oxides (SO_x)
- Nitrogen oxides (NO_x)
- Carbon dioxide (CO₂)
- Carbon monoxide (CO)
- Hydrochloric (HCL) emissions
- Dioxins and furans (PCDD/Fs)
- Polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs)
- Dust
- Chlorofluorocarbons (CFCs)

Given the damaging consequences that could potentially arise from these emissions, substantial resources have been allocated to this area to ensure that the incineration plants operate safely. Emission limits, such as those given in European Directive 2000/76/EC on the incineration of waste (European Community, 2000), are required to be tightly adhered to by plant operators. Incineration plants with effective cleaning systems ensure that emissions released through the stack are at an acceptable level.

2.6 Post-Incineration Processing

After incineration, various additional treatments of the ashes are usually implemented to improve their suitability for their end destination. This can include the recovery of valuable metals from the ashes or upgrading the quality of the material for use in construction, particularly in relation to its leaching characteristics. It should be noted that more of the interest regarding the following treatments has been directed at MIBA, rather than SSA, as the former is the much larger waste stream and more development has taken place regarding

its use in construction, predominantly in road pavement applications. The following treatments have been applied to the incinerated ashes:

- Quenching/air cooling: After the high temperatures of combustion, the ashes can be
 rapidly cooled in water (quenched) or slowly air cooled. The rate of cooling directly impacts
 the ash mineralogy, with the slower cooling tending to lead to a more crystalline material.
- <u>Drying</u>: After quenching, the moisture content of the ash may be quite high and drying can be undertaken to make it more suitable for further treatment or its end use. In other cases, the bottom ash may be stored and aged outdoors for long periods of time, which reduces the water content by evaporation.
- <u>Size separation</u>: In its as-produced form, MIBA can contain particles up to 100 mm in size.
 The oversized fraction is typically removed by screening. Further sieving and grinding may be adopted for MIBA and the finer SSA to match the requirements for their use as construction materials.
- Ferrous and non-ferrous metal removal: Although varying with the composition of the waste input, typically MIBA contains around 7%–15% ferrous metals (e.g., tin cans, nuts, bolts, pins, etc.) and 1%–2% non-ferrous metals (mainly aluminium, copper and zinc) by weight. It is standard procedure in the processing of MIBA to separate the ferrous metals through magnetic separation, whilst removal of the non-ferrous metals by eddy-current separation may also be done in some cases. The separation of these fractions not only creates a stream of income from selling the scrap, but also improves the properties of the ash to make it more suitable for utilisation.
- Ageing: This involves storage of the material for a period of time to allow the carbonation and hydration reactions to occur. Carbonation involves the reaction of the alkaline ash with the CO₂ in the atmosphere, resulting in the formation of carbonates. Hydration reactions also result in a swelling of the material. These changes bring about the formation of more stable mineral species and a reduction in trace element contents, including heavy metals. Rapid hardening of the material can also occur, as well as reduction in the porosity due to the swelling, leading to improved engineering properties. The pH of the ash changes from an alkaline value around 12 to more neutral conditions, resulting in improved

leaching behaviour and acid neutralisation capacity. The storage time required for ageing depends on the application requirements, varying from a minimum of 6 weeks (Van Beurden et al., 1997) to 6 weeks to 6 months (Sabbas et al., 2003) or 2 to 3 months (Astrup, 2007). The leachate generated during the storage period must also be taken into account and managed appropriately, whilst there is the additional health concern of dust during handling or wind blowing the ash. An accelerated ageing treatment can also be carried out by optimising the ageing conditions (water content and the partial pressure of CO₂) to promote the reactions. Although there has been a lot of research on this topic, the accelerated experiments appear to have been carried out only at a laboratory scale.

- Washing and extraction: Washing is a process of upgrading the ashes by using water as a leachant for the removal of soluble salts, chlorides, sulphates, alkalis and heavy metals. This process is an economic and energy-efficient way of improving the ash quality, although it may need to be used alongside other processes such as ageing or stabilisation to produce ash that exhibits the desired leaching characteristics. The treatment efficiency depends on factors such as the particle size and leachant concentration. The process of extraction uses additional chemical additives for further removal or recovery of heavy metals from the incinerated ash. A number of extraction solvents have been tried, such as hydrochloric acid, nitric acid, sulphuric acid, aqua regia and chelating agents. Processes to remove the metals from the leachate include precipitation, ion exchange and absorption. The effectiveness of this process is mostly determined by the solvent used, the solvent concentration and the pH of the ash, whilst there also needs to be a high content of heavy metals present. At the moment, this process is really only at a development stage because of the higher costs of the currently available extraction procedures and it may be a more suitable treatment option only for the fly ashes with an abundance of heavy metals.
- <u>Solidification/stabilisation (S/S)</u>: These terms are often used together, though the processes treat the ashes in different ways. Solidification involves transformation of the ash into a more solid monolithic form so that the trace elements are encapsulated within the matrix, whilst stabilisation involves a chemical change that reduces the solubility and therefore the mobility of these fractions. These treatments are often adopted in combination, typically when managing fly ash. Materials that are used for the S/S process

include cement, lime, other pozzolanic materials, polymers, bitumen and phosphate. Cement is particularly effective as it creates a solid monolithic form and transforms portions of heavy metal into insoluble compounds. Lime can have similar stabilisation effects of raising the pH of the ash into a range in which heavy metals become less mobile. Whilst the use of some of these materials for S/S treatments can be very beneficial in upgrading the leaching performance of the ash, one disadvantage is that with the addition of these binders, a larger volume and weight of material to be managed is created, which may lead to additional transport costs or disposal costs, if that is to be the end use.

- Thermal treatments: thermal treatment of the ash can be used to achieve improved leaching performance, along with a consistent denser residue. This process is typically used in lightweight aggregate or ceramics production. The thermal treatment can be split into three different types: vitrification, melting and sintering. Vitrification involves mixing the ash with a glass precursor and melting the mix (typically within the 1000°C to 1500°C temperature range) and then cooling it to form a single-phase amorphous, homogeneous residue. The melting treatment is comparable to vitrification, except no glass precursor is present and a multiple-phased product is formed rather than the single phase. The last process, sintering, involves the heating of the MIBA residues to a temperature to bring about a bonding of particles and cause changes to the configuration of the chemical phases. The end result is a less porous and higher strength specimen (Chandler et al, 1997). Although these thermal processes are commonly used, there are drawbacks, such as a high energy demand and concerns about the mobilisation of certain volatile heavy metals that may occur during the high-temperature treatment.
- Phosphorus recovery: With caution surrounding the direct application of SS to agricultural lands as fertiliser, treatments have been developed to recover the valuable phosphorus from SSA. The key focus of this treatment is to minimise the heavy metal contents and increase the phosphorus bioavailability. The main methods developed are wet chemical and thermochemical treatments. The former process involves the removal of phosphorus from SSA by elution, typically using strongly acidic solvents such as H₂SO₄, which are subsequently recovered by precipitation or ion exchange. Thermochemical treatment involves the addition of chlorination agents such as CaCl₂ or MgCl₂ during heat treatment

of SSA. The chlorination agents react with the heavy metals to form volatile chlorides, which are subsequently evaporated at the high temperatures, whilst the P- bioavailability improves with the formation of phosphorus-bearing minerals such as chlorapatite, Mg-phosphates and Mg-Ca-phosphates.

The viability of these processing techniques will be very much dependent on local circumstances. The assessment should consider factors such as the distance from the incinerator to a suitable upgrading facility or the potential setup costs, storage area demands, labour costs, maintenance costs and environmental impacts arising from dust, noise and energy usage. Although not favourable from the environmental point of view, in practice, parameters such as the costs associated with landfilling and the availability of local natural materials, as alternative short-term options, may come into play and affect decisions on how the ashes are processed.

2.7 Summary

This chapter presents a background overview of the production of the MIBA and SSA. These two materials are produced from the incineration of MSW and SS, respectively. MSW is described as waste from households, whilst SS is generated during wastewater treatment. After collection, the raw SS undergoes a series of treatments that can include thickening, stabilisation, condition, dewatering, drying and thermal processing before its use or disposal.

The composition of MSW can be highly variable, influenced by economic status, cultural issues and waste management policies, whilst the properties of SS are determined by the populations' density and habits, treatments undertaken and the industrial contribution. Organics, paper/cardboard, plastics are the main components in MSW. SS contains high organics and volatile matter, and has a low solids content. Heavy metals are present in both materials in notable quantities that demand attention from the environmental standpoint. Prior to being fed into the incinerator, sorting techniques can be adopted to lessen the aforementioned variability of the waste and indeed, modifications at this stage may become further refined, as recognition of resultant ashes as valuable construction materials grows.

Driven by sustainable waste management legislation and policies, landfilling is declining in European countries, though this outlet still makes up a high proportion of overall treatment, particularly for MSW, whilst the management of SS relies more on its use as a fertiliser on agricultural lands. Incineration plays an important role for both materials, accounting for 27% and 36% of the total treatment of MSW and SS, respectively, in the 28 European Union countries. This treatment results in an approximately 70% reduction by mass and 90% reduction by volume of the material, whilst energy can also be recovered. Owing to high setup costs, more densely populated areas are favoured for incineration facilities, though with landfill costs on the rise, it is expected that the rate of incineration of MSW and SS will continue to increase, along with other more environmentally desirable options such as recycling.

The incineration process consists of three key parts: combustion, energy recovery and air pollution control. For combustion, grate furnace for MSW and fluidised bed combustors for SS are the most commonly used setups. Under the applied high temperatures, volatile and organic components are burnt off and for the grate system, a bottom ash is produced. The gases are conveyed out of the chamber, along with the fine particulate inorganic matter, and pass through a heat exchanger, where the energy can be reused as heat or recovered as electricity. The flue gases then pass through the cleaning system to remove the particulates and harmful gases, leaving behind residual fly ash and other air pollution control residues. Provided the air pollution control system is operating effectively, emissions released from the stack should remain within the regulatory thresholds, though a large proportion of the heavy metals end up in the residual ashes.

Post-incineration processing is generally adopted, mostly with MIBA, to improve its suitability for disposal/use. This can include quenching, drying, size separation, ferrous and non-ferrous metal removal, ageing, washing, extraction, solidification, stabilisation and vitrification, melting or sintering. For SSA, recovery of phosphorus using wet chemical or thermochemical treatments has been undertaken, though this is done for its value in agriculture, rather than to improve the ash characteristics for its end use.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter describes the novel approach adopted for the methodology of this study. A fundamental part of research is exploring new ways to more effectively contribute to the knowledge in the area of interest, whilst originality and innovation are also strongly championed for successful academic work. In the area of concrete technology, the general approach to research that centres around tightly focused laboratory testing has been proven as an effective method over the years, though given the previous considerations, it seems incongruous for researchers to be tied down to any one system. There is a crucial need for different approaches to be received with an open mind, though each methodology should be rigorously judged on its merits.

The focus of this project is very much on the aspect of analysing and evaluating data, and subsequent modelling work. These data were comprehensively sourced from globally undertaken research, rather than being produced in a single laboratory or from a localised area. *Analytical systemisation* is the name given in this thesis to the full process that involves obtaining and preparing the data, followed by the analysis, evaluation and modelling work.

3.2 Analytical Systemisation

The *analytical systemisation method* developed in this work served as a very useful tool in initially establishing the current status regarding the characteristics of MIBA and SSA and their use as construction materials. This was followed by a comprehensive analysis and evaluation of the collective data to further advance their safe and sustainable use, with a specific focus on concrete-related applications. Culminating with a specific focus on one key aspect of concrete performance, compressive strength, robust and practical models for the prediction of this parameter with the use of these incinerated ashes were developed.

3.2.1 Identifying and Sourcing the Data

The first step of the methodology consisted of a comprehensive search of the global literature, using an extensive list of keywords relating to incinerated ashes and a large number of search engines and websites. Sourcing of the data was a mammoth task that was undertaken until the rate of emerging publications, over a prolonged time-period, ground to a halt and the search was deemed exhausted. In addition to the open search, the reference list of each publication was also explored to further boost the obtained data. The local library, as well as the inter-library loan services offered by the university, was also utilised. The search in this project was limited to literature published in English for practical purposes, though it is envisioned that in the future this approach could be potentially expanded to multiple languages with a team of international researchers.

Over 2000 publications were sourced in total, covering all aspects relating to MIBA and SSA, including the ashes' production, their use in many construction applications such as concrete, road pavements, bricks, tiles and glass-ceramics and the environmental assessment. In the initial stages of the project, all of these aspects were dealt with and indeed, three papers were eventually published in reputable journals on the use of MIBA and SSA in the fields of road pavement and ceramics, which are not covered in this final thesis (Lynn et al., 2016a, 2016b and 2017a). As the work progressed and the picture became clearer, it was decided to firmly focus on concrete applications. This was identified as the area in which the most effective contribution could be made, as there are significant data available and a major scope for development of this use in practice.

With the refined approach targeted at the use of MIBA and SSA in concrete-related applications, a total of 614 publications are cited in this thesis. In addition, 49 standards and specifications and 40 supplementary references are also referred to as required in the various chapters. A summary of the metadata for these main MIBA and SSA publications is presented in Table 3.1.

Table 3.1: Metadata for publications on MIBA/SSA and their use in concrete

PARAMETER	COVERAGE	HIGHEST FREQUENCY
Year	1972-2017	2006 (46 publications), 2007 (43), 2012 & 2014 (41)
Countries	54 Countries Worldwide	UK (74 publications), Taiwan (71), USA (56), China (49)
Continents	6 Continents	Europe (367 publications), Asia (246)
Authors	1397 Authors	Lin K L (20 publications), Adam C (17), Cheeseman C (17), Lin D F (15), Dhir R K (14)
Institutions/ Organisation	495 Institutions s	BAM Federal Institute for Materials Research and Testing (23 publications), Imperial College London (19), Nanyang Technological University (19)
Publication Types	Journal papers (76%), Conference Proc. (9%), Reports (8%), Book/ extracts (3%), Others (4%)	e Journals: Waste Management (88 publications), Journal of Hazardous Materials (41), Construction and Building Materials (21)

Proc – Proceedings

The huge scale and global nature of the obtained literature are evident from this information. Work on this subject has been undertaken by a staggering 1397 authors, from 495 institutions/organisations, stemming from 54 countries, covering 6 continents, over 45 years. On the publication timeline, the quantity of work undertaken really started to pick up around the turn of the 21st century, reached a peak in 2006 and has remained consistently high over the past 10 years or so. A large proportion has been carried out in developed European and Asian countries, with the UK and Taiwan leading the way. The most active researchers and institutions/organisations are based in Taiwan, Germany and the UK. Peer-reviewed journal papers comprise the bulk of the publications, of which Elsevier journals have contributed a particularly high number.

The immense diversity and overall quantity of the collective data obtained greatly surpass what could be achieved by a single individual. This gives a great advantage to the robustness of the subsequent analysis and evaluation undertaken. The emerging findings also carry a higher likelihood of being more widely applicable, with greater repeatability and reproducibility by other parties in different locations.

3.2.2 Building the Data Matrix

This stage of the process essentially lays down the foundation for the succeeding analysis, evaluation and modelling work. As mentioned previously, the *analytical systemisation method* offers many advantages; however, at the same time, managing this large collection of data in itself presented a major challenge that demanded meticulous organisation and intelligent decision-making throughout, to handle the inevitable differences that can be present in many aspects, such as the material properties, the concrete mix designs and the test methods adopted. It was imperative that the initial base was set up correctly, as a weakness in the thoroughness could greatly compromise the reliability of findings emerging at the later stages.

The building of the data matrix began with an initial sorting. A screenshot showing a sample of this process is presented in Figure 3.1. This essentially involved vetting of the publications to ensure each contained relevant data and at the same time sorting them under initial broad headings such as 'MIBA properties' or 'Cement'. This was then followed by a more specific data sorting into further subdivisions, for example, for the ash characteristics, this entailed subheadings such as 'Density', 'Particle Size Distribution' and 'Oxide Composition'.

Data mining was the next step in the process and involved the extraction of both qualitative descriptive information in text form and quantitative data from Tables and Figures in the publications. For the data specifically given in Figures, the software package Plot Digitizer was useful for getting a precise reading of the values. This mining process was undertaken for each of the subheadings developed in the previous step, yielding a large amount of data. In total, the data matrix was built up to over 200,000 data points, which in a systematically coordinated form, served as a very valuable assortment of information on the ashes' properties and their performance as a concrete material.

A sample of the data mining process and formation of the data matrix is presented in Figure 3.2. This shows particle size distribution data that were extracted from the literature, mostly from Figures, for both as-produced and ground SSA samples, and subsequently used in the creation of Figure 4.1 (b) on the ash characteristics.

or		try	Published	Processing	Incineration	MIBA Properties	ant	ar	Concrete	Lightweight agg	Road pavements	Geotechnics	Ceramics	Agriculture	Other Applications	Envir impact	Case Studies	Standards & Specs	Management
Author	Year	Country	inbli	IO C	ě	₩.	Cement	Mortar	000	ight	oad	eot	9	ij.	the	ž	ase	tanc	Jana
Fernandez	-		Green		=	y Z	-		-		-	6	0	ব্	0	У	0	S	_
Fernandez				-		y	У									,			
Fernandez		-		-		y	,												
Fernandez		-				,								у					
Ferrari et						У								,					
Ferraris e						y							у						
Ferraris e		-				y		у	у				,						
Ferre-Hug		-		,		,		,	,							у			
Filipponi						У	У									,			
Flehoc et a		-				y	7												
Floret et a					у	,										у			
Floret et a					y											y			
Flyhamma					y	У					У					y			
Forestier :					у	У					y					y			
Forrester				v	y	y										y			у
Forrester			Journa	_		у						У					У		y
Forstner						У						У				У	У		У
Forteza et				-		У						у				У			
Forth et al		-	Procee			У			у		у	y							
Francois a						y			y		У	y				y	у		
Francois e											y	y				y	y		
Freyssinet						v					У					v			
Frogner K				-		y					у					У			
Fruergaar				_	v	y					y					у			У
Fuchs et a					У									v					У
Fujii and I		-												У		У			
Gagnepair		-			У	v										v			
			Maste	v		y										y			
Gao et al.				-		У										У			
Gao et al. Garcia et				•		v								v					
Garcia-Ub					v	У								У					
Gardner e					У	v										v			
Gardner e Garrick ar			Transi			У					.,					У			
											У					.,			
Gau and C																У			
Gau and J														.,		У			
			Biores											У					
Giampaol		-				У	У									У			
Gines et a			Proces		v	y v			У							У			

Figure 3.1: Sample of the initial sorting process

AUTHORS	YEAR	PROCESSING	APPLICATION	PARTICLE SIZE DISTRIBUTION DATA											
Al Charifand A	2044		Soil Stabilization	Size, mm	0.001	0.005	0.02	0.074	0.21	0.42	0.85	2	2.38	4.76	
Al-Sharif and At	2014	Unprocessed		Passing, %	2	12	20	56	83	98	100	100	100	100	
	2005	Unanananan	Mantan	Size, mm	0.0004	0.0040	0.016	0.033	0.059	0.079	0.110	0.169	0.213	0.378	0.501
Alcocel et al. (20	2006	Unprocessed	Mortar	Passing, %	1.13	4.52	16.27	29.31	44.60	60.09	75.00	85.92	94.22	99.69	99.995
Anderson and C	derson and S 2003 Unprocessed		Bricks	Size, mm	0.001	0.005	0.01	0.05	0.1	0.3					
Anderson and S		Unprocessed		Passing, %	0	7	12	58	84	100					
Dhatty and Daid	y and Reid 1989 Unprocesse	Unprocessed	Lightwoight agg	Size, mm	0.1	0.07	0.056	0.038	0.027	0.018	0.013				
Briatty and Reid		Unprocessed	Lightweight agg	Passing, %	92	82.5	68.4	52	38.9	28.75	22.8				
Chaaramanana	2005	Unprocessed	Lightweight agg	Size, mm	0.0010	0.0041	0.010	0.026	0.044	0.068	0.092	0.121	0.201	0.361	0.553
Cheeseman and	neeseman and 2005 Unpr	onprocessed		Passing, %	0.50	4.3	11.1	22.4	33.3	46.5	60.8	74.0	89.3	99.1	100
Coutand et al. (2 2006	Processed	Mortar	Size, mm	0.001	0.005	0.01	0.05	0.1							
			Passing, %	0	10	25	85	100							
Donatello et al. 2010	2010	Unprocessed	Cement	Size, mm	0.0010	0.013	0.033	0.056	0.075	0.128	0.159	0.196	0.226	0.394	0.501
Donateno et al.	Jonateno et al. 2010			Passing, %	1.34	10.9	24.7	35.9	43.7	58.1	66.1	75.5	82.4	97.1	100
Donatello et al. 2010	Unprocessed	Cement	Size, mm	0.0135	0.1068	0.3144									
Donateno et al.	2010	onprocessed	Cement	Passing, %	10	50	90								
EWTCI (2012)	2012	Unprocessed	Pavement	Size, mm	0.063	0.125	0.25	0.5	1	2	4				
LW1C1 (2012)	2012			Passing, %	1.73	3.26	5.01	7.49	18.22	79.08	99.93				
Gever et al. (200	2002	Processed	Concrete	Size, mm	0.0005	0.0009	0.003	0.005	0.009	0.02	0.04	0.08	0.2	0.3	0.37
deyer et al. (200	et al. (200 2002 Processed	Frocessed		Passing, %	2	4	9	14	20.5	32.5	48	67	88	97	100
Fontes et al. (20	tes et al. (20 2004 Processed	Drocessed	Mortar/Concrete	Size, mm	0.0001	0.0002	0.0004	0.001	0.003	0.006	0.01	0.02	0.03	0.05	0.07
rontes et al. (20		Frocessed	Wortar/Concrete	Passing, %	0	3	10	17	30	45	58	78	89	97	100
Franz (2008) A 2008	2008	Unprocessed	Fertiliser	Size, mm	0.06	0.09	0.13	0.26	0.5	0.7	1	2	4		
11a112 (2008) A	2008			Passing, %	6	8	10	18	49	75	96	100	100		
Franz (2008) B	2008	3 Unprocessed	Fertiliser	Size, mm	0.06	0.09	0.13	0.26	0.5	0.7	1	2	4		
F1 a112 (2006) B 2008	onprocessed	rentiliser	Passing, %	22	31	40	74	98	99	100	100	100			
Khanbilvardi an 1995	Unprocessed	Concrete agg	Size, mm	0.075	0.15	0.3	0.6	0.85	1.18	2.36	4.75				
Kilalibilyaldi ali	Mianonvarurani 1995	onprocessed	Concrete agg	Passing, %	38.8	53.2	65.6	80.6	86.7	92.4	98.3	100			
Khanbilvardi an		Unprocessed	Concrete, Pavement	Size, mm	0.074	0.149	0.177	0.297	0.42	0.595	0.841	1.19	2.38	4.76	
Kilalibilyalul all	2002			Passing, %	49	63	66	76	83	87	90	91	95	96	
Koisor-Kazberul	2011	Unprocessed	Concrete agg	Size, mm	0	0.25	0.5	1	2	4	8				
koisor-kazberui 20:	2011	onprocessed	concrete agg	Passing, %	0	7.5	11	19.2	42.2	93.2	100				

Figure 3.2: Sample of the data mining process on the particle size distribution of SSA, as part of the data matrix formation

3.2.3 Analysis, Evaluation and Modelling

The analysis, evaluation and modelling is the critical stage from which emerged the salient findings on the characteristics of the incinerated ashes and their use in concrete. As discussed previously, one of the key challenges is managing the variability present in many aspects of the data, including in the quantity of results available, the form in which they are presented and, indeed, the magnitude of the values. The approach in the analysis and evaluation undertaken for a particular set of data was very much dependent on these parameters. Keen attention to detail, sensitivity and strong independent judgement were required in the handling of the data and, indeed, the decisions made throughout were grounded in a solid understanding of concrete technology.

There was no one optimal strategy that was followed throughout for the analysis and evaluation process, and the organised manner in which the data matrix was assembled allowed for a great deal of flexibility in managing the collective results. Data from individual studies, lying in separate pockets, were naturally somewhat disconnected, and as such, with the goal of coordinating and bringing together the collective results, an imaginative touch was required in determining the most effective way to uncover and present the key findings to the reader. Converting raw results into relative values and comparing with control mix performance or other accepted construction materials were frequently adopted over the course of the work, whilst standards and specifications were also referred to when applicable for assessment of the material properties and the concrete performance.

A sample of the analysis and evaluation process is presented in Figure 3.3, which is taken from Section 5.2.1 on the effect of MIBA as an aggregate replacement on the relationship between the compressive strength and the elastic modulus of concrete, with comparisons to the curves established for natural aggregates in Eurocode 2 (BS EN 1992-1-1, 2004). Data shown on the left side were organised into a number of mix series (note: not all the data are shown in this screenshot), which were then plotted in the graph to provide an overall comparison of the results for MIBA mixes with the control and Eurocode 2 values. Thereafter, on the right side, the data for the all MIBA mixes were combined together and sorted, according to the

replacement level, into three categories: 20%–30%, 50% and 70%–100%. Trend lines were then plotted to represent each of these three categories in the corresponding graph on the bottom right, which provides an important insight into the effect of the MIBA replacement level on elastic modulus performance.

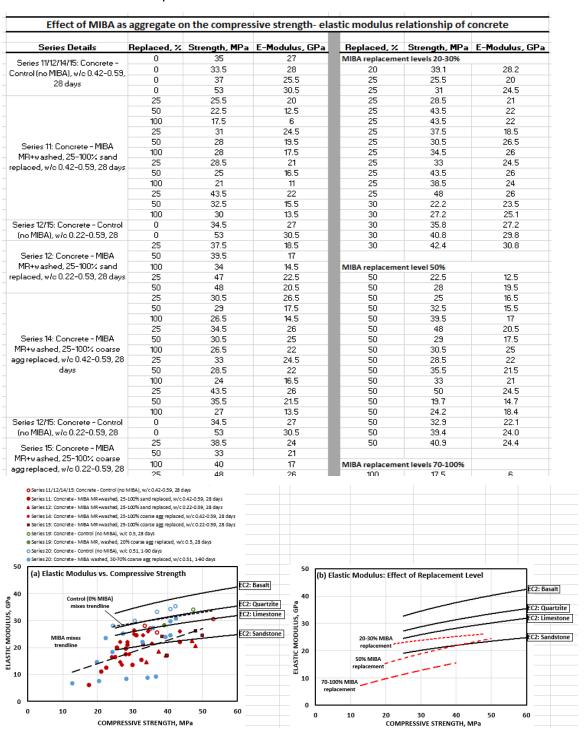


Figure 3.3: Sample of the analysis and evaluation work on the effect of MIBA as aggregate on the relationship between compression strength and elastic modulus of concrete

For the modelling work, the goal was to develop a further in-depth understanding of one particular aspect of concrete performance, which in this case was compressive strength. Priority was given to ensuring that the models retained a degree of familiarity to established models for normal concrete mixes and were straightforward to operate, using input parameters that would be generally available to the engineer. Two models were developed, one for use of the incinerated ash as an aggregate component, and the other as a cement component. The models were set up to account for the characteristics of the ashes and were not designed specifically for either MIBA or SSA. As such, with the influx of further data on other incinerated ashes, it is envisaged that the models could be extended to operate for incinerated ashes in general. This work is the first of its kind for estimating concrete strength with incinerated ashes. It offers an informed view of how the properties of these materials affect the strength behaviour, which can assist the designer in proactively evaluating the properties of local ashes and, in doing so, avoid potential large strength reductions, whilst the strength estimates should also lead to much more focused initial trial testing with MIBA and SSA in concrete mixes.

3.3 Peer-Review Feedback and Dissemination

From the beginning, an approach was adopted to continually publish the work as the project progressed. There were two strong reasons for this, the first of which was to obtain valuable feedback from esteemed colleagues in this field. As the work in the various subject areas was completed, the information was converted into a paper format and was submitted to reputable journals including the *Magazine of Concrete Research* and *Environmental Geotechnics*, published by the Institute of Civil Engineers, and *Construction and Building Materials*, published by Elsevier. Comments received from the reviewers were very useful in improving the quality of the final published papers and indeed the constructive points were carried back to contribute positively to the main thesis.

The second reason great emphasis was placed on publishing the work was to disseminate the findings to a wide audience and make a tangible original contribution to the knowledge in this field. The goal was to produce work of publishable quality that could be of use to the industry.

In doing so, it is hoped that this project can contribute to progressing the safe and sustainable use of MIBA and SSA in concrete applications, as well as reducing repetitive work that is wasteful of resources and instead stimulating more effective further innovative research.

The work in the main body of this thesis on the ash characteristics and their use in concrete applications, has already been published, after converting it into the paper format (see the List of Publications). For completeness, it is also planned to publish a final paper on the closing modelling work, immediately after the initial submission of the thesis.

3.4 Summary

An original *analytical systemisation method* has been adopted in this project covering the characteristics of incinerated ashes and their use in concrete-related applications. This process began with the comprehensive identification and sourcing of the global data on this subject. A huge quantity and immensely diverse collection of experimental results was obtained, stemming from 1397 researchers, 495 institutions/organisations, 54 countries and 6 continents, over a period of 45 years.

The individual data were, however, naturally somewhat disconnected and needed to be systematically coordinated in their collective form to allow the full benefit to be gained from the subsequent analysis and evaluation work. This preparation stage involved the forming of a data matrix through the initial sorting of the literature under main headings and subdivisions, followed by data mining, consisting of the extraction of qualitative descriptive information and quantitative data. This yielded a final data matrix with over 200,000 data points.

Analysis and evaluation of the data were subsequently undertaken to uncover and present the salient findings on the ashes' properties and their performance as concrete materials. The approach at this stage had to be somewhat flexible, depending on the nature of the data available, though it required a keen attention to detail and sensitivity throughout, along with a strong independent judgement and solid understanding of concrete technology.

Comparisons to control mixes and other accepted construction materials were frequently utilised, along with regular tests of compliance according to standards and specifications.

The work culminated with the first comprehensive modelling work on incinerated ashes in concrete, as both aggregate and cement components, focusing on compressive strength. The developed models offer an informed view of how the ash properties affect the strength of concrete, which allows proactive evaluation of the suitability of local ashes. The strength estimates produced by the models allow more focused initial trial testing to be undertaken with MIBA and SSA in concrete. There is also potential for these models to be extended in future to be applicable for all types of incinerated ashes.

The approach taken over the course of the project was to continually publish the work in reputable journals. This was done to obtain the valuable peer-review feedback from esteemed colleagues in this subject area and also to disseminate the findings to a wide audience and make a useful original contribution to the knowledge on this topic, whilst also lessening wasteful repetitive work.

CHAPTER 4

CHARACTERISTICS OF INCINERATED ASHES AND IMPLICATIONS FOR USE IN CONCRETE

4.1 Introduction

Greater efforts are being made to explore the uses of recycled and secondary materials, such as SSA and MIBA, with the ultimate goal of transitioning towards a zero-waste society. As such, there is an imperative need for a wide-ranging and robust assessment of the properties of these emerging materials, to establish their suitability as potential construction materials and how they can be most effectively exploited, in a manner that satisfies the performance requirements, is safe and remains commercially viable.

The focus of this project is on concrete-related applications and as such, the characteristics of MIBA and SSA are examined in view of the potential implications, both positive and negative, on the performance if incorporated into these products. This work considers the prospect of using incinerated ashes in a variety of forms, including as potential fine, coarse and lightweight aggregates and in ground form, as part of the raw feed for cement clinker manufacturing and as a cementitious component.

The shift in the construction industry towards more performance-based specifications, rather than material-based, offers more flexibility for incorporating emerging materials such SSA and MIBA, provided that performance criteria can be satisfied. The requirements outlined in the relevant standards regarding the material properties, both for aggregate and cement components, were referred to frequently throughout the assessment of the ash characteristics. The traditional natural aggregate and Portland cement materials, as well as more established secondary materials such as coal fly ash and GGBS also served as useful benchmarks for evaluating the suitability of MIBA and SSA. However, it is important to note that the new materials can bring their own unique challenges that may not be covered in existing standards.

The analysis and evaluation in this chapter covers a wide range of physical and chemical properties of the two materials, including the grading, density, morphology, water absorption, permeability, oxide composition, loss on ignition, mineralogy, pH, element composition and organic compound contents. There has been a strong interest in developing the use of these materials and, as such, this has provided a diverse and comprehensive stock of global data. These collective results have originated from 46 countries worldwide, dating back to 1972. The largest contribution has come from Europe-based researchers (288 publications), followed by Asia (200) and North America (45), with the highest individual outputs from Taiwan (61 publications) and the UK (51).

4.2 Physical Properties

4.2.1 Particle Size Distribution

The grading of the incinerated ashes is a key physical property that can strongly influence the mechanical performance and durability of the concrete products. It also affects how the materials are likely to be used, whether as a coarse, fine or filler aggregate or a cement component. Grinding is typically required for the material to be in a reactive form for use as a cementitious component, and various levels of sieving may be needed for use as a sand replacement.

Particle size distribution curves for MIBA and SSA samples in as-produced form and subjected to varying levels of processing are presented in Figure 4.1 (a) and (b), respectively. Overall sand grading limits and typical curves for coal fly ash and Portland cement are also given as reference points. Dealing first with MIBA, the material remaining after incineration contains particles up to 100mm in size, consisting of metallics, ceramics, stones, glass and unburnt organics. However, the oversized particles (ranging from 20-50mm upwards) makes up only a small proportion of the total residue and are removed as part of the standard screening, thus leaving the MIBA grading curves shown in blue in Figure 4.1 (a). The screened MIBA falls on the coarser side of the fine aggregate grading limits, consisting of a close to even split between sand- and gravel-sized particles, with a low silt fraction. Further sieving has been undertaken for use as fine aggregate (shown in green), involving the removal of the gravel fraction.

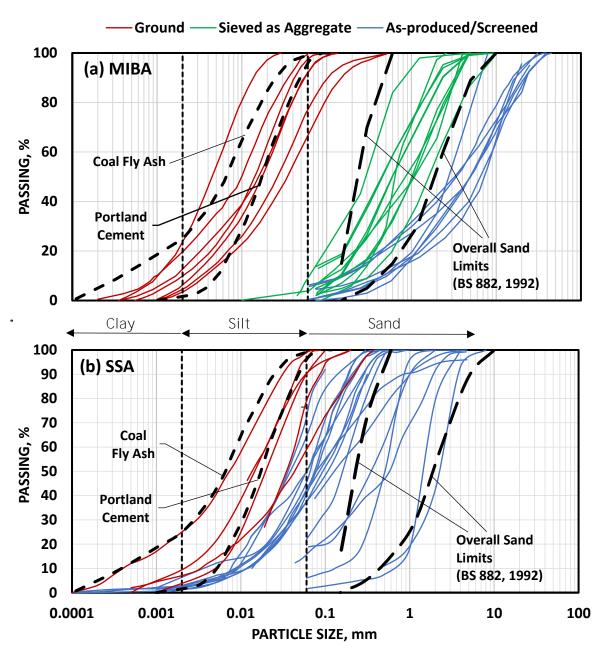


Figure 4.1: Particle size distribution curves of unprocessed/screened, sieved and ground (a)

MIBA and (b) SSA samples

Data from: MIBA - Afrani et al. (2001); Bavar et al. (2014); Bequart et al. (2009); Berg and Neal (1998a); Bertolini et al. (2004); Cheng et al. (2011b); Gines et al., (2009); Hjelmar et al. (2007); Hu et al. (2010); IEABioenergy (2000); Izquierdo et al. (2011); Jones et al. (2005); Keppert et al. (2012a); Kim et al. (2015); Qiao et al. (2008a); Saccani et al. (2005); Saikia et al. (2008); Saikia et al. (2015); Siong and Cheong (2004); Song et al. (2010); Song et al. (2015); Tang et al. (2015); Tang et al. (2016); Valle-Zermeno et al. (2015); Weng et al. (2015); Yu et al. (2014). SSA - Al-Sharif and Attom (2014); Alcocel et al. (2006), Anderson and Skerratt (2003), Bhatty and Reid (1989b), Cheeseman and Virdi (2005), Coutand et al. (2006), Danish Technological Institute (2008), Donatello et al. (2010a), Donatello et al. (2010c), Environmental & Water Technology Centre of Innovation Ngee Ann Polytechnic (2012), Fontes et al. (2004), Franz (2008), Garces et al. (2008), Geyer et al. (2002), Hu et al. (2012a), Khanbilvardi and Afshari-Tork (1995), Khanbilvardi and Afshari-Tork (2002a), Kosior-Kazberuk (2011), Krejcirikova (2015), Ksepko (2014), Maozhe et al. (2013), Monzo et al. (1996), Petavratzi (2007) and Wang et al. (2005a).

The grinding of MIBA to a powder form is a prerequisite for its use as a cementitious component. It is evident from the red curves in Figure 4.1 (a) that these samples achieved well graded distributions comparable to coal fly ash and Portland cement. The hardness of MIBA and the associated grinding energy requirements will influence the economic viability of its use as a cementitious component. Although no data on the material hardness was available, the metals that are present in MIBA in significant quantities tend to have high hardness values. However, ferrous and non-ferrous removal treatments are regularly implemented, as the recovered metals are valuable commodities in their own right. There would be extra incentive to adopt these treatments to improve its grindability, as well as to address environmental concerns, when using the ash as a cement component.

It is evident from Figure 4.1 (b) that the as-produced SSA samples are considerably finer than their MIBA counterparts and consisted largely of silt and fine sand size fractions. The Figure also provides a clear picture of the variability in the SSA grading, which can have important implications for the use of samples around the world as construction materials. At times, SSA can be naturally suitable for use as a filler or fine aggregate in concrete applications. Grinding is required for its use as a cementitious component and these SSA samples achieved well-graded particle size distributions comparable to coal fly ash and Portland cement. Details of the material's grindability are lacking; however, it is apparent from Figure 4.1 that the ground MIBA samples tended to be finer than their SSA counterparts, perhaps suggesting that the former material was easier to grind.

Additional Blaine and BET specific surface area tests provide a more convenient measure of particle size as a single value. Results for SSA samples are given in Table 4.1, along with typical values for coal fly ash and Portland cement.

Large variation is evident in the SSA fineness results and these values often exceeded those of coal fly ash and Portland cement, despite the fact that SSA had coarser particle size distribution curves. The unexpected high fineness values for SSA may be attributable to its irregularly shaped particles, rough particle surface texture and high porosity (see Section

4.2.3). As such, the comparison with coal fly ash in particular, which consists of spherically shaped particles, does not provide the most representative measure of the relative fineness of the materials. Additional tests on the effect of grinding time (10-360 min) on the fineness of SSA showed that the Blaine value increased from $500 \text{ m}^2/\text{kg}$ after 10 min grinding up to close to $1000 \text{ m}^2/\text{kg}$ after 60 min and did not increase further after longer grinding times. The BET fineness showed no reliable increase with longer grinding times, as the increase in the outer surface area due to particles splitting, had little impact on the overall pore surface area, owing to the abundance of inner pores in SSA particles. These results again indicate that the Blaine and BET fineness may not be the most suitable measures to use with SSA. D_{10} , D_{50} and D_{90} values can perhaps be used as an alternative simple representation of SSA particle sizes.

Table 4.1: Data on the fineness of SSA (adapted from Lynn et al., 2015)

NAATEDIAI	FINENESSS RESULTS							
MATERIAL		Blaine, m ² /kg	BET, m ² /g					
<u>SSA</u>	No. of Samples	7	8					
	Range	160-640	5.4-23.8					
	Average	335	10.8					
Reference Mate	rials							
Coal fly ash		380	2					
Portland Cemen	it	340	-					

Data from: Alcocel et al. (2006), Baeza et al. (2014), Cyr et al. (2007b), Cyr et al. (2012), Donatello and Cheeseman (2013), Environmental & Water Technology Centre of Innovation Ngee Ann Polytechnic (2008), Fernandez Ferreras et al. (2010), Gil-Lalaguna et al. (2015), Halliday et al. (2012), Merino et al. (2005), Paya et al. (2002), Perez-Carrion et al. (2013), Sasaoka et al. (2006), Sato et al. (2013).

4.2.2 Density

Density is an important characteristic to consider in the transportation and batching of the incinerated ashes and is also a controlling factor regarding its use in lightweight applications such as aerated concrete and foamed concrete. Specific gravity results are presented in Figure 4.2 (a) and (b) for MIBA and SSA, respectively, with the values separated based on how the materials were processed. For MIBA samples with no details given on their processing, it was assumed that standard screening was most likely, whilst for SSA, the screening step is not needed and, as such, the unspecified treatment and as-produced results were grouped together.

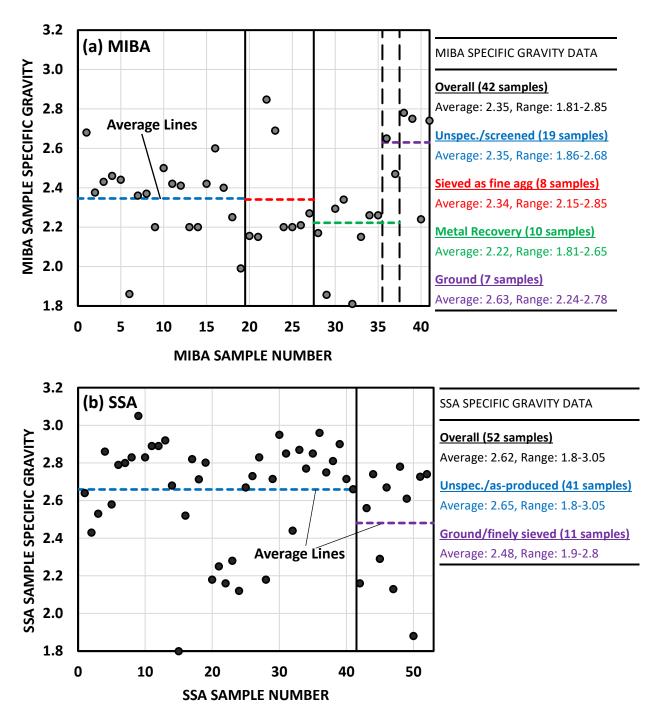


Figure 4.2: Density of (a) MIBA and (b) SSA subjected to varying levels of processing Unspec. – Unspecified

Data from: MIBA – Al Muhit et al. (2015), Alhassan and Tanko (2012), An et al. (2014), Becquart and Abriak (2013), Becquart et al. (2009), Chen et al. (2008), Cheng (2012), Deepak and Ramesh (2015), Dhir et al. (2011), Filipponi et al. (2003), Goni et al. (2007), Jones et al. (2005), Keulen et al. (2016), Kim et al. (2014), Kruger et al. (2012), Kuo et al. (2015), Mohamedzein and Al-Aghbari (2012), Mohamedzein et al. (2006), Monteiro et al. (2008), Patil et al. (2016), Qiao et al. (2009), Roessler et al. (2016), Song et al. (2010), Song et al. (2015), Tang et al. (2014), Tang et al. (2016), Van der Wegen et al. (2013), Vu et al. (2012), Wang et al. (2016), Weng et al. (2015), Yang et al. (2014c), Yao et al. (2014),

Yu et al. (2014). SSA – Barton (2007), Coutand et al. (2006), Cyr et al. (2007a), Daylan and Beulah (2014), De Lima et al. (2015), Donatello et al. (2010b), Donatello et al. (2010d), Forth et al. (2006), Geyer et al. (2002), Halliday (2008), Halliday et al. (2012), Jamshidi et al. (2012), Kjersgaard (2007), Kosior-Kazberuk (2011), Lin et al. (2014a), Luo et al. (2009), Merino et al. (2007), NCHRP (2013), Pade and Jakobsen (2007), Pan et al. (2003b); Pinarli and Kaymal (1994), Sasaoka et al. (2006); Sato et al. (2013), Tay and Show (1992a), Tenza-Abril et al. (2014), Tseng and Pan (2000), Vouk et al. (2015), Wang et al. (2005b), Weng (2002), Wiebusch and Siefried (1997), Yip and Tay (1990).

It was found that MIBA had the lower density of the two ashes, with an overall average specific gravity of 2.35, compared to 2.62 for SSA. The density of MIBA falls in between the typical values of 2.15 for furnace bottom ash produced in coal-fired power stations (Torii and Kawamura, 1991) and 2.65 for natural sand (Jackson and Dhir, 1996), whilst the SSA value is close to the sand value. Both materials exhibited quite a high degree of variability in the results; however, for MIBA the distribution of densities above and below the average line appears quite even, whereas for SSA there appears to be two groups of data, with the bulk of the samples fitting in the upper end of the 1.8–3.05 range established.

It is evident from Figure 4.2 (a) that the metal recovery treatment led to a decrease in the density, due to the removal of the denser ferrous and non-ferrous metals such as iron, aluminium, lead and copper. The opposite behaviour of increasing density was observed for the ground MIBA samples, owing to their more compact particles. The average specific gravity of 2.63 indicates that ground MIBA is much denser than coal fly ash (typical specific gravity of 2.3, though still well below the 3.15 value for Portland cement) (Jackson and Dhir, 1996). In contrast, the average value for the ground SSA unexpectedly worked out to be lower than that of the as-produced ash. The reason for this behaviour is not clear, though it may have been caused by variability in the original sewage sludge generation and treatment that led to a number of lower density ash samples.

The incineration conditions have also been identified as an important influencing factor on the ash density. Temperatures upwards of 800°C are generally used for MSW incineration, but the specific conditions used for the tested MIBA samples were not generally specified. However, incineration temperatures were more readily available for the SSA samples and as

such, the relationship between the temperature and the ash specific gravity was examined and is shown in Figure 4.3.

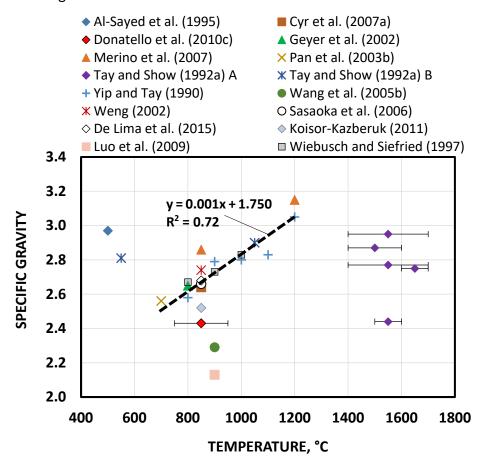


Figure 4.3: Relationship between the density of SSA and the incineration temperature

Based on the total data, the correlation between temperature and density is quite low. However, the majority of SS incineration plants operate within 700°C–1200°C and when considering the appropriate SSA results in this range, a trend of a linear increase in density with increasing temperature is noticeable, increasing at a rate close to 5% per 100°C, from 2.5 at 700°C to 3.1 at 1200°C. The increasing densification can be attributed to the decrease in the pore spaces within the particles and the fusing together of the contained metal components, as the vitrification process develops (Yip and Tay, 1990); however, it is expected that this would continue only up to a certain point, after which large isolated pores would begin to form, leading to density reductions. At the upper end, SSA samples approach the heavyweight aggregate category and owing to the denser microstructure, would be likely to possess greater mechanical strength for use in concrete applications.

Bulk density tests, which take into account the air voids, revealed that MIBA exhibited values from 1000 to 1600 kg/m³, with an average value of 1291 kg/m³, whilst SSA values ranged from 436 to 1545 kg/m³, with an average value of 906 kg/m³. Similar to the particle density results, the SSA results appear to be bimodally distributed and split into two groups, one varying from around 400 to 700 kg/m³ and the other from 1300 to 1500 kg/m³. The cause of this is unclear, though it is possibly related to differences in the original sewage sludge generation and treatment. The low ratios of particle density to bulk density for the two incinerated ashes are indicative of porous microstructures.

4.2.3 Morphology

Considering MIBA and SSA as potential concrete materials, performance aspects such as strength, durability and workability will all be influenced somewhat by their morphology. Scanning electron microscope imaging has been used to examine the morphology of MIBA and SSA samples worldwide (MIBA data from: Bayuseno, 2006; Chang and Wey, 2006; Frogner Kockum et al., 2012; Holmes et al., 2016; Hu et al., 2010; Huang et al., 2006; Inkaew et al., 2016; Izquierdo et al., 2011; Keppert et al., 2012b; Li et al., 2012; Liu et al., 2014; Tang et al., 2016; Tasneem et al., 2016; Xie et al., 2016; Yang et al., 2014b; Yao et al., 2014; Yu et al., 2013; Yu et al., 2014 and Zekkos et al., 2013. SSA data from: Alcocel et al., 2006; Anderson, 2002; CIRIA, 2004; Coutand et al., 2006; Cyr et al., 2007a; De Lima et al., 2015; Garces et al., 2008; Halliday, 2008; Kjersgaard, 2007; Kosior-Kazberuk, 2011; Monzo et al., 1996; Pade and Jakobsen, 2007; Pan et al., 2003a; Paya et al., 2002; Rink et al., 2005, Sato et al., 2012; Sato et al., 2013; Wang et al., 2005a; Weng, 2002; Weng and Pan, 2006 and Zeedan, 2010).

The two materials are produced using similar heat treatments and it was found that both consisted of irregularly shaped particles with rough surface texture. However, as the retention time in the furnace during incineration lengthened, the rough texture of the ash particles became smoother, owing to increases in surface agglomeration (Chang and Wey, 2006). Consistent with the bulk density results, MIBA and SSA were found to have porous microstructures, due to the development of gas bubbles during the heating and cooling involved in the incineration process.

The rough-textured irregularly shaped particles and the associated higher specific surface area, combined with the porous microstructure, may lead to higher water demands when using the materials, because of their greater capacity for water absorption. As such, if the ash is not accommodated appropriately, the workability of concrete products in the fresh state may be negatively affected. The ash porosity could be advantageous in certain concrete applications that require lower thermal conductivity and lightweight properties.

4.2.4 Water Absorption

The water absorption of the incinerated ashes will directly affect the water demand and consistence of concrete products, in the fresh state, along with the long term durability in the hardened state. In agreement with the previous findings on the porosity and irregular shaped ash particles, direct testing confirmed that both MIBA and SSA had high water absorption properties. The data revealed the following:

- It was determined that MIBA had an average water absorption of 9.0%, though the results varied substantially from 2.2 to 14.9%. Fine MIBA fractions had higher absorption properties compared to the coarse, due to a higher specific surface area.
- High water absorption properties were also evident for SSA, with an average value of 17.9%. The variability in the SSA results was even more pronounced and included two extreme values of 1.6% and 48%. The finer grading of SSA compared with MIBA (see Figure 4.1) also contributed to its higher absorption capacity.

Data from: MIBA - An et al. (2014); Chen et al. (2008); Dhir et al. (2011); Forth et al. (2006); Jones et al. (2005); Kim et al. (2014); Kuo et al. (2015); Pera et al. (1997); Roessler et al. (2016); Song et al. (2010); Tang et al. (2015); Van den Heede et al. (2015); Vu et al. (2012); Yang et al. (2014c); Zhang and Zhao (2014), SSA - Al-Sharif and Attom (2014); Barton (2007); Environmental & Waste Technology Centre of Innovation Ngee Ann Polytechnic (2008); Forth et al. (2006); MWCC (1990); Tay and Show (1992a); Tay and Yip (1989).

The water absorption of both MIBA and SSA is substantially higher than the typical values of 1%–3% for natural aggregates (Neville, 1995), though the two materials fall above and below the 15% value of the commercially available Lytag lightweight aggregate (Lytag Ltd, 2009). The

water demand is expected to increase when using the ashes in concrete products and this needs to be accounted for in the mix design. Ensuring that the aggregates are added in the saturated surface dry state, provided that the size of the particles allows this, should improve mixability and limit decreases in the mixture consistence. The high absorption properties also suggest that the ashes may not be the most suitable for use in concrete in highly aggressive environments.

4.2.5 Permeability

The permeability of a material is closely related to its pore structure and particle morphology. This parameter can have a notable effect on the concrete durability. From the environmental standpoint, the rainfall infiltration rate, controlled by the product permeability, will strongly affect the leaching potential of the heavy metals present in MIBA and SSA; however, this is less of a concern with concrete, because of the solidification and stabilisation effects of cement after hardening.

The extracted data on the permeability of MIBA and SSA samples are presented in Figure 4.4, plotted on a log scale. Permeability ranges for different soil types are also shown for comparison purposes (Head, 2006). The MIBA results were particularly variable, with differences of almost 6 orders of magnitude evident between the maximum and the minimum values. The permeability was extremely sensitive to changes in the moisture content, as is evident from the large spread in the results recorded by Cosentino et al. (1995b), with moisture contents varying above and below optimal conditions (corresponding to the maximum dry density). The material grading was another important influencing factor.

The permeability results for SSA were more tightly bunched, though limited in number. It is expected that the finer grading of SSA would lead to lower permeability compared to MIBA and indeed this trend is visible to a certain extent in Figure 4.4. The permeability of the SSA samples was comparable to that of similarly graded fine sands and, although with some exceptions, the results for MIBA, in many cases, were higher, ending up around the clean sand range outlined by Head (2006).

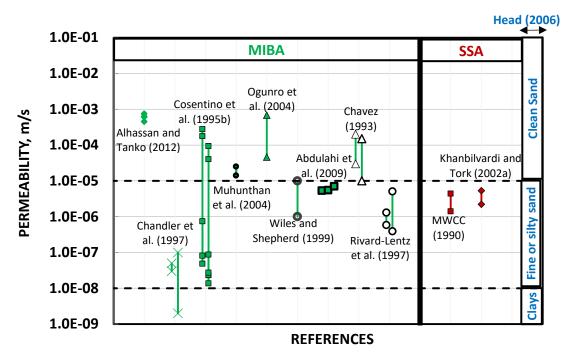


Figure 4.4: Permeability results for MIBA and SSA samples

4.3 Chemical Properties

4.3.1 Oxide Composition

The oxide composition of the ashes plays a crucial role in determining their suitability for use in a reactive form after grinding, as a cementitious component. The composition is dependent on a number of factors, including the make-up of the original municipal solid waste and sewage sludge, which has been shown in Chapter 2 to be quite variable, and the processing of the material prior to incineration, which for sewage sludge can be particularly intensive. In addition, the inclusion of additives during the incineration process can also affect the chemical make-up of the ashes. Testing of the oxide contents of the materials has been routinely undertaken and, as a result, a large collection of data from sources all across the world has been accumulated, for both MIBA and SSA. This provided a great opportunity to give the reader a clear picture of the variability in the oxide contents of the materials.

Box-and-whisker plots have been developed (Figure 4.5) showing the maximum, 75th percentile, 50th percentile (median), 25th percentile and minimum values calculated for each oxide of the MIBA and SSA samples. The faint grey dots represent the individual oxide contents

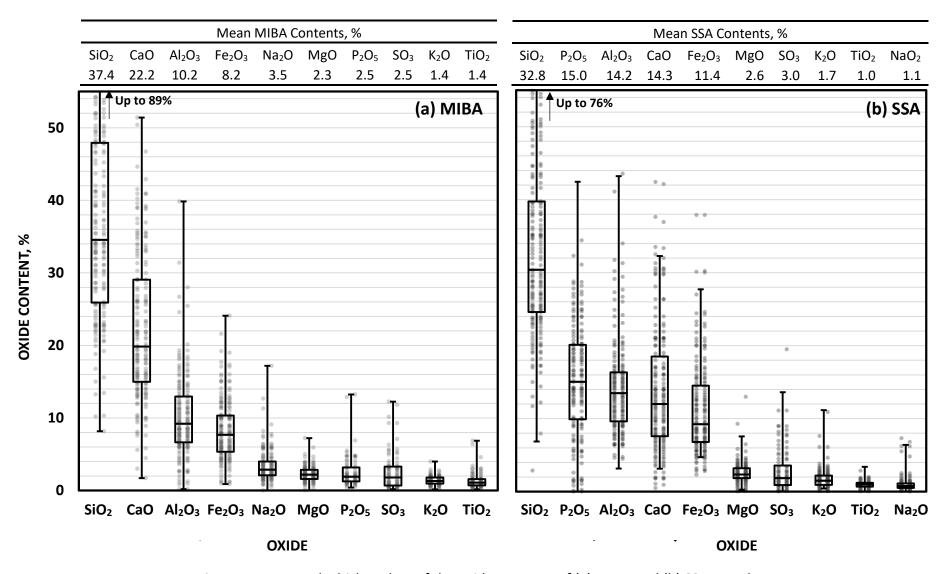


Figure 4.5: Box and whisker plots of the oxide contents of (a) MIBA and (b) SSA sample

Note: the data sources for this figure are the same as those listed for the upcoming Figure 4.6

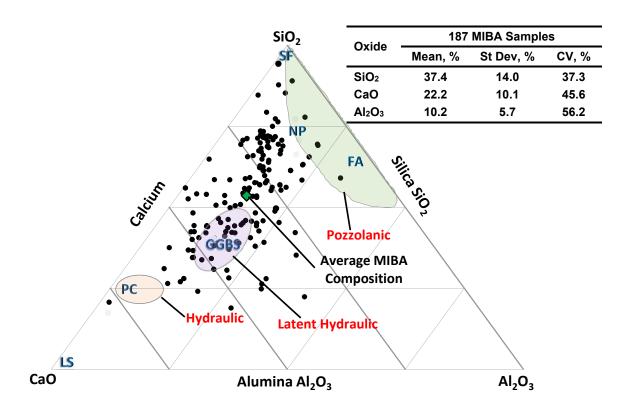
of the individual ash samples. For both ashes, SiO_2 , CaO, Al_2O_3 and Fe_2O_3 were present in large quantities, whilst, for SSA, P_2O_5 was also established as the second most abundant oxide present, behind SiO_2 . Other oxides such as Na_2O , MgO, P_2O_5 (in MIBA), SO_3 , K_2O and TiO_2 were also identified in smaller quantities in the two materials.

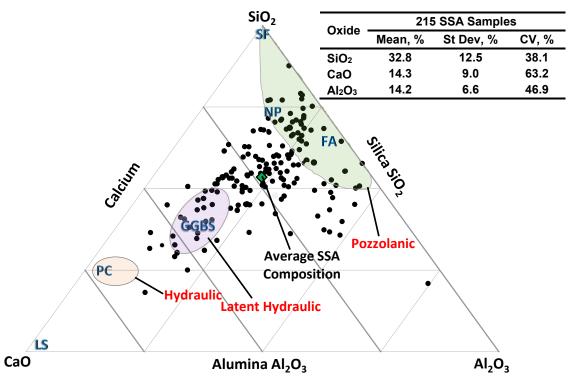
Figure 4.5 also highlights the great variability evident, particularly in the SiO_2 contents, in worldwide MIBA and SSA samples. The relative variability in the other main oxides, CaO, P_2O_5 (in SSA), Al_2O_3 and Fe_2O_3 , was also substantial. The interquartile range (25th–75th percentile range) tended to be tighter for Al_2O_3 and Fe_2O_3 , but with some extremely high outlying values. Regarding the remaining minor oxides, SO_3 stands outs as one of the more variable components in MIBA and SSA, and indeed, as is discussed later in this section, the sulphate content can have important implications for the durability of cement-based products.

It is encouraging to find that the two incinerated ashes share the same main oxides, SiO_2 , Al_2O_3 , CaO and Fe_2O_3 , as those typically found in established cementitious materials such as Portland cement, fly ash from coal fired power stations and GGBS. The materials may also offer value as part of the raw feed for cement clinker production, by contributing to the quotas of CaO, Al_2O_3 , Fe_2O_3 and SiO_2 needed to produce the desired cement clinker phases.

The contents of three of the main oxides of the ashes, SiO₂, CaO and Al₂O₃, are plotted in the form of a ternary diagram in Figure 4.6 (a) for MIBA and 4.6 (b) for SSA, to establish if the materials are likely to provide any pozzolanic activity. Each black dot represents the composition of a single MIBA or SSA sample, and the compositions of the established cement components are also marked, along with highlighted pozzolanic, latent hydraulic and hydraulic regions. The accompanying mean, standard deviation and coefficient of variation values for the three oxides are also provided.

The sum of SiO_2 , CaO and Al_2O_3 accounts for, on average, 70% and 61% of the total content of MIBA and SSA, respectively, and as such, the variability evident in Figure 4.6, provides a good representation of the overall variability of the materials. The distribution of the MIBA and SSA





Note: PC = Portland cement, GGBS = ground granulated blastfurnace slag, FA = coal fly ash, NP = natural pozzolan, SF = silica fume and LS = limestone.

Figure 4.6: Ternary diagram of the contents of the main oxides SiO_2 , Al_2O_3 and CaO found in (a) MIBA and (b) SSA samples.

Data for Figure 4.5 and 4.6 from: MIBA - Abba et al. (2014); Al-Rawas et al. (2005); Andreola et al. (2008); Aouad et al. (2008); Arvelakis and Frandsen (2005); Barberio et al. (2010); Bayuseno and Schmahl (2010); Bertolini et al. (2005); Bethanis (2007); Bourtsalas et al. (2015); Carsana et al. (2016); Chang and Wey (2006); Chen and Chiou (2007); Chen and Lin (2006); Chen et al. (2014); Chen et al. (2016); Cheng (2012); Cheng et al. (2011b); Chiang et al. (2011); Chou et al. (2008); Cioffi et al. (2011); Cobb et al. (2009); Costa et al. (2007); Dabo et al. (2009); Ecke and Aberg (2006); Erdem et al. (2011); Etoh et al. (2009); Funari et al. (2015); Garcia-Lodeiro et al. (2016); Gines et al. (2009); Gomez et al. (2009); Goni et al. (2007); Gunning et al. (2009); Heuss-Abbichler et al. (2010); Huang et al. (2006); Inkaew et al. (2016); Ito et al. (2006); Jing et al. (2013); Jing et al. (2010); Jing et al. (2007a); Jo et al. (2008); Juric et al. (2006); Keppert et al. (2012a); Kim et al. (2015); Kim et al. (2016); Kokalj et al. (2005); Lam et al. (2010a); Lam et al. (2010b); Lam et al. (2011); Lancellotti et al. (2015); Lancellotti et al. (2014); Lapa et al. (2006); Lee et al. (2014); Li et al. (2007); Li et al. (2010); Li et al. (2012); Lidelow and Lagerkvist (2007); Lin and Chang (2006); Lin and Lin (2006a); Lin et al. (2006a); Lin et al. (2008a); Lin et al. (2012); Liu et al. (2008); Liu et al. (2014); Lu and Chuang (2016); Mohamedzein and Aghbari et al. (2012); Monteiro et al. (2006); Monteiro et al. (2008); Muller and Rubner (2006); Pan et al. (2008); Pavlik et al. (2011); Pena et al. (2006); Polettini et al. (2005); Polettini et al. (2009); Qian et al. (2006); Qiao et al. (2008a); Qiao et al. (2008b); Quek et al. (2015); Rambaldi et al. (2010); Rendek et al. (2006); Rendek et al. (2007); Saccani et al. (2005); Saikia et al. (2006); Saikia et al. (2008); Saikia et al. (2015); SAMARIS (2006); Schabbach et al. (2011); Schabbach et al. (2012); Shan et al. (2012); Siddique et al. (2010a); Siong and Cheong (2004); Song et al. (2015); Song et al. (2016); Tang et al. (2014); Tang et al. (2015); Tang et al. (2016); Tasneem et al. (2016); Taurino et al. (2017); Traina et al. (2007); Valle-Zermeno et al. (2013); Valle-Zermeno et al. (2014); Valle-Zermeno et al. (2015); Vichaphund et al. (2012); Vizcarra et al. (2014); Vu et al. (2012); Vu and Forth (2014); Wang et al. (2016); Wei and Jiang (2012); Wei et al. (2011b); Weng et al. (2010); Weng et al. (2015); Whittaker et al. (2009a); Wu et al. (2016); Xie et al. (2016); Yang et al. (2013); Yang et al. (2014a); Yang et al. (2014b); Yang et al. (2016); Yao et al. (2014); Yu et al. (2014); Zhang and Zhao (2014); Zhang et al. (2015a); Zhen et al. (2013). SSA - Adam et al. (2009), Adam et al. (2013), Al Sayed et al. (1995), Alcocel et al. (2006), Al-Sharif and Attom (2014), Anderson (2002), Anderson and Skerratt (2003), Anderson et al. (2002), Baeza et al. (2014), Baeza-Brotons et al. (2014), Baeza-Brotons et al. (2015), Bhatty and Reid (1989a), Bhatty et al. (1992), Chang et al. (2010), Chen et al. (2006a), Chen et al. (2012), Chiou et al. (2006), CIRIA (2004), Coutand et al. (2006), Cyr et al. (2007a), Cyr et al. (2007b), Cyr et al. (2012), Damtoft et al. (2001), Danish Technological Institute (2007), De Lima et al. (2015), Donatello et al. (2010a), Donatello et al. (2010b), Dyer et al. (2001), Dyer et al. (2011), Eliche-Quesada et al. (2011), Elouear et al. (2010), Endo et al. (1997), Fontes et al. (2004), Franz (2008), Garces et al. (2008), Geyer et al. (2002), Haglund et al. (1997), Halliday (2008), Halliday et al. (2012), Hnat et al. (1999), Hong et al. (2005), Hu and Hu (2014), Hu et al. (2012a), Hu et al. (2012b), Huang and Li (2003), Hultmann et al. (2001), Jamshidi et al. (2011), Jamshidi et al. (2012), Khanbilvardi and Afshari-Tork (1995), Kikuchi (2001), Kjersgaard (2007), Kosior-Kazberuk (2011), Kozai et al. (2015), Ksepko (2014), Lam et al. (2010a), Latosinka (2014), Levlin (1999), Li et al (2015), Lin (2006), Lin and Lin (2004), Lin and Lin (2005), Lin and Lin (2006b), Lin et al. (2005a), Lin et al. (2005b), Lin et al. (2006b), Lin et al. (2007), Lin et al. (2008c), Lin et al. (2009a), Lin et al. (2009b), Lin et al. (2014a), Lin et al. (2014b), Mahieux et al. (2010), Maozhe et al. (2013), Merino et al. (2005), Merino et al. (2007), Monzo et al. (1996), Monzo et al. (1997), Monzo et al. (1999a), Monzo et al. (1999b), Monzo et al. (2003), Monzo et al. (2004), Morais et al. (2005), Morais et al. (2006), Morais et al. (2009), Najarjuna et al. (2015), NCHRP (2013), Nomura (1998), Ohbuchi et al. (2008), Okufuji (1990), Ozaki et al. (1997), Pade and Jakobsen (2007), Pai et al. (2010), Pan et al. (2003a), Pan et al. (2003b), Park et al. (2003), Paya et al. (2002), Perez-Carrion et al. (2013), Pinarli (2000), Pinarli and Kaymal (1994), Rapf et al. (2012), Saikia et al. (2006), Sasaoka et al. (2006), Sato et al. (2012), Schaum et al. (2011), Shih (2009), Stark et al. (2006), Suzuki et al. (1997), Takahasi et al. (1997), Takahasi et al. (2001), Takaoka et al. (1997), Takeuchi et al. (1999), Tantawy et al. (2012), Tantawy et al. (2013), Tay and Show (1991), Tay and Show (1992a), Tay and Show (1992b), Tay and Show (1994), Tenza-Abril et al. (2014), Tsai et al. (2006), Tseng and Pan (2000), US EPA (1972), Vouk et al. (2015), Wang and Chiou (2004), Wang et al. (2005a), Wang et al. (2005b), Wiebusch and Seyfried (1997), Xu et al. (2012), Yoon and Yun (2011), Zeedan (2010), Zhang et al. (2013), Zhang et al. (2015b), Zhu et al. (2015).

samples are quite similar, though the SSA results lean more towards the bottom right corner, because of their higher proportion of Al_2O_3 . Most of the MIBA and SSA samples lie around the pozzolanic and latent hydraulic regions, which suggests that the materials may offer some value when used in ground form as a cement component, potentially by contributing small amounts of CaO to the cement hydration reaction, and by reacting with calcium hydroxide to produce additional calcium silica hydrate phases.

The two incinerated ashes have higher CaO contents than the typical value of coal fly ash, though well below that of Portland cement. Al₂O₃ contents of MIBA and SSA are below that of coal fly ash, though similar to that of GGBS. For both fly ash and GGBS, their amorphous Al₂O₃ fraction is known to benefit the durability of concrete products, owing to the associated chloride binding capacity. Similar positive effects may also be evident with the incinerated ashes. On the other hand, important concerns have been raised, particularly with MIBA, regarding the reaction of the metallic aluminium fraction in the alkaline cement environment to form hydrogen gas, leading to potentially damaging expansive behaviour and ultimately cracking in the end product. This is a key issue to address with the use of these ashes in concrete-related applications. However, for the special case of aerated concrete, the expansion could be advantageous in contributing to the desired lightweight properties.

Phosphorus is present in significant quantities in both materials, though particularly in SSA. The high phosphorus content in the original SS has led to its widespread use as fertiliser on agricultural soils in the past, though this has been restricted somewhat in recent years, because of health concerns. Phosphorus is generally not present in large quantities in common cement components, though EN 450 (2012) sets out a limit of 5% for the use of coal fly ash in concrete, to avoid negative effects on the product's setting behaviour. Meeting this limit should not be a problem for MIBA, as 94% of the tested samples were within the 5% value. On the other hand, only 11% of SSA samples had P_2O_5 contents below 5%. As such, the setting behaviour is certainly a key point of interest when evaluating the use of SSA in concrete. In addition, as there is a demand for phosphorus recovery, the approach of combining phosphorus recovery treatment of SSA with its subsequent use as a cement component could be an interesting complementary strategy.

To avoid soundness problems, the sulphate content of concrete constituents must also be controlled. Limits of 3% and 5% SO₃ are specified for the use of coal fly ash in concrete in the EN 450 (2012) and ASTM C618 (2015) standards, respectively. The majority of incinerated ash samples satisfy the more lenient American criteria, as 85% of MIBA samples and 80% of SSA samples had SO₃ contents below 5%, whilst 69% of MIBA samples and 71% of SSA samples

were compliant with the more stringent 3% SO₃ limit. As such, the SO₃ content should be flagged as an important parameter to evaluate when considering these materials as cement components.

Magnesium oxide is another component found in minor amounts in both MIBA and SSA that can affect the soundness of concrete products. Average MgO contents of 2.3% and 2.6% have been calculated for MIBA and SSA, which are well below the specified limits of 5% for Portland cement in EN 197 (2011) and 4% for coal fly ash in EN 450 (2012). Indeed, 96% of the MIBA samples and 92% of the SSA samples were within the stricter 4% limit, which indicates that this parameter should generally not restrict the use of these incinerated ashes.

Alkalis present in cement components can, in the presence of moisture, react with certain aggregates and lead to the formation of a damaging expansive gel in concrete products. With mean Na₂O contents of 3.5% for MIBA and 1.1% for SSA, both materials mostly satisfy the 5% limit outlined for coal fly ash in concrete (EN 450, 2012). Indeed, the incinerated ashes may have an overall positive effect on this aspect of the concrete durability, due to the reduction in alkalinity when used as a cement replacement.

4.3.2 Loss on Ignition

Volatiles and organic matter present in the initial municipal solid waste and sewage sludge are predominantly burnt off as gases during incineration. However, there are generally small amounts of the organic fraction left over in the residual ashes. This organic matter can increase the water absorption properties and heterogeneity of MIBA and SSA, which can compromise their value as construction materials, in terms of both mechanical performance and aesthetics. Excessive organics can also interfere with the performance of admixtures that may be included in concrete mixes.

Loss on ignition (LOI) has been used to provide a measure of the organics in MIBA and SSA, by calculating the difference in mass of the sample before and after ignition. Varying ignition temperatures have been adopted when testing the materials, though they can be broadly

separated into two groups, with the first using temperatures around 550°C–600°C and the second around 950°C–1100°C. At the higher temperatures, the LOI value may slightly overestimate the organic fraction owing to the additional decomposition of the inorganic components such as calcite (Lynn et al., 2017a).

The incineration conditions, specifically the incineration temperature, residence time, turbulence and oxygen availability (Lam et al., 2010b), play a key role in determining the residual organics present in MIBA and SSA. The variability in the composition of the initial MSW and SS and post-incineration processing adopted also influence the ash organic content.

Loss on ignition results for MIBA and SSA samples have been collated and presented in ascending order in Figure 4.7. Limits for cement and coal fly ash for use in concrete from both the American and the European standards are also outlined. An analysis of the combined data for both materials is also provided in the accompanying Table 4.2.

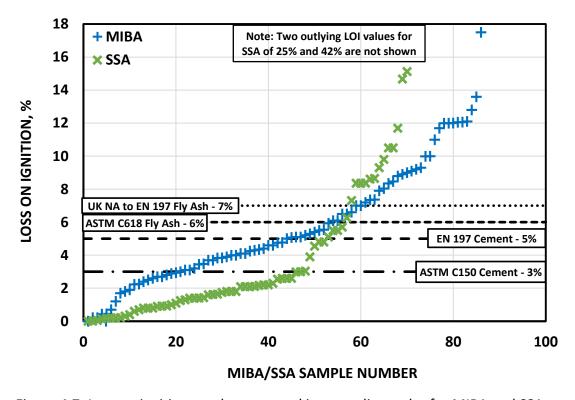


Figure 4.7: Loss on ignition results, arranged in ascending order for MIBA and SSA samples

Table 4.2: Accompanying loss on ignition data analysis for MIBA and SSA samples

PARAMETER	MIBA	SSA
Incineration Temperatures	800-1200°C	500-950°C
LOI Test Temperatures	440-1100°C	550-975°C
Number of Samples	86	72
Mean LOI	5.7%	4.3%
Median LOI	4.9%	2.1%
Standard Deviation	3.6%	6.3%
Coefficient of Variation	63%	145.0%
Samples with LOI < 5%	43/86 (50%)	52/72 (72%)
Samples with LOI < 7%	60/86 (70%)	57/72 (80%)
-	•	

Data from: MIBA - Alhassan and Tanko (2012); Arm (2000); Barberio et al. (2010); Bethanis et al. (2002); Chang et al. (2004); Cobb et al. (2009); Cosentino et al. (1995a); Dhir et al. (2011); Ecke and Aberg (2006); Filipponi et al. (2003); Garcia-Lodeiro et al. (2016); Goni et al. (2007); Heuss-Abbichler et al. (2010); Inkaew et al. (2016); Ishii et al. (2009); Izquierdo et al. (2001); Jing et al. (2010); Jo et al. (2008); Lam et al. (2011); Lancellotti et al. (2013); Lancellotti et al. (2014); Li et al. (2007); Li et al. (2012); Li et al. (2016); Lin et al. (2006a); Monteiro et al. (2006); Muller and Rubner (2006); Pena et al. (2006); Qiao et al. (2008c); Rocca et al. (2012); Roethel (2007); Saikia et al. (2008); Saikia et al. (2015); Santos et al. (2013); Schabbach et al. (2011); Siddique (2010b); Tang et al. (2016); Taurino et al. (2017); Todorovic and Ecke (2006); Todorovic et al. (2006); Traina et al. (2007); Valle-Zermeno et al. (2015); Van der Wegen et al. (2013); Vu and Forth (2014); Vu et al. (2012); Wang et al. (2016); Yang et al. (2013); Yang et al. (2014b); Yang et al. (2016); Yao et al. (2010); Yao et al. (2013); Yin et al. (2007); Zhang and Shimaoka (2007). SSA - Al Sayed et al. (1995), Alcocel et al. (2006), Al-Sharif and Attom (2014), Anderson et al. (2002), Baeza et al. (2014), Bhatty et al. (1992), BRE (2007), Cheeseman and Virdi (2005), Cheeseman et al. (2003), Coutand et al. (2006), Cyr et al. (2012), Damtoft et al. (2001), De Lima et al. (2015), Donatello et al. (2010a), Donatello et al. (2010b), Donatello et al. (2010d), Eliche-Quesada et al. (2011), Federal Highway Administration (1997), Franz (2008), Garces et al. (2008), Guedes et al. (2014), Halliday (2008), Hu and Hu (2014), Hu et al. (2012a), Hu et al. (2012b), Kamon et al. (2001), Kikuchi (2001), Kjersgaard (2007), Kosior-Kazberuk (2011), Kruger et al. (2014), Ksepko (2014), Lam et al. (2010a), Latosinka (2014), Lin (2006), Lin and Lin (2005), Lin and Lin (2006b), Mahieux et al. (2010), Merino et al. (2005), Merino et al. (2007), Monzo et al. (1996), Monzo et al. (1997), Monzo et al. (1999b), Monzo et al. (2003), NCHRP (2013), Ottosen et al. (2013), Ottosen et al. (2014), Pade and Jakobsen (2007), Park et al. (2003), Paya et al. (2002), Perez-Carrion et al. (2013), Petavratzi (2007), Pinarli and Kaymal (1994), Saikia et al. (2006), Sato et al. (2012), Takeuchi et al. (1999), Tantawy et al. (2012), Tantawy et al. (2013), Tay and Show (1991), Tay and Show (1992a), Tay and Show (1992b), Tay and Show (1994), US EPA (1972), Wang et al. (2005a), Zeedan (2010).

The wide variability in both incineration ashes is immediately evident, ranging from essentially 0% up to 15% and 18% for SSA and MIBA respectively, in Figure 4.7. There were two additional outlying LOI values of 24% and 42% measured for SSA samples produced with inadequately low incineration temperatures of 550°C and 500°C, respectively. There is a much larger proportion of SSA samples with LOI values less than the strictest 3% for cement (ASTM C150, 2017), compared to their MIBA counterparts. However, the discrepancies (in the proportion

of samples satisfying specific limits) between the two materials gradually reduces, when judging based on the higher limits of 6% and 7% set out for coal fly ash.

The mean LOI values calculated for MIBA and SSA are both skewed, though to a greater extent for SSA, owing to the small number of very high results arising from poor combustion. Though it is dependent on which limits are applicable, it appears that the majority of the MIBA and SSA samples could be suitable for use as a cement component, based on their LOIs. However, adopting the approach of the Netherlands, which involved limiting the LOI of MIBA to a maximum of 5% at the point of incineration (LAP, 2009), close to complete use of the two incinerated ashes can be targeted.

The effects of incineration temperature, LOI test conditions and material processing were difficult to quantify, owing to the scarcity of background information available. However, the following points are presented:

- Furnace type no discernible trend in LOI values for MIBA and SSA samples produced
 in a grate furnace, circulating fluidised bed or rotary kiln.
- Incineration conditions temperatures of 500°C and 550°C were not sufficient for thorough combustion and the resultant SSA samples had extremely high LOI values and would be unsuitable for use.
- LOI test temperature though often not specified, samples tested with a higher ignition temperature tended to exhibit higher LOI values, as expected, due to the additional calcite decomposition. For example, using the same MIBA sample, LOI values of 2.4% and 5.0% were reported with ignition temperatures of 550°C and 970°C, respectively (Traina et al., 2007).
- Variation with time MIBA samples were collected across three seasons from autumn to spring, from four incineration plants in Sweden (Arm, 2000). The LOI results were quite consistent at the individual plants, varying from 3.2% to 4.3%, 2.8% to 4.0%, 3.8% to 5.0% and 6.5% to 9.2%, respectively, over that time period.
- Variation with size fraction MIBA samples have, in some cases, been split into a number of size fractions (Yang et al., 2014b; Müller and Rubner, 2006; Xie et al., 2016).

The LOI mostly increased as the particle size decreased, suggesting that most of the organic matter resides in the finer fractions.

4.3.3 Mineralogy

Mineralogy plays a key role in the potential reactivity of the incineration ashes, in their ground form, as cement components. The leaching behaviour of MIBA and SSA, and the associated environmental impacts arising from their use in concrete-related applications, will also be influenced by the mineral composition.

X-ray diffraction analysis has been used to identify the minerals present in MIBA and SSA. Both materials undergo similar heat treatments and as such, share similarities in their mineralogy. It has been found that quartz is the most abundant mineral in both ashes. Calcite and hematite were also frequently present in both MIBA and SSA, and in addition, magnetite and gehlenite were commonly present, specifically in MIBA. The ashes shares some similarities with other cement components in this regard, as these same minerals are commonly found in those established materials.

A large number of other less commonly identified silicates, aluminates, iron oxides, iron phosphates, calcium silicates, calcium phosphates, sulphates, salts, aluminosilicates and aluminium phosphates have been identified in the two materials. A list of these minerals is given in Table 4.3.

The mineralogy of MIBA undergoes significant changes when the material is first exposed to outside conditions. The alkalinity of MIBA reacts with the CO₂ in the air, which results in the formation of carbonates, mainly calcite. Along with the hydration and biodegradation reactions that take place at the same time in the material, this leads to the development of more stable minerals, reduces the pH and lowers the mobility of certain constituents. This improves both the mechanical stability and the leaching properties of MIBA. As such, ageing can be a very useful treatment to adopt in preparing the ash for use as a construction material.

Table 4.3: Additional minerals identified in MIBA and SSA

In Both	MIBA (Only	SS	A Only
Albite	Akermanite	Laumontite	Berlinite	Mica
Anhydrite	Apatite	Magnesite	Brushite	Microcline
Anorthite	Barite	Mayenite	Caulinite	Moganite
Corundum	Biotite	Mellite	Dolomite,	Mulite
Ettringite	Chromite	Olivine	Fe ₃ PO ₇	Muscovite
Forsterite	Dicalcium silicate	Plagioclase	Gibbsite,	Picromerite
Gibbsite	Diopside	Portlandite	Goethite	Rhodochrosite
Goethite	Graphite	Pyroxene	Gypsum	Rodolicoite
Gypsum	Halite	Sanidine	Illite	Rutile
Lime	Hardystonite	Statlingite	Kalsilite	Talc
Sylvite	Hydrocalumite	Tobermorite	Maghemite	Tridymite
Wollastonite	Hydrotalcite	Wairakite	Margerite	Whitlockite
	Larnite		Metakaolinite	Wyllieite

Data from: MIBA - Astrup et al., 2016; Baciocchi et al., 2010; Bayuseno and Schmahl, 2010; Bourtsalas et al., 2015; Chang and Wey, 2006; Chen and Lin, 2006; Chiang et al., 2011; Chiang et al., 2013; Chiang et al., 2014; Cornelis et al., 2012; Dabo et al., 2009; Dijkstra et al., 2008; Etoh et al., 2009; Flehoc et al., 2006; Garcia-Lodeiro et al., 2016; Goni et al., 2007; Gori et al., 2011; Gori et al., 2013; Gunning et al., 2009; Gunning et al., 2010; IEABioenergy, 2000; Inkaew et al., 2016; Ito et al., 2006; Jing et al., 2010; Jing et al., 2013; Kim et al., 2015; Kim et al. 2016; Lancellotti et al., 2013; Liu et al., 2014; Monteiro et al., 2006; Monteiro et al., 2008; Müller and Rubner, 2006; Onori et al., 2011; Paine et al., 2002; Pena et al., 2006; Qiao et al., 2008a; Qiao et al., 2008b; Qiao et al., 2008c; Qiao et al., 2009; Rambaldi et al., 2010; Rigo et al., 2009; Rocca et al., 2012; Saffarzadeh et al., 2011; Saikia et al., 2008; Saikia et al., 2015; Santos et al., 2013; Schabbach et al., 2012; Shan et al., 2012; Song et al., 2015; Song et al., 2016; Su et al., 2013; Tang et al., 2014; Tang et al., 2015; Tang et al., 2016; Tasneem et al., 2016; Tyrer, 2013; Um et al., 2013; Vegas et al., 2008; Vichaphund et al., 2012; Wang et al., 2016; Wei et al., 2011a; Wei et al., 2011b; Wei and Jiang, 2012; Yang et al., 2013; Yang et al., 2014b; Yang et al., 2016; Yao et al., 2014; Yao et al., 2015, Yu et al., 2013; Yu et al., 2014; Zhang et al., 2015a; Zhen et al., 2012; . SSA - Adam et al., 2009; Al-Sharif and Attom, 2014; Anderson, 2002; Anderson and Skerratt, 2003; Anderson et al., 2002; Atienza-Martinez et al., 2014; Bapat, 2013; Cheeseman and Virdi, 2005; Cheeseman et al., 2003; Coutand et al., 2006; Cyr et al., 2004; Cyr et al., 2007a; Cyr et al., 2007b; Cyr et al., 2012; De Lima et al., 2015; Dhir et al., 2002; Donatello and Cheeseman, 2013; Donatello et al., 2010b; Donatello et al., 2010c; Donatello et al., 2010d; Dyer et al., 2001; Dyer et al., 2011; Eliche-Quesada et al., 2011; Elouear et al., 2010; Environmental & Water Technology Centre of Innovation Ngee Ann Polytechnic, 2012; Fontes et al., 2004; Franz, 2008; Garces et al., 2008; Geyer et al., 2002; Gunning et al., 2009; Halliday et al., 2012; Hu and Hu, 2014; Hu et al., 2012b; Jamshidi et al., 2011; Jo et al., 2007; Johnson, 2003; Kjersgaard, 2007; Kosior-Kazberuk, 2011; Ksepko, 2014; Latosinka, 2014; Latosinka and Gawdzik, 2012; Lin, 2006; Lin et al., 2006b; Lin et al., 2008b; Lin et al., 2008c; Lin et al., 2009a; Mahieux et al., 2010; Merino et al., 2005; Merino et al., 2007; Morais et al., 2009; Najarjuna et al., 2015; Nanzer et al., 2014; Nowak et al., 2013; Ohbuchi et al., 2008; Okufuji, 1990; Ottosen et al., 2013; Ottosen et al., 2014; Pade and Jakobsen, 2007; Pazos et al., 2010; Peplinski et al., 2011; Peplinski et al., 2013; Peplinski et al., 2014; Perez-Carrion et al., 2013; Petavratzi, 2007; Petzet et al., 2012; Saikia et al., 2006; Suzuki et al., 1997; Tantawy et al., 2012; Tantawy et al., 2013; Tsai et al., 2006; Tseng and Pan, 2000; Vogel et al., 2010; Wang et al., 2012; Wzorek et al., 2006; Yoon and Yun, 2011; Yoshida and Noike, 2004 and Zhang et al., 2013.

In addition to the crystalline phases, the amorphous fraction of the incineration ashes is a key parameter to consider, as it provides an indication of their potential reactivity, when used in ground form as a cement component. Data on the amorphous contents of MIBA and SSA have been limited; however, the available results are described in Table 4.4. Both MIBA and SSA contain significant amorphous fractions. These glassy phases are formed during the high-temperature treatment of incineration and the subsequent cooling process. The accompanying crystalline phases become embedded within this glass melt matrix.

Table 4.4: Description of the information on the glass contents of MIBA and SSA

PUBLICATION	DESCRIPTION
(a) MIBA	
Bayuseno and Schmahl (2010)	Incineration temperature 850-1000°C. MIBA subsequently quenched. Amorphous phases: Fresh MIBA - 34 wt. %, Aged MIBA - 31 wt. %.
Chiang et al. (2011)	Incineration temperature: > 1500°C. MIBA subjected to rapid quenching. MIBA described as characteristically amorphous, though no content given.
Dijkstra et al. (2008)	Incineration temperature not given. Freshly quenched MIBA. Amorphous Al and Fe (hydro) oxides are identified, but no contents given.
Etoh et al. (2009)	Incineration temperature > 850°C. Quenched MIBA. Amorphous phases were recognisable but no content given.
Lancellotti et al. (2013)	Incineration temperature or processing was not specified. The amorphous content comprised the main phases of the material.
Inkaew et al. (2016)	Incineration temperature between 850-1100°C. Quenched MIBA Amorphous solids, such as C-S-H phases, visible on the particle surfaces.
Monteiro et al. (2008)	MIBA was pre-treated, with no further related details given. Glass is present but in lower quantities than crystalline minerals.
Paine et al. (2002)	Processed MIBA, with no further related details given. Amorphous phase > 67% in MIBA.
Wei et al. (2011b)	Incineration temperature: > 850°C. Freshly quenched. 4 tests: MIBA amorphous contents of 54, 48, 54 and 51%.
(b) SSA	
Anderson et al. (2002)	SSA from fluidised bed incinerator. No details on temperature or processing. Amorphous phases of approximately 65%.
Anderson and Skerratt (2003)	Incineration temperature not specified. No treatment processes details given. High amorphous phases of approximately 65%.
Bapat (2013)	No details on incineration temperature or the processing treatment of SSA. Amorphous phases of 40-74%.
Coutand et al. (2006)	Incineration temperature not specified. No treatment processes details given. Glassy phases of around 40%.
Zhang et al. (2015b)	Incinerated at 900°C and ground. Greater than 70% glass-formers and intermediates.

Comparable amorphous contents ranging from 31% to 67% for MIBA and 40% to 74% for SSA have been measured. The quenching process, in particular, has an important effect on the glass content of the ashes, though it is difficult to precisely quantify this impact because of the limited data. A comparison of fresh and aged MIBA samples indicated that the ageing process and the associated reactions did not significantly change the amorphous content. Overall, the data suggest that there is some level of reactivity in the materials and, when combined with the previous encouraging findings on the oxide composition, the ashes may be suitable for use as an alternative cement component. Additional tests of pozzolanic activity, such as the strength activity index of mortar and concrete mixes incorporating MIBA and SSA, can be useful in establishing a more conclusive judgement about their reactivity.

4.3.4 Element Composition

The acceptance of MIBA and SSA as construction materials will be predicated on ensuring that there are no detrimental environmental impacts. Their use carries a positive environmental effect due to the associated savings in landfill space and reduced consumption of natural resources. However, the primary environmental concern centres around the potential release of harmful heavy metals in MIBA/SSA into nearby sensitive receptors. Certain heavy metals and salts can also interfere with the strength, durability and aesthetics of concrete products.

Data have been extensively extracted from a large number of MIBA and SSA samples from around the world. An analysis of the total element composition of the two materials has been undertaken and the results are presented in Table 4.5. The elements were arranged in descending order, based on the mean contents of MIBA. It should be noted that many of the researchers focused solely on the more toxic elements such as Zn, Cu, Pb, Cr and Ni and, as a result, there ended up being a higher number of sample results for these elements.

Si, Ca, Fe and Al were the most abundant elements present in the two incinerated ashes, whilst P was also found in large quantities in SSA only. The contents of the more toxic elements such as As, Cd, Cr, Cu, Ni, Pb and Zn are of greater consequence from the environmental standpoint;

however, as these elements are generally not present in traditional construction materials, the relevant standards have generally not yet developed to consider these leaching issues.

Table 4.5: Analysis of the element composition of MIBA and SSA samples

MIBA			SSA			LAGA			
ELEMENT		Mean,		CV, %	No. of	Mean,	St Dev,	CV, %	LIMITS,
	Samples	mg/kg	mg/kg		Samples	mg/kg	mg/kg	01,70	mg/kg
Si	13	210893	64046	30	20	114497	48202	42	-
Ca	33	118510	59160	50	41	84628	41060	49	-
Fe	38	54081	35753	66	48	79578	55333	70	-
Al	36	43435	15841	36	45	48253	27668	57	-
Na	25	21924	16777	77	29	9170	17000	185	-
Mg	31	14454	8606	60	37	12975	5664	44	-
Cl	37	8944	9443	106	42	1087	2634	242	-
K	31	8043	4630	58	34	11765	15476	132	-
Ti	12	6632	5553	84	14	2772	2873	104	-
Р	11	4775	3794	79	46	68359	35202	51	-
Zn	82	3976	2925	74	103	2964	3257	110	10000
Cu	80	2980	2768	93	117	1673	2713	162	7000
Pb	77	1575	1207	77	115	321	402	125	6000
Ва	32	1290	904	70	27	1663	1174	71	-
Mn	43	917	585	64	36	1481	1512	102	-
Cr	81	390	322	83	106	477	928	195	2000
Sr	18	385	176	46	12	441	173	39	-
Sn	10	257	316	123	16	124	133	108	-
Sb	19	245	695	283	11	33	24	73	-
Ni	62	175	131	74	96	198	325	164	500
V	23	161	281	174	30	135	129	96	-
Zr	9	128	165	129	12	421	327	78	-
Se	10	61	111	183	11	57	154	270	-
As	49	51	62	121	47	30	52	173	-
Co	25	50	102	203	17	137	172	126	-
Mo	21	27	26	98	36	29	30	101	-
Cd	54	14	22	161	84	17	57	328	20
Ag	7	9	5	62	12	192	189	98	-
Hg	19	1	4	295	44	2.2	3.3	148	-

St Dev – Standard deviation, CV – Coefficient of variation

Data from: MIBA - Aouad et al. (2008); Astrup and Christensen (2005); Baruzzo et al. (2006); Bayard et al. (2009); Bayuseno and Schmahl (2010); Bethanis (2007); Boni et al. (2007); Chang et al. (2009); Chen and Chou (2007); Cioffi et al. (2011); Cornelis et al. (2012); Cossu et al. (2012); Dabo et al. (2009); Dhir et al. (2011); Ecke and Aberg (2006); Flyhammer and Bendz (2006); Garcia-Lodeiro et al. (2016); Goni et al. (2007); Gori et al. (2011); Gunning et al. (2010); Heuss-Abbichler et al. (2010); Hyks et al. (2009); Inkaew et al. (2016); Ito et al. (2006); Izquierdo et al. (2008); Jung et al. (2006); Juric et al. (2006); Kumpiene et al. (2011); Lam et al. (2010b); Lancellotti et al. (2013); Lapa et al. (2006); Li et al. (2010); Lidelow and Lagerkvist (2007); Lin and Chen (2006); Lin et al. (2008a); Lind et al. (2008); Lo and Liao (2007); Marchese and Genon (2009); Ore et al. (2007); Pena et al. (2006); Phongphiphat et al. (2011); Polettini et al. (2009); Puma et al. (2013); Qiao et al. (2008c); Quek et al. (2015); Rambaldi et al. (2010); Rigo et al. (2009); Rocca et al. (2012); Roethel (2007); Rogbeck and Hartlen (1996); Rosende et al. (2008); Saffarzadeh et al. (2011); Saikia et al. (2008); Saikia et al. (2015); Siddique (2010b); Sivula et al. (2012); Sorlini et al. (2011); Sormunen et al. (2016); Tang et al. (2015); Tang et al. (2016); Toller (2008); Toraldo and Saponaro (2015); Traina et al. (2007); Travar et al. (2009); Wei et al. (2011b); Wu et al. (2016); Xiao et al. (2008); Xie et al. (2016); Yao et al. (2012); Zhang and Shimaoka (2007). SSA - Adam et al. (2009), Adam et al. (2012), Al-Sharif and Attom (2014), Anderson and Skerratt (2003), Biatowiec et al. (2014), Biswas (2009), Cenni et al. (2001), Chang et al. (2010), Cheeseman and Virdi (2005), Cheeseman et al. (2003), Chen and Lin (2009a), Chen and Lin (2009b), Chen et al. (2006a), CIRIA (2004), Cohen (2009), Coutand et al. (2006), Cyr et al. (2007a), Cyr et al. (2007b), Damtoft et al. (2001), Danish Technological Institute (2007), Danish Technological Institute (2008), Dayalan and Beulah (2014), Donatello et al. (2010b), Ebbers et al. (2015), Elouear et al. (2010), Federal Highway Administration (1997), Fraissler et al. (2009), Franz (2008), Fraser and Lum (1983), Furr et al. (1979), Gil-Lalaguna et al. (2015), Gorazda et al. (2013), Guedes et al. (2014), Gulbranden et al. (1978), Halliday (2008), Halliday et al. (2012), Havukainen et al. (2012), Jamshidi et al. (2012), Johnson (2003), Kakumazaki et al. (2014), Khanbilvardi and Afshari-Tork (1995), Kosior-Kazberuk (2011), Kruger and Adam (2015), Kruger et al. (2014), Kruger et al. (2015), Lapa et al. (2007), Latosinka (2014), Latosinka and Gawdzik (2012), Li et al. (2015), Lin and Lin (2004), Lin and Lin (2005), Lin and Lin (2006b), Lin et al. (2005a), Lin et al. (2005b), Lin et al. (2005c), Lin et al. (2009a), Mattenberger et al. (2008), Mattenberger et al. (2010), Morais et al. (2005), Morais et al. (2006), Morais et al. (2009), NCHRP (2013), Nowak et al. (2011), Nowak et al. (2012), Nowak et al. (2013), Ohbuchi et al. (2008), Ottosen et al. (2013), Ottosen et al. (2014), Pade and Jakobsen (2007), Pan et al. (2003a), Pan et al. (2003b), Paramasivam et al. (2005), Pazos et al. (2010), Peplinski et al. (2011), Peplinski et al. (2013), Peplinski et al. (2014), Petzet et al. (2011), Petzet et al. (2012), Rapf et al. (2012), Saikia et al. (2006), Sasaoka et al. (2006), Schaum et al. (2011), Stemann et al. (2015), Sturm et al. (2010), Takaoka et al. (2008), Tateda et al. (1997), Tay and Show (1992a), Tay and Show (1997), Tempest and Pando (2013), Tsai et al. (2006), US EPA (1972), Van de Velden et al. (2008), Vogel and Adam (2011), Vogel et al. (2010), Vogel et al. (2013), Vogel et al. (2014), Vogel et al. (2015a), Vogel et al. (2015b), Vouk et al. (2015), Weigand et al. (2013), Xu et al. (2012), Yip and Tay (1990), Zhang et al. (2001), Zhang et al. (2002a), Zhang et al. (2002b), Zhang et al. (2013), Zhang et al. (2015b).

Landfilling is one area in which the heavy metals in the incoming wastes are of primary concern. As ancillary legislation to EU landfill Directive 99/31/EC (European Community, 1999), Council Decision 2003/33/EC (European Community, 2003) established classification limits for inert, non-hazardous and hazardous waste materials. These limits, however, are based on testing the leached element concentrations, rather than the total contents in the incoming landfill materials. The total contents, by themselves, do not provide an accurate prediction of the leached contents, as only a fraction of the totals will be available for leaching, depending on factors such as pH, complexing components, mineralogical reactions and rainfall infiltration rate (Lynn et al., 2016a). However, the German Länderarbeitsgemeinschaft Abfall document (LAGA, 1994), on the valorisation of residues, gives target total element contents

limits prior to the more extensive leaching assessment. These LAGA guidance limits are given in Table 4.5 to provide some useful context for the MIBA and SSA results. It is encouraging that the mean contents of each of the elements in MIBA and SSA were below these limits, though this is only an initial check and, as such, should be interpreted cautiously.

Chlorides present in constituent materials can compromise their suitability for use in concrete. Mean chloride contents of 0.9% and 0.1% have been established for MIBA and SSA, respectively. The SSA chloride value is right at the 0.1% limit set for cement and coal fly ash in EN 197 (2011) and EN 450 (2012), respectively. However, the mean SSA content was skewed by a small number of very high values and, indeed, 80% of the tested SSA samples had chloride contents below 0.1%. As such, SSA should be mostly suitable for use, in this regard, though washing treatment may be required at times. With MIBA, treatment would be required for most samples to reduce the chloride content to acceptable levels, particularly in application types such as pre-stressed concrete, where the limits are much stricter than that of reinforced concrete.

The presence of reactive metallic aluminium, specifically in MIBA, has been highlighted as a crucial issue that can negatively affect its potential for use in concrete products. This metallic fraction reacts under the alkaline conditions in cement, resulting in the formation of aluminium hydroxide and expansive hydrogen gas. These expansive reactions may continue in the concrete beyond the fresh state and into the hardened state and, as such, lead to damaging spalling behaviour that can severely compromise all aspects of performance. It is difficult to directly measure the reactive metallic aluminium fraction in the incinerated ashes, though it can be established indirectly by assessing the expansion evident in the resultant concrete products. The effects of the expansive behaviour are examined in greater detail in Chapter 5 on the use of MIBA in concrete; however, it appears that processing to either remove the metallic aluminium or dissipate the expansive reactions before use may be required. Aerated concrete is the one exception in which expansive behaviour can actually be beneficial in promoting the desired lightweight properties.

Revisiting the element composition data in Table 4.5, there is a high degree of variability in the element contents found in both MIBA and SSA samples around the world, with coefficients of variation frequently greater than 100%. Factors that contribute to this variability include differences in the waste and wastewater composition, variable treatment systems, nature of the incineration conditions and additives used and different test methods used to measure the element contents.

It was of interest to examine the changeability of the element composition when dealing solely with samples from a single country. Italy was one of the countries for which data were available for a large number of MIBA samples and, as such, a further analysis of the element contents for Italian samples is presented in Table 4.6. The previous data for MIBA from Table 4.5 are also given in parentheses to allow convenient comparison of the variability of Italian samples to worldwide samples.

Table 4.6: Element variability of Italian MIBA samples (previous data for worldwide MIBA samples given in parentheses for comparison)

ELEMENT	NO. OF MIBA SAMPLES	MEAN, mg/kg	ST DEV, mg/kg	CV, %
Fe	15	45285 (54081)	38432 (35753)	85 (66)
Al	14	38733 (43435)	16200 (15841)	42 (36)
Zn	20	3639 (3976)	4328 (2925)	119 (74)
Cu	20	2917 (2980)	1864 (2768)	64 (93)
Pb	19	1899 (1575)	1274 (1207)	67 (77)
Ва	10	1217 (1290)	1383 (904)	114 (70)
V	3	305(161)	448 (281)	147 (174)
Cr	19	375 (390)	422 (322)	113 (83)
Ni	14	154 (175)	102 (131)	66 (74)
Со	3	90 (50)	102 (102)	113 (203)
As	8	38 (51)	46 (62)	121 (121)
Cd	13	14 (14)	25 (22)	179 (161)

ST DEV – Standard deviation, CV – Coefficient of variation

Notwithstanding the smaller sample size, the Italian results exhibited a similar high variability compared with the worldwide samples. It is expected that the MSW composition would be

somewhat more controlled within a single country; however, despite this, the changeability remained high. Dealing with variability is standard with most types of construction materials, though with these incinerated ashes in particular, close monitoring of their characteristics is essential. In the future, it is expected that as the use of the ashes continues to develop, there will greater incentive to reduce their variability, to maximise their value as reliable construction materials.

The element composition of MIBA and SSA is also prone to change over time. As an example, the contents of cadmium, lead and mercury in MIBA samples produced in two plants in the United States have been monitored, one for 4 years between 2004 and 2007 and the other for 13 years between 1995 and 2007 (Aucott et al., 2009). The trend of increasing cadmium contents, with time, at both plants, was evident, which was attributed to the increase in nickel—cadmium batteries and other electronic devices. However, the contents of lead measured showed no significant change, whilst mercury levels showed a decline over the time periods at both facilities.

Variations in the element content per size fraction have also been examined. In most cases, the element contents were higher in the finer particle fractions, with greater contents of lead, chromium, antimony, chlorine and calcium. Magnesium, copper and zinc were more evenly spread across the various size fractions, whilst arsenic tended to be more abundant in the coarser fraction (Baciocchi et al., 2010; Bendz et al., 2007; Etoh et al., 2009; Lin and Yeh, 2010; Liu et al., 2008; Onori et al., 2011 and Sorlini et al., 2011).

In addition to the aforementioned primary elements, further constituents have also been found, specifically in some SSA samples. Platinum group elements, including platinum, palladium, rhodium, iridium, osmium and ruthenium, and rare earth elements, including cerium, lanthanum, scandium, neodymium and samarium, have been identified. The presence of these elements in SSA is dependent on factors such as the surrounding population size, the number of vehicles on the road and nearby industrial sources.

As these elements are present only in minute quantities in SSA, they should not present a pressing environmental concern when using the ash in construction. However, many of these elements are particularly valuable commodities, owing to the combination of low supply in certain locations and high economic importance associated with their uses and the value that this adds to the economy. It is very rare to find a source that can provide such a wide array of valuable components and as such, the potential recovery of these elements from SSA, along with the aforementioned primary elements, could offer many benefits, not only economical, but also from the social and environmental standpoint.

4.3.5 Organic Compounds

Organic compounds such as polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), polychlorinated biphenyls (PCB) and polycyclic aromatic hydrocarbons (PAHs) can form during the combustion process involved in the incineration of MSW and SS. As these compounds are highly toxic and classified as human carcinogens, it is important to examine if they are present in the ashes at a level that could lead to possible health and safety risks if the materials are used in construction.

The exact specifics involved in the formation of PCDDs and PCDFs (PCDD/Fs) have not been fully established. However, generally the temperatures involved in waste incineration are well above the 200°C–600°C that is most conducive to the formation of dioxins (Dhir et al., 2005) and it seems that in well-regulated incineration plants, the emission levels of these compounds should not lead to substantial negative environmental impacts. The introduction of EU Directives and national legislation to control the levels of dioxins and furans released into the environment has led to reductions in the concentrations of these compounds in the past 20 years. The group of 2,3,7,8-Tetrachlorodibenzodioxins (TCDDs) are the most highly toxic congeners and generally, the measure of the PCDD/Fs is expressed in the form of a single number, as a total concentration of congeners in terms of an international toxic equivalency (I-TEQ).

The regulatory limits for these compounds mostly apply to the allowable concentrations in the emissions from an incineration plant, or the tolerable intake levels for humans and animals. However, from the point of view of evaluating the use of incinerated ashes as construction materials, specifically in concrete applications, some of the content limits outlined in the research, which are presented in Table 4.7, can provide a useful comparison to the contents found in MIBA and SSA samples.

Table 4.7: PCDD/F concentration limits outlined in the literature

REGULATORY LIMITS	PCDD/F CONTENTS ng I-TEQ/kg
Dhir et al. (2005)	
UK Government suggested limits for use in construction products/materials	50
Roethel (2007)	
American Centers for Disease Control and Prevention recommended limit for residential soil	1000
Yang et al. (2012)	
Taiwan Environmental Protection Administration regulatory limits for MIBA	Type I < 100
in reused products	Type II < 100
	Type III < 1000

Results on the PCDD/F contents in MIBA and SSA samples from around the world are presented in Table 4.8. The formation of these compounds is given more attention during the incineration of MSW; however, these organic pollutants are also found in SSA, though to a lesser extent. The fluidised bed furnace type, which is commonly adopted in the incineration of sewage sludge, has been reported to have a high removal efficiency of PCDD/Fs and PCBs (Dhir et al., 2005). Nevertheless, Table 4.8 shows that the dioxins and furans in both incinerated ashes are low and, indeed, compared to the concentration limits outlined previously in Table 4.7, all the results are within the strictest limit of 50 ng I-TEQ/kg. This is encouraging for the use of these materials in concrete applications and suggests that the presence of these dioxins and furans would not lead to negative environmental impacts.

Table 4.8: PCDD/F contents in (a) MIBA and (b) SSA

DESCRIPTION OF INCINERATED ASH SAMPLE	PCDD/F CONTENT, ng I-TEQ/k
(a) MIBA	
Badreddine and Francois (2009)	
Sampled from a trial of MIBA in a road in France	32.6
Chen et al. (2006b)	
MIBA from a southern Taiwan incinerator	5.2
MIBA from a northern Taiwan incinerator	1.6
Dhir et al. (2005)	
UK incinerators (Dyke et al., 1997)	5.0
Average worldwide data (Environment Australia, 2002)	30.0
UK Incinerators (Environmental Agency, 2002)	0.6-23.0
German Incinerators, (Danish EPA, 1997)	5.0-50.0
UK Incinerators (Eduljee et al., 1996)	20.0
Dhir et al. (2011)	
Typical MIBA sample in UK	< 10.0
Roethel (2007)	
Senoko Incinerator in Singapore	4.5
Tuas Incinerator in Singapore	4.5
Tuas South Incinerator in Singapore	6.6
Zhang et al. (2012)	
MIBA from an incinerator in southern China	12.94
(b) SSA	
Halliday (2002)	
UK samples	0.3
	1.2
	3.0
	3.1
Danish EPA (1997)	
Danish sample	6.3

PCBs and PAHs are closely related to the dioxins and furans and can potentially pose a similar threat to the health of humans and animals. However, the data available on these organic compounds are very limited and indeed, results were obtainable only for MIBA and not SSA. It was found that:

- PAH contents ranging from 44 to 6884 μg/kg have been reported for MIBA samples from China, Sweden, Italy and France (Liu et al., 2008 and Shen et al., 2010).
- PCB contents from 0.62 to 1.31 μg/kg have been measured in MIBA samples from China and Japan (Shen et al., 2010).

No accompanying limits for PAHs and PCBs were given in the literature, and without guidance regarding the allowable contents for construction materials or concrete products, it is difficult to pass judgement on the incinerated ashes. The materials have not been flagged as particularly harmful in this regard and the contents of the other dioxins and furans present were at levels similar to those of established cementitious materials produced during thermal processes, such as coal fly ash and cement kiln dust. It has been established that increasing the combustion efficiency, for which the fluidised bed combustor was favoured over the grate furnace system, and lowering the plastic contents and other chlorine sources in the wastes incinerated, are two methods to reduce the PAH and PCB concentrations in the ashes (Shen et al., 2010).

4.4 Summary

This chapter has assessed the characteristics of MIBA and SSA, dealing with both their physical properties (particle size distribution, density, morphology, water absorption and permeability) and their chemical properties (oxide composition, loss on ignition, mineralogy, element composition and organic compounds).

In its as-produced form after incineration, MIBA is a coarse material, consisting of metallics, ceramics, stones, glass fragments and unburnt organic matter, with particles up to 100 mm in size. The oversized fraction (particles greater than 20/30/40 mm) of MIBA makes up only a small proportion of the full sample and is typically removed straightaway as part of the standard screening process. Conversely, SSA is a more fine-grained material, containing mainly silt- and fine sand-sized fractions. Varying levels of sieving would be needed for the use of MIBA as an aggregate in concrete, whilst the grading of SSA suggests natural suitability as a

filler or fine aggregate. Both materials require grinding for use as a cement component in concrete-related applications.

The density of MIBA, with an average specific gravity of 2.35, falls in between the typical values of 2.15 for furnace bottom ash and 2.65 for natural sand, whilst SSA, with a higher average value of 2.62, is marginally below the natural sand value. The density of both varies somewhat depending on the incineration temperature and the processing treatments applied. The two materials exhibit low bulk density-to-particle density ratios, which is suggestive of porous materials. This was confirmed by scanning electron microscope imaging, which revealed that the materials contain irregular, angularly shaped, rough-textured particles, formed during the heating and cooling that take place during incineration. The porous microstructure and higher specific surface area of the incinerated ashes contribute to high water absorption properties, which are substantially higher than natural sand, though on a level comparable to commercial lightweight aggregates. The permeabilities of MIBA and SSA, though sensitive to grading and moisture conditions, are comparable to those of similarly graded soils.

Regarding the chemical properties, SiO₂, CaO, Al₂O₃ and Fe₂O₃ are present in large quantities in both ashes, whilst in addition, in SSA only, P₂O₅ is the second most abundant oxide. Na₂O, MgO, P₂O₅ (just MIBA), SO₃, K₂O and TiO₂ were also identified in smaller quantities in the two materials. The contents of their main oxides suggest that MIBA and SSA may have potential, in ground form, for use as cement components or as part of the raw feed in cement clinker production. Minor residual organic fractions are present in the two materials. Based on LOI results, the majority of the MIBA and SSA samples could be suitable for use as cement components, though a thorough burn would be needed to limit the organics and facilitate widespread use of the materials. Quartz, calcite and hematite are the main minerals present in both MIBA and SSA, and in addition, magnetite and gehlenite are frequently present in the former. When exposed to outside conditions, the mineralogy of MIBA undergoes significant change, due to the carbonation and hydration reactions that take place, leading to a reduction in the pH and the conversion towards a more stable mineral species. Amorphous phases are

present in both MIBA and SSA, suggesting that the materials may be somewhat reactive when used in ground form as a cement component in concrete products.

The presence of heavy metals such As, Cd, Cu, Cr, Ni, Pb and Zn in MIBA and SSA raises some environmental concerns, though encouragingly, the average element contents in both were below the initial German target limits for construction materials. The chloride content of SSA indicates that most samples would meet the specified limits for cement components; however, the contents for MIBA suggest that a treatment such as washing may be required to reduce the chlorides to an acceptable level to avoid durability problems in reinforced concrete. Expansive behaviour arising from the reaction of the metallic aluminium in MIBA under the alkaline conditions in cement has been highlighted as a crucial issue that may demand treatment, when using the material in concrete applications. Organic pollutants formed during the waste combustion process are found in MIBA and SSA, though the dioxin and furan contents of both materials were within outlined limits for construction materials.

CHAPTER 5

USE OF MUNICIPAL INCINERATED BOTTOM ASH IN CONCRETE

5.1 Introduction

Concrete producers have, in the past, relied on natural sources to produce the constituent aggregate and cement. However, nowadays increasing importance is given to sustainability in construction, calling for conservation of natural resources, reduction in CO₂ emissions and sustainable management of waste materials. This has prompted changes in the materials considered for use in concrete-related applications, more towards recycled and secondary sources. Ever increasing levies on the extraction of aggregates and landfilling also continue to favour this transition. In addition, standards and specifications have evolved to be more accommodating to secondary materials: EN 197 (2011) recognises GGBS, coal fly ash and silica fume as permissible cementitious constituents, whilst EN 12620 (2002) permits the use of aggregates from both manufactured and recycled sources in concrete.

As established in Chapter 4, MIBA is one such secondary material that appears to be potentially suitable for use as aggregate and cement components in concrete. Use of MIBA in road pavements and geotechnics are areas that are much more developed and indeed these applications have been studied extensively in earlier parts of this project, which was published in peer-reviewed journals, based on data from over 300 studies from 29 countries worldwide, dating back to 1975 (Lynn et al., 2016a and Lynn et al., 2017a). However, the more challenging prospect of using MIBA in concrete-related applications, as shown in this Chapter, can offer a higher value outlet for the material, particularly as a cementitious component.

The adoption of MIBA as aggregate is covered first in this chapter, followed by its use as part of the raw feed in cement clinker production and, after grinding, as a cement component. Environmental impacts associated with MIBA-concrete products are also dealt with, along with the case studies.

5.2 Use as Aggregate

5.2.1 Mortar and Concrete

Mortar plays an important role in the construction industry, commonly acting as rendering and bonding elements that hold together masonry units. Compared with concrete, mortar products share many similarities, though are generally weaker, are less durable and have a shorter life span. This make the use of MIBA in mortars a more accessible outlet, though concrete offers a much larger market. Concrete mixes have lower cement ratios and contain an additional coarse aggregate fraction, though the two products share many similarities in how they behave, and as such, mortar and concrete mixes containing MIBA are analysed together in this section.

This work aims to develop the use of MIBA as partial and complete fine and coarse aggregate components. As is evident from the particle size distribution curves shown previously in Figure 4.1, screening and sieving are required for the use of the ash as aggregate and this requirement should be taken care of by the supplier of the material. Given the nature of the as-produced material, the need for further treatments of MIBA before its use in mortar and concrete has also been recognised. This consisted of:

- Ferrous and non-ferrous metal removal (Almeida and Lopes, 1998; Dhir et al., 2002; Ferraris et al., 2009; Tang et al., 2015);
- Washing (Dhir et al., 2002; Erdem et al., 2011; Kuo et al, 2015; Rashid and Frantz, 1992;
 Saikia et al., 2008, Saikia et al., 2015; Sorlini et al., 2011; Tay, 1988; Van den Heede et al.,
 2015; Van der Wegen et al., 2013; Zhang and Zhao, 2014);
- Chemical treatment (Kim et al., 2015; Pera et al., 1997; Saikia et al., 2015);
- Thermal treatment (Ferraris et al., 2009; Saikia et al., 2015).

Examining the fresh properties, flow results showing the effects of MIBA as a sand replacement on the mortar consistence are presented in Table 5.1, along with accompanying slump results in Figure 5.1 (a) for the ash as a sand replacement and 5.1 (b) for the ash as a gravel replacement. As established in Chapter 4, the absorption of MIBA is significantly higher than those of natural sand and gravel, with an average value close to 10%. As such, MIBA is

expected to increase the mix water demand. However, this should not greatly compromise the consistence, if the mix is designed appropriately to allow for the higher ash absorption. Alternatively, water-reducing admixtures can be incorporated to compensate for the increased water demand of the MIBA mixes.

Table 5.1: Consistence results for mortars using MIBA as a fine aggregate replacement

MIX		RESULTS			
Series 1	Water, kg/m ³	³ Cement, kg/m ³	Sand, kg/m ³	MIBA, kg/m ³	Flow, mm
Control	283	514	1545	-	131
10% sand replaced	283	514	1390	155	119
20% sand replaced	283	514	1236	309	120
30% sand replaced	283	514	1081	464	119
40% sand replaced	283	514	927	618	101
Series 2	Water, g	Cement, g	Sand, g	MIBA, g	Flow, %
Control	196	300	1685	-	4.6
100% sand replaced (UWt MIBA)	*284	300	-	1049	5.4
100% sand replaced (Wt MIBA)	*288	300	-	1088	6.6

^{*}Includes extra water added to allow for the absorption of the MIBA aggregates UW_t – unwashed, W_t – washed.

Data from: Cheng (2012) and Rashid and Frantz (1992).

For the eight mortar and concrete mix series produced, w/c ratios ranging from 0.42 to 0.86 were adopted, whilst different cement types and contents and various MIBA treatments were also used. The flow and slump results in Table 5.1 and Figure 5.1 indicate a slight overall downward trend in the consistence of the mortar and concrete, as the MIBA replacement level was increased. However, for practical purposes, the values were generally quite close to those of the mixes using natural aggregate, for MIBA contents up to 50%. Some increases in the slumps were evident in Figure 5.1 with lower MIBA contents, though given the physical properties of the ash relative to natural aggregate, this cannot be attributable to MIBA directly, but rather to adjustments in the mix water content.

Although extra water was added to allow for the higher absorption of the MIBA, its irregular particle shape can cause a further increase in the water demand which was not allowed for

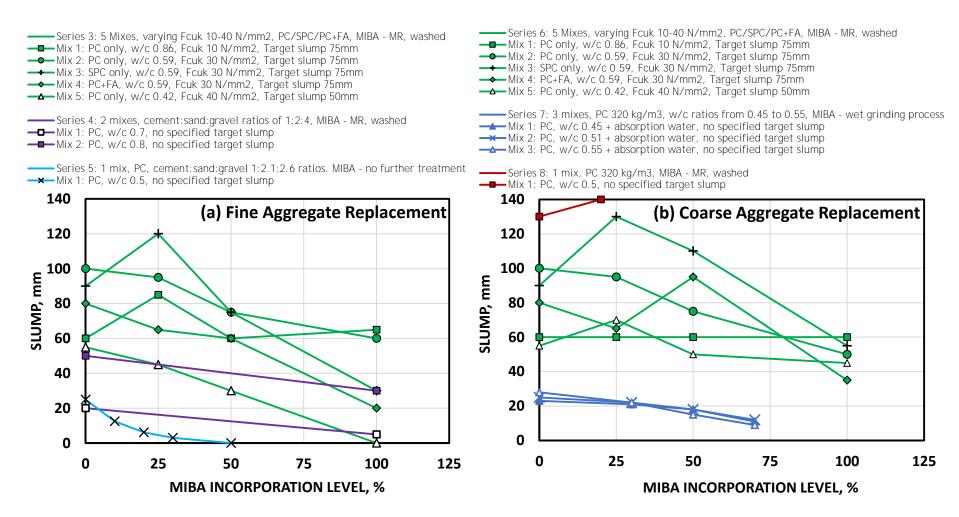


Figure 5.1: Effect of MIBA on concrete consistence (workability) as (a) fine aggregate and (b) coarse aggregate replacement Data from: Al Muhit et al. (2015); Dhir et al. (2002); Tay (1988); Van der Wegen et al. (2013); Zhang and Zhao (2014)

PC - Portland cement, SPC – sulphate resistant cement, FA – coal fly ash, MR – metal removal (ferrous, non-ferrous), F_{cuk} - target concrete cube strength at 28 days.

in some cases and manifested itself in lower flow and slump values. These decreases became more significant with high MIBA contents and also when using the material as fine aggregate compared with as coarse aggregate, owing to the higher specific surface area of the fine particles. Changes in the cement type, the water/cement ratio or the MIBA treatment, did not significantly alter the effect of the ash on the mix consistence. Reductions in consistence that incurred with MIBA were generally quite gradual, with the exception of Series 5, which exhibited sharp slump losses at low MIBA contents. This can likely be attributable to overlooking the extra water needed to bring the MIBA aggregate to the required saturated surface dry condition prior to its use.

Based on the flow and slump data, it would be advisable to limit the MIBA replacement to contents up to 50%. As the ash is a new material in concrete applications, it would be practical to initially ease it in at lower contents, in any case. Indeed, ready-mixed concrete producers often incorporate a recipe of primary and various secondary aggregates into the mix design, depending on the locally available resources. In regions with a scarcity of suitable natural sands and gravels, producers can turn to other options such as crushed-rock coarse and fine aggregate and quarry fines as full or partial replacements of natural fine aggregate. These materials can be usable, though as with every aggregate, adjustments are required to accommodate their specific characteristics. In the case of MIBA some reductions in the consistence can be expected. However, within the recommended replacement level, slumps should be within 20% of the established natural aggregate mixes, and for practical purposes, bearing in mind the impreciseness of the slump test, this level of performance should be manageable, when accounted for by producers.

Data on additional fresh properties of mortar and concrete mixes incorporating MIBA as fine and coarse aggregate components, are presented in Figure 5.2, covering fresh unit weight, setting behaviour and bleeding, for replacement levels from 0% to 40%. Reductions in the fresh unit weight are evident, and are attributed to the lower ash density (average specific gravity of 2.35 established in Chapter 4) compared to the replaced sand (typical specific gravity 2.65).

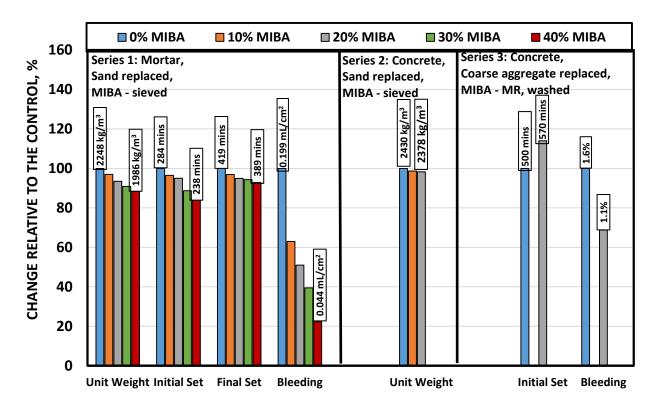


Figure 5.2: Effects of MIBA as aggregate on other fresh properties of mortar/concrete MR – metal removal (ferrous and non-ferrous)

Data from: Al Muhit et al. (2015), Cheng (2012), Van den Wegen et al. (2013)

Mix Series 1 and 3 experienced contrasting shortening and lengthening setting behaviour, respectively, whilst a further mix Series (Dhir et al., 2002), though with no numerical, recorded no change in the time to set with MIBA as complete fine or coarse aggregate replacements in concrete. The contents of phosphorus and soluble lead and zinc in MIBA could potentially explain the retardation, though this effect was evident in only one mix series. The shortened setting time was curiously attributed by Cheng (2012) to a faster hydration reaction of lime and the higher C₃A content in MIBA, though this claim appears to be questionable.

Bleeding was reduced with MIBA (Figure 5.2). Similar to previous slump and flow results, this is attributed to the irregular particle shape of the ash and the associated increased water retention. As such, concrete producers should be aware that the improved bleeding associated with MIBA can equate to greater cohesiveness and stability in mixes. There was one exception, however, in which segregation issues were visible with MIBA as a sand replacement in concrete. This could be explained by the complete loss of workability evident

from corresponding slump results for this mix [Series 5 in Figure 5.1 (a)], indicating an unsuitable mix design.

In the hardened state, the mix compressive strength performance is a key aspect to examine. Based on the material characteristics, the initial indications are that further treatment of MIBA, beyond standard sieving, may be required in some cases to deliver suitable mechanical strength. However, to first establish the baseline behaviour, compressive strength results for mortar and concrete mixes (at 28 days) using standard processed MIBA as a fine aggregate replacement are presented in Figure 5.3, as a percentage of the control natural aggregate mix strength.

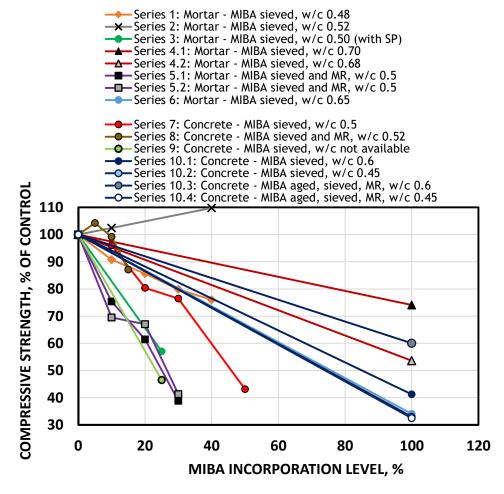


Figure 5.3: Effects of standard processed MIBA on 28-day compressive strength as fine aggregate in mortar/concrete

SP – superplasticiser. Data from: Al Muhit et al. (2015); Cheng (2012); Dhir et al. (2002); Keppert et al. (2012a); Pavlik et al. (2011/2012), Rashid and Frantz (1992); Saikia et al. (2015), Tang et al. (2015); Yang et al. (2014c).

It is evident that mixes using MIBA as a fine aggregate exhibited lower strength, with the one exception of Series 2, compared to the conventional natural aggregate products. Strength decreases, to some degree, are anticipated when incorporating recycled and secondary aggregates and should not be overly concerning. The increase in Series 2 may have been caused by a positive filling effect, but it appears unlikely that this improvement could be achieved consistently with the use of the ash. Though the control results serve as a useful benchmark, the aim with all MIBA mixes should not be to outperform natural aggregate mixes, but instead to show that the material can be usable, as there is ample scope to include these types of materials in many less demanding applications. Having said that, there is significant variation in the losses incurred with the standard processed MIBA aggregate, and a number of key issues have to be managed when using the material in these products. The potentially problematic issues are:

- Metallic aluminium expansion
- High organic matter
- Inhibited hydration reactions

Examining further the 10 mix series in Figure 5.3, damaging expansive behaviour that leads to cracking and compromises the overall performance, has been identified in four of them (Series 3, 6, 7 and 10) using the standard processed MIBA. This expansion is caused by the reaction of the metallic aluminium in MIBA under the alkaline conditions in the cement matrix, to form hydrogen gas. The degree of reactivity increases with increasing MIBA fineness, amongst other factors, and as such, this expansion is expected to be more prominent when using the ash as a cement component, though nevertheless, it has also occurred at times, as a fine aggregate.

High contents of organics negatively affect the structural integrity and homogeneity of MIBA aggregates and, therefore, the resultant concrete strength. Though it was established previously in Chapter 4 that MIBA had a median LOI of around 5%, some samples have significantly higher values. Indeed, in Series 3 in Figure 5.3, the high LOI value of 12% of the MIBA sample contributed to lower strength performance. Ideally, samples with lower organics should be initially selected for use; nonetheless, in this case, further treatment to minimise the organic matter would have a favourable effect on the concrete compressive strength.

The higher water absorption of MIBA must be accounted for in the mix design, including its irregular particle shape, which also contributes to a higher water demand. Although the amount of water needed for the hydration reactions to develop in concrete is low, without an appropriate mix design to accommodate the ash, the soaking up of water may potentially negatively affect the cement hydration and the placing and compaction process, thus inhibiting strength development. For example, Series 5 (Figure 5.3) exhibited low strength, despite no visible cracking due to hydrogen gas expansion, and this was attributed to the absorption of MIBA inhibiting the cement hydration reactions. This problem can be overcome by allowing for the extra water absorbed by MIBA, or by using a water-reducing admixture.

Based on the aforementioned concerns, further treatment of MIBA beyond sieving, consisting of washing, chemical or thermal treatment, has been adopted in some cases, prior to its use. Data showing comparisons between the 28-day compressive strengths of mortar and concrete mixes using MIBA samples before and after subjecting them to further treatment are presented in Table 5.2.

It is evident that washing, chemical and thermal treatment upgraded the strength of the MIBA products. Washing was somewhat effective in improving the strength, though it did not completely eradicate the metallic aluminium expansion behaviour in mixes that experienced these reactions (Series 3 and 6). The benefits from washing also were lessened if the ash had previously been subjected to ferrous and non-ferrous metal removal processing. Chemical treatment of MIBA with NaOH or Na₂CO can be adopted to convert the reactive metallic aluminium into a stable aluminate and prevent damaging expansion. However, the presence of a high amount of organics (MIBA used in Series 3 had an LOI of 12%) inhibited the effectiveness of this treatment. As such, thermally treating the ash at 675°C to reduce the organic fraction, prior to the chemical treatment, yielded much improved compressive strength. The addition of superplasticiser to the thermal + chemically treated MIBA also led to a further large improvement in the resultant mortar strength (63% higher than the sieved MIBA mix in Series 3), owing to the lower allowable effective water/cement ratio.

Table 5.2: Effects of further processing on the compressive strength of mortar and concrete mixes containing MIBA as fine or coarse aggregate components

SERIES NO.	MIX	MIBA PROCESSING	COMP STRENGTH, (28 days) MPa
3 (Mortar)	MIBA as 25% of fine agg	Untreated	30.7
		Washed	31.2 (+2%)
		Chemical (0.1 Na ₂ CO)	31.3 (+2%)
		Chemical (0.25 Na ₂ CO)	32.6 (+6%)
		Thermal (675°c)	34.6 (+13%)
		Chemical+thermal	29.0 (-6%)
		Thermal+chemical	40.5 (+32%)
		Thermal+chemical+SP	50.0 (+63%)
6 (Mortar)	MIBA as 100% of fine agg	Unwashed	8.7
		Washed	15.9 (+83%)
10 (Concrete)	MIBA as 100% of fine agg	Unwashed	16.5
,		Washed	21.5 (+30%)
	MIBA (with MR, ageing)	Unwashed	24.0
	as 100% of fine agg	Washed	24.5 (+2%)
	MIBA as 100% of coarse	Unwashed	16.5
	agg	Washed	23.0 (+39%)
	MIBA (with MR, ageing)	Unwashed	25.5
	as 100% of coarse agg	Washed	24.0 (-6%)
11 (Concrete)	MIBA as 10% of fine agg	Unwashed	48.0
		Chemical (NaOH)	48.0 (0%)
	MIBA as 20% of fine agg	Unwashed	43.0
		Chemical (NaOH)	45.0 (+5%)
	MIBA as 30% of fine agg	Unwashed	38.0
		Chemical (NaOH)	41.5 (+9%)
	MIBA as 50% of fine agg	Unwashed	21.5
	55	Chemical (NaOH)	29.0 (+35%)

Green - strength increase, Red - strength decrease, relative to the standard processed ash mix SP – superplasticiser, MR – metal removal (ferrous and non-ferrous), agg – aggregate. Data from: Dhir et al. (2002), Kim et al. (2015); Rashid and Frantz (1992), Saikia et al. (2015)

The total compressive strength results, converted to a percentage of the control natural aggregate mix results, for mortar and concrete mixes incorporating further processed MIBA are presented in Figure 5.4 (a) for the material as a fine aggregate and 5.4 (b) as a coarse aggregate. Results with the further processed MIBA are more favourable than the previous strengths with the standard processed ash (Figure 5.3). The higher temperature thermal treatment of MIBA, at 1450°C, delivered compressive strengths comparable to those of

natural aggregate concrete mixes, though this would have to be balanced against the associated higher energy costs. In general, the most appropriate treatment plan should be selected based on any potential problems that may arise with that ash sample. If the key pitfalls (reactive metallic aluminium, high organics, absorption inhibiting hydration) are avoided, the compressive strength performance with MIBA aggregates can be manageable and the material can be usable, as a partial aggregate component, in many applications in which premium aggregates are not essential, such as in mortars and non-structural concrete.

A different approach was adopted for Series 12 [Figure 4.5 (a)] and Series 15 [Figure 4.5 (b)]. The water contents of these mixes were reduced as the MIBA incorporation level increased. This was done to target strengths equal to the control mixes, for all mixes containing MIBA, though it is evident that some of the ash-concrete mix results actually ended up exceeding this target compressive strength. Given the previously shown effects of MIBA on the mix fresh properties, this approach would certainly require the use of a superplastiser.

Based on indications from the data that the ceiling strength of the material is not a limiting factor, MIBA also shows promise for use in higher strength applications. Failure mode testing as a sand replacement showed that the cement-aggregate bond failed before the MIBA aggregates were crushed. The rate of strength loss with MIBA was similar for varying levels of target strength, as is highlighted from the results of Series 11 and 14 in Figure 5.3. Using data from an additional high-performance concrete mix (Series 23, Yu et al., 2014), it is concluded that it is possible to reach ultra-high compressive strengths around 115 MPa, using MIBA as a 20% sand replacement with additions of nano-silica, steel fibres and polypropylene fibres.

The use of MIBA as a combined fine + coarse aggregate replacement in concrete is another option. Compressive strength data for two additional mix series (Afriani et al., 2001; Van der Wegen et al., 2013), revealed that reductions on par with the cumulative decreases evident in Figure 5.4 (a) and (b), relative to the control natural aggregate mixes, are to be expected. As such, it is recommended to adopt low replacement levels when using MIBA in this manner as aggregate in concrete, at least at the initial stages.

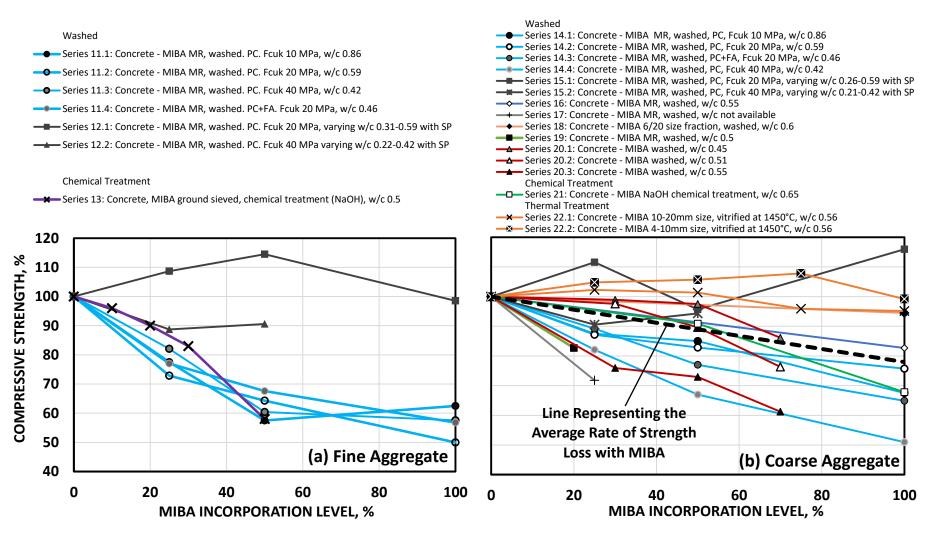


Figure 5.4: Compressive strength of mortar and concrete mixes using further processed MIBA as (a) fine aggregate and (b) coarse aggregate MR – metal removal treatment (ferrous and non-ferrous fractions), PC – Portland cement, FA – fly ash, F_{cuk} - target concrete cube strength at 28 days. Data from: Dhir et al. (2002); Erdem et al. (2011); Ferraris et al. (2009); Kim et al. (2015); Pera et al. (1997); Sorlini et al. (2011); Van der Wegen et al. (2013); Zhang and Zhao (2014).

Compressive strength is closely linked to tensile and flexural resistance. In design, flexural and tensile strength values are commonly deduced based on established relationships with the tested compressive strength. As such, it is important not only to establish the effects of MIBA on these parameters, but also to verify the relationship with the compressive strength.

Using MIBA as a partial and complete replacement of fine or coarse aggregate in mortar and concrete products, it is found that the tensile and flexural strength mirrors the compressive strength performance, with both showing decreases, compared to natural aggregate mixes, as the replacement level increases. Data on the effects of MIBA on the relationship between compressive strength and flexural and tensile strengths are presented in Figure 5.5 (a) and (b), respectively, using MIBA as a coarse or fine aggregate, as partial or complete natural aggregate replacements, for curing ages from 1 to 90 days (though mainly at 28 days). Control mixes are represented by the open markers, whilst the MIBA counterparts are shown as filled markers in the graphs.

It is evident from Figure 5.5 (a) that the flexural-to-compressive strength ratios for natural aggregate mixes and MIBA mixes were comparable, within the natural level of variability, though the latter results are shifted to the left, owing to the lower overall strength. For MIBA and non-MIBA mixes, flexural strength ranged from 9% to 29% and 11% to 26% of the compressive strengths, respectively. Indeed, the best-fit power trend lines for the two groups are very similar. As such, a further trend line for the combined results is provided, and indeed, for practical purposes, it can be concluded that MIBA does not significantly alter the flexural-to-compressive strength relationship of mortar and concrete mixes.

Similar trends are also evident on the tensile-to-compressive strength relationship, given in Figure 5.5 (b). It is of additional interest to determine if the relationship given in Eurocode 2 (BS EN 1992-1-1, 2004) between compressive and flexural strength remains valid for MIBA mixes. In representing the Eurocode 2 relationship between compressive and flexural strength, mean cylinder compressive strengths were converted to mean cube compressive strengths by interpolation, to be consistent with the mean cube results of the MIBA mixes.

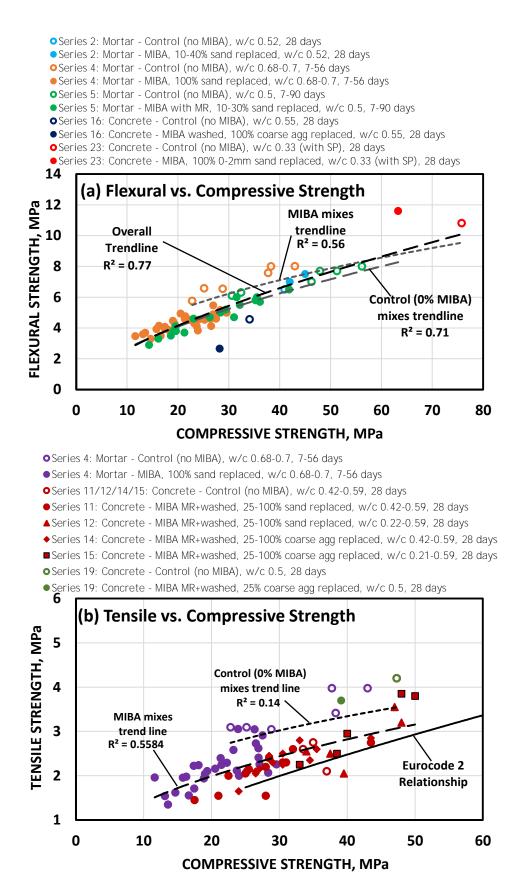


Figure 5.5: Effects of MIBA as aggregate in mortar/concrete on the relationship between compressive strength and (a) flexural strength and (b) tensile strength

SP - superplasticiser

Data from: Dhir et al. (2002); Erdem et al. (2011); Pavlik et al. (2011/2012); Tang et al. (2015); Van der Wegen et al. (2013); Yang et al. (2014c); Yu et al. (2014).

The trend lines established for both the natural aggregate and the MIBA aggregate mixes both remained on the safe side of the line representing the Eurocode 2 tensile-to-compressive strength relationship, though the MIBA line was the lower of the two groups of data. As such, this Eurocode 2 relationship can be applicable to MIBA concrete products.

The aggregate in concrete plays an important role in determining the elastic deformation properties. Eurocode 2 outlines an expected mean elastic modulus based on the concrete strength class and aggregate type. Using the same process as outlined with the tensile strength, it is of interest to establish if the Eurocode 2 relationship between compressive strength and elastic modulus remains accurate for MIBA mixes. As such, data showing the effects of MIBA as coarse or fine aggregate on the elastic modulus-compressive strength relationship of concrete mixes are presented in Figure 5.6 (a), along with Eurocode 2 curves for mixes using natural aggregates.

By comparing the control open markers to the MIBA closed markers in Figure 5.6 (a), it is clear that the elastic modulus-compressive strength ratio is lower for the MIBA mixes. This is normal for many recycled aggregate types. The higher porosity and lower density of the ash, suggests that it has a lower hardness than natural aggregate, which explains the decrease in stiffness. The level of reduction in the elastic modulus-compressive strength ratio increased as the MIBA content increased. To quantify the effect of the replacement level, the results were split into three groups: MIBA as 20% to 30%, 50% and 70% to 100% of the coarse/fine aggregate in the concrete. Trend lines were then plotted for each group, as is shown in Figure 5.6 (b). For lower replacement levels of 20%-30%, the MIBA remained within the confines of the Eurocode 2 elastic modulus-compressive strength ratio for natural aggregates. The early stages of bringing an emerging material such as MIBA into concrete mixes are likely to involve easing it in at lower aggregate contents. However, specifiers should be aware that with higher replacement levels, reductions in the elastic modulus-compressive strength ratio should be expected.

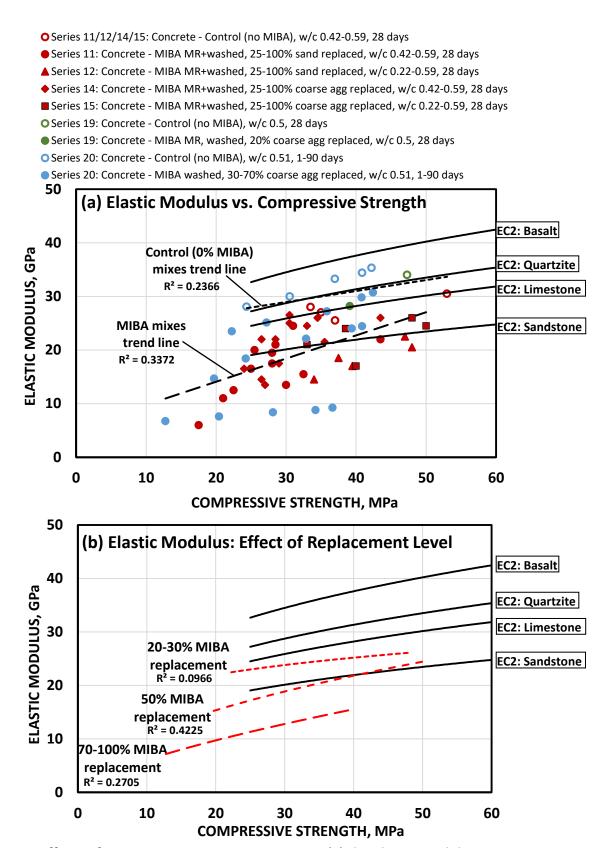


Figure 5.6: Effects of MIBA as aggregate in concrete on (a) the elastic-modulus-compressive strength ratio and (b) effect of aggregate replacement level on elastic modulus

Data from: Dhir et al. (2002); Van der Wegen et al. (2013); Zhang and Zhao (2014)

Drying shrinkage is another important property that affects the long-term concrete performance. Shrinkages recorded for mixes using MIBA as fine or coarse aggregate components, at time periods up to 1 year, are presented in Table 5.3.

Table 5.3: Effects of MIBA as fine/coarse aggregate on the concrete drying shrinkage

PUBLICATIO	ON TEST CONDITIONS	MIX	MIBA, %	SHRIN	KAGE, %	
Fine Aggre	gate Replacement					
Series 1	Air cured at 20°C at	Equal cement and	0	-0.	058	
	55% RH, tested at 200 days	water: 320 kg/m ³ cement, 190 kg/m ³	25	-0.	.087	
	200 days	free water	50	-0.	083	
			100	-0.	107	
Series 2	Air cured at 20°C at	Equal strength: 320-	0	-0.	058	
	55% RH, tested at	540 kg/m ³ cement,	25	-0.	098	
	200 days	190-140 kg/m³ free	50	-0.	101	
		water	100	-0	.11	
Coarse Agg	regate Replacement					
Series 3	Air cured at 20°C at 55% RH, tested at 200 days	Equal cement and	0	-0.058		
		water: 320 kg/m ³ cement, 190 kg/m ³ free water	25	-0.07		
			50	-0.073		
			100	-0.	.082	
Series 4	Air cured at 20°C at	Equal strength: 320-	0	-0.058		
	55% RH, tested at	540 kg/m ³ cement,	25	-0.067		
	200 days	190-140 kg/m³ free	50	-0.07		
		water	100	-0.133		
Series 5	After 1 year. Test	324 kg/m³ Cement,	0	-0	.36	
	conditions unknown	173 & 180 kg/m ³ water, w/c 0.5	20	-0.39		
				Drying period	Wetting period	
Series 6	14 day drying (20°C	Equal cement: 290	0	-0.03	-0.01	
	at 50% RH) and	kg/m³ cement, water	50	-0.03	-0.01	
	wetting (in water at 20°C) cycles	190, 183, 183 kg/m ³	100	-0.025	-0.005	

RH – relative humidity

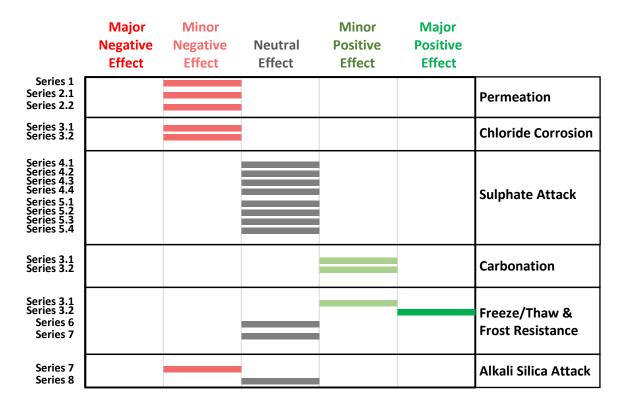
Data from: Dhir et al. (2002); Pera et al. (1997); Van der Wegen et al. (2013)

Although there is a degree of fluctuation in the data, it is evident that the shrinkage generally increased as the MIBA replacement level increased, indirectly caused by the higher water contents in the mix, to allow for the high water absorption of the MIBA aggregates, and also by the lower resistance to movement, due to the lower stiffness. In equal-strength mixes in Series 2 and 4, the higher cement proportion adopted to compensate for strength reductions with MIBA also contributed to higher shrinkage. Equal or lower shrinkage results were achieved for Series 6 using MIBA as a coarse aggregate replacement, albeit at shorter test ages of 14 and 28 days (after the wetting and drying periods). The MIBA used in these mixes had an unusually low absorption value of 2.4%, compared to the average 10% determined in Chapter 4, which explains this atypical result.

Creep is also closely related to water movement and as such, trends similar to the shrinkage behaviour were anticipated for the mortar and concrete products. Initial tests in parallel with shrinkage tests for Series 5 in Table 5.3 supported this assertion, as the mix with 20% MIBA as sand exhibited a slightly higher creep of 0.32%, compared with 0.31% for the natural aggregate mix.

Regarding the durability of concrete, the effects of MIBA on permeation, chloride corrosion, sulphate attack, carbonation, freeze/thaw and frost resistance and alkali-silica reactivity have been covered. For each mix series, the impacts of the ash as fine or coarse aggregate replacement on these parameters are categorised from 'major negative effect' to 'major positive effect' and the results are given in Figure 5.7.

The permeation properties, one aspect of which is water absorption, are known to correlate with the durability of concrete. Tests have measured the overall absorption (ASTM C1585, 2013, Series 1) and the initial surface absorption (BS 1881: Part 5, 1970, Series 2) of MIBA concrete mixes, though the latter procedure generally carries more practical value, as it is specifically the ease with which contaminants can pass through the cover concrete, which protects the reinforcement, that is of most interest. Consistent trends of increasing absorption



Series 1: Concrete, w/c 0.5, up to 50% of fine agg replaced;

Series 2.1: Concrete - MIBA MR+washed, Fcuk 40 MPa, w/c 0.42, up to 50% of fine agg replaced;

Series 2.2: Concrete – MIBA MR+washed, F_{cuk} 40 MPa, w/c 0.42, up to 50% of coarse agg replaced;

Series 3.1: Concrete – MIBA washed, w/c 0.45, 20% coarse agg replaced;

Series 3.2: Concrete - MIBA washed, w/c 0.45, 20% fine + coarse agg replaced;

Series 4.1: Concrete – MIBA MR+washed, PC, Fcuk 10 MPa, w/c 0.86, 25-100% fine agg replaced;

Series 4.2: Concrete - MIBA MR+washed, PC, F_{cuk} 20 MPa, w/c 0.59, 25-100% fine agg replaced;

Series 4.3: Concrete – MIBA MR+washed, PC+FA, F_{cuk} 20 MPa, w/c 0.59, 25-100% fine agg replaced;

Series 4.4: Concrete – MIBA MR+washed, SPC, F_{cuk} 20 MPa, w/c 0.59, 25-100% fine agg replaced;

Series 5.1-5.4 is the same as Series 4.1-2.4, except MIBA replaced the coarse agg.

Series 6: Concrete – MIBA MR, w/c 0.52, 10% sand replaced;

Series 7: Concrete – MIBA MR+washed, w/c 0.65, 2/6 and 6/20 limestone fractions replaced;

Series 8: Concrete – MIBA, w/c 0.76, 100% of gravel replaced.

MR – metal removal treatment (ferrous and non-ferrous fractions), PC – Portland cement, FA – fly ash, SPC – sulphate resistant cement, F_{cuk} - target concrete cube strength at 28 days,

Data from: Al Muhit et al. (2015); Dhir et al. (2002); Keppert et al. (2012a); Van den Heede et al. (2015); Van der Wegen et al. (2013)

Figure 5.7: Ranking of the effects of MIBA as aggregate on concrete durability

are evident in the data from the two tests, using MIBA as replacements for up to 50% of natural sand or gravel. The results are given as follows:

Water absorption (ASTM C1585, 2013): increase from 11% (natural aggregate mix),
 to 16% (MIBA as 50% of the fine aggregate)

• Initial surface absorption (BS 1881: Part 5, 1970): increase from 0.28 ml/m²/s (natural aggregate mix) to 0.72 ml/m²/s (MIBA as 50% of the fine aggregate) and 0.74 ml/m²/s (MIBA as 50% of the coarse aggregate)

It is found that for MIBA replacement levels greater than 25%, the initial surface absorption values of the concrete drifted above the 0.5 ml/m²/s upper limit for normal-strength concrete. As such, it is recommended to control the MIBA aggregate replacement level to below 25%, in structural concrete applications. There may scope to adopt higher contents in less susceptible products, provided that the associated effects on durability are deemed acceptable.

Washing can be adopted to reduce the chloride content of MIBA to meet the EN 12620 (2002) limits for aggregate in concrete. However, increases in the chloride diffusion coefficient still occurred with the ash as 20% coarse aggregate or 20% fine + coarse aggregate replacement, owing to the higher mix porosity (minor negative effect for Series 3). Secondary cementitious materials such as GGBS and coal fly ash are known to have beneficial chloride-binding abilities and filling effects and could be incorporated into the mix, along with MIBA, to counteract the higher chloride diffusion rate and thereby allow the use of the ash in a wider range of applications.

The effect on the sulphate attack susceptibility was classed as neutral, as concrete containing 25%–100% MIBA (with metal removal and washing treatments) as fine and coarse aggregate replacements exhibited no extra expansion, over a testing period of 200 days. Concrete carbonation resistance improved slightly, as is evident from reduced carbonation depths measured with MIBA. Optimal carbonation conditions require a balance between too much and too little moisture, and as such, the extra water absorbed by the MIBA aggregate appeared to be beneficial in slowing down the rate of carbonation ingress, despite higher permeation properties.

Concrete mixes incorporating MIBA as 20% coarse aggregate or 20% fine + coarse aggregate displayed superior freeze/thaw resistance compared to the control (positive effects for Series 3.1 and 3.2 in Figure 5.7). This is due to the higher porosity of the aggregates, which act as an air-entrainer. Good frost resistance was also achieved in concrete using MIBA to replace 10% of the sand fraction (classed as a neutral effect), with no strength deterioration after 125 freezing cycles.

When the alkali-silica expansion potential was measured in accordance with the modified Oberholster test (Oberholster, 1983), the MIBA concrete values exceeded those of the natural aggregate mix (minor negative impact for Series 7). However, the validity of this test outcome appears questionable, as the control limestone blend also greatly exceeded the 0.1% limit that signifies potential alkali-silica reactivity. Indeed, additional testing in Series 8, involving both laboratory and field work, revealed no evidence of deleterious action of silicate gel expansion with MIBA as a coarse aggregate in concrete.

As discussed during the analysis of the compressive strength, hydrogen gas expansion in concrete is a problem specific to MIBA, which can greatly affect the product durability. This behaviour arises from the reaction of the metallic aluminium in MIBA under the alkaline conditions in cement, to form aluminium hydroxide and expansive hydrogen gas. The expansive hydrogen gas formation and associated cracking were visible in only some products (just over one-third of the Series using standard processed MIBA as aggregate in mortar and concrete mixes). However, as it is difficult to directly measure the reactive metallic aluminium fraction in MIBA, a method to distinguish beforehand between ash samples that will or will not cause expansion has not yet been developed. Nonetheless, if expansion is going to occur, it will be evident almost immediately when mixing the MIBA with the cement and water, from the gas bubbles forming and the associated visible cracking. At this point, this collection of MIBA samples can be appropriately treated to remove the reactive metallic aluminium. In most cases, washing or chemical treatment has been adopted to reduce or stabilize the metallic aluminium in MIBA. Chemical treatment is a more effective option to specifically reduce the hydrogen gas evolution/metallic aluminium content.

5.2.2 Masonry Blocks

Masonry blocks are arguably more suited to the use of MIBA, given the large market and the wide range of strength quality levels. BS 6073-2 (2008) on Precast Concrete Masonry Units, outlines nine unit strengths ranging from 2.9 to 40 N/mm². The use of MIBA has been explored in a variety of block applications, as fine coarse and all-in aggregate replacements (Table 5.4).

Table 5.4: Work undertaken on the use of MIBA as aggregate in concrete blocks

SERIES	APPLICATION TYPE	MIBA PROCESSING	MIX DESIGN
1	Concrete masonry unit	Not specified	MIBA as aggregate
2	Concrete masonry unit	Sieved, ferrous removed, washed, aged	65% and 100% sand replaced by MIBA.
3	Concrete masonry unit	Sieved, ferrous & non- ferrous removed, aged	MIBA as 65% sand replacement
4	Case studies: Artificial reef, shore protection	Not specified	MIBA as aggregate
5	Block making	Not specified	MIBA + MIFA as the raw materials
6	Concrete paving blocks	Sieved, ground to 4 mm and 6 mm size fractions	Replaced 4 mm and 6 mm agg. fractions
7	Concrete masonry unit	Sieved as fine aggregate	MIBA as 10-100% of fine agg.
8	Hollow building stones	Screened, sieved, ferrous and non-ferrous removed, air separation, aged	MIBA as complete gravel substitute
9	Interlocking block	Screened, ferrous removed, plasma melted	MIBA slag as fine agg (43% of total mix)
10	Concrete masonry unit	Not specified	MIBA as aggregate
11	Single layer block, Double layer block.	Magnetically separated, screened, sintered	MIBA as 91% of block base layer, and as 45% of surface layer
12	Hollow concrete masonry blocks	Not specified	55% MIBA as agg (of overall mix)
13	Non-structural blocks	As produced	MIBA as all-in agg, 50-69% of total mix
14	Masonry units with blended organic binders	Separated into 5-10 mm and <5 mm sizes	40:60 ratio MIBA:coal fly ash as aggregate-filler
15	Case studies: Blocks in walls, boathouse, artificial reef, curbing, revetments, facades	Not specified	MIBA as aggregate

MIFA – municipal incinerated fly ash, agg - aggregate

Data from: Berg (1993); Berg and Neal (1998a); Berg and Neal (1998b); Breslin et al. (2003); Environment Agency (2002); Ganjian et al. (2015); Holmes et al. (2016); Jansegers (1997); Katou et al. (2001); Lauer (1979); Nishigaki (2000); Roethel and Breslin (1995); Siong and Cheong (2004); Vu and Forth (2014); Wiles and Shepherd (1999).

Screening, sieving, metal removal and ageing treatments have been commonly adopted and, to a less frequent degree, thermal treatment. The use of the ash in block applications has progressed to a stage at which a number of case studies have been carried out. Analysis of the data on the performance of these blocks has yielded the following findings:

<u>Appearance</u>: Ferrous metals in MIBA can stain the outer surface of the blocks (Series 3 and 15); however, this can be managed by standard magnetic separation treatment of the ash to reduce these ferrous components. No other negative effects on the aesthetics of the products were incurred, and indeed, MIBA blocks have been used in interior wall renderings (Series 8), with no unsightly defects.

<u>Unit Weight:</u> The lower specific gravity of MIBA (average of 2.35 determined in Chapter 4) resulted in a decrease in the unit weight of the blocks when used as a replacement for sand and gravel; however, MIBA blocks were generally still categorised as medium-weight rather than lightweight (Series 2, 3, 6, 7, 10, 13). The irregular particle shape, porosity and high water absorption can affect the volumetric filling during moulding, if the water demand is not properly accounted for. In this regard, the inclusion of coal fly ash as a cement component, which is known to have a lubricating ball-bearing effect, and superplasticiser as an admixture, both led to increases in the mix density (Series 2), due to improved volumetric filling during moulding.

Strength: As found previously with the mortar and concrete products, similar reductions in compressive and tensile strengths are evident with blocks using MIBA as aggregate. However, strength requirements in many block applications are not overly demanding. Indeed, mixes incorporating MIBA have consistently satisfied the respective strength requirements of non-load-bearing units (Series 13), load-bearing units (Series 1, 2 and 13), paving units (with fibre addition) (Series 6) and interlocking blocks (MIBA slag) (Series 9). The key with MIBA aggregate

in blocks is to ensure effective volumetric filling, and indeed, similar to the density behaviour, the inclusion of coal fly ash as a cement component or a superplasticiser admixture improves the filling behaviour and results in enhanced strength performance (Series 2).

Water Absorption: Increases in absorption have been measured with MIBA aggregate blocks. Replacing the fine aggregate again led to larger increases in absorption, compared with the coarse substitution. For example, with MIBA used to replace the 4 mm and 6 mm aggregate size fractions (Series 6), the finer fraction replacement led to a doubling of the mix absorption, whilst blocks with the 6 mm MIBA aggregate performed comparable to the natural aggregate mix and had absorption values below the 6% target limit for paving units of BS EN 1338 (2003). Fine aggregate replacement levels will, in certain applications, need to be limited, as was the case for the load-bearing masonry blocks in Series 6, for which the MIBA fine aggregate replacement level was controlled to 20% to meet the target 12% limit given in ASTM C90-11b (2011). In other applications, such as the hollow building stone products in Series 8, the higher absorption of MIBA blocks can be acceptable, provided no associated durability problems are evident.

Shrinkage: Despite the higher water absorption, hollow building stones, with MIBA as a complete coarse aggregate replacement (Series 8) exhibited no adverse effects on the drying shrinkage. Indeed, masonry units made with MIBA as a sand substitute (Series 2) compare favourably with lightweight units with commercial aggregate, in terms of the drying shrinkage behaviour.

<u>Pop-outs:</u> In addition to altering the appearance, if not managed, metals present in MIBA can, also affect the block structure. Metals caused pop-outs and spalling in some mixes (Series 3 and 15), though again, this can be overcome by reducing the ferrous and non-ferrous metals in MIBA, using standard magnetic separation and eddy current treatments.

<u>Freeze/Thaw Resistance:</u> Masonry units containing MIBA as a fine aggregate, delivered freeze/thaw resistance on the same level as commercial masonry units and thus satisfied

ASTM C90-11b (2011) requirements for load-bearing masonry units (Series 2), whilst hollow building blocks with MIBA as a coarse aggregate, and paving blocks with MIBA replacing the 6-mm aggregate fraction, achieved similar resilient frost resistance. However, compared to the freeze/thaw limits for paving units in BS EN 1338 (2003), one mixes in Series 6, using MIBA to replace both 4 + 6-mm-size fractions, did not meet the requirements. The findings match up with previous water absorption results and suggest that the MIBA replacement level may have to be limited in certain block applications, particularly when substituting finer size fractions.

<u>Fire Resistance:</u> Blocks containing MIBA aggregate delivered good resistance to fire exposure and indeed compared favourably, in this regard, with conventional blocks (Series 4).

<u>Slip Resistance</u>: In certain applications, such as paving blocks, resistance to slipping is an important requirement. Blocks using MIBA as a replacement for the 4-mm, 6-mm or 4 + 6-mm aggregate fractions (Series 6) delivered excellent slip resistance, classified as having extremely low potential for slip, according to BS EN 1338 (2003). This favourable effect was attributed to the irregular shape of the MIBA particles.

5.2.3 Lightweight Aggregate Production

Lightweight aggregate production aims to manufacture a material with higher porosity and associated light weight that has a strong surface layer and good structural performance. These aggregates, which are also referred to as artificial or synthetic aggregates, can be used in lightweight concrete and a growing number of other applications including lightweight fill and insulation products. The effect of the thermal treatment stage in binding the heavy metals in the ash within the resultant pellets and thereby lessening the leaching potential, is an added benefit of this use.

Processing of the ash adopted for this use included standard particle size adjustments, weathering/ageing and ferrous and non-ferrous metal removal treatments. The ash was then mixed with various combinations of coal fly ash, cement, lime, clay and sand, though MIBA

has also been used, at times, as the sole input material (not including water additions). The blend then went through a series of steps, including milling, sieving, drying and pelletising and ended usually with the key thermal treatment (sintering). Details of the MIBA processing, mix constituents and main steps involved in the lightweight aggregate production process are given in Table 5.5 for each mix series.

Table 5.5: Lightweight aggregate mix series produced using MIBA

SERIES	MIBA PROCESSING	MIX CONSTITUENTS	AGGREGATE PRODUCTION
1	Aged, metal removal, sieved < 8mm	40% MIBA, 60% FA; 40% MIBA, 50% FA, 10% clay	Mix milled, sieved, dried, pelletised, sintered at 1100°C
2	Aged, metal removal, sieved < 8mm	40% MIBA, 60% FA, with 0-12% activated carbon	Mix milled, sieved, dried, pelletised, sintered at 1040-1140°C
3, 4	Aged, metal removal, sieved < 8mm	100% MIBA	Mix milled, sieved, dried, pressed, sintered at 1020-1100°C, 1 hour
5	Aged, sieved < 8mm, milled	100% MIBA	Pelletised with water, dried, sintered at 1000-1060°C $$
6	Screened	100% MIBA	Melted at 1000°C, crushed
7	Metal removal	95% MIBA, 5% clay	Extruded, pelletised, sintered at 950-1000°C
8	Sieved, metal removal	82% MIBA, 18% clay; 90% MIBA, 10% clay	Extruded, pelletised, dried, fired at 1100-1230°C
9	Crushed, metal removal	82% MIBA, 18% clay; 90% MIBA, 10% clay	Extruded, pelletised, dried, fired at 1100-1230°C
10, 11, 12	Crushed, metal removal	85% MIBA, 15% clay	Pelletised and fired at 975°C, 13.5 minute retention time
13	Aged, metal removal, milled	80% MIBA, 20% cement	Thermally treated at 600-900°C
14	Aged, metal removal, sieved	100% MIBA	Pelletised, sintered at 400-600°C, for 60-240 mins
15	Size fraction < 0.5mm used	30% MIBA, 10% FA, 30% sand, 15% cement, 10% clay	Mix granulated; no thermal treatment
16	Metal removal, milled	60-80% MIBA, 10-30% cement, 7- 30% lime, 13-27% FA	Granulated with water additions, cured

FA – coal fly ash

Data from: Almeida and Lopes (1998); Bethanis (2007); Bethanis and Cheeseman (2004); Bethanis et al. (2002); Bethanis et al. (2004); Cheeseman et al. (2005); Cioffi et al. (2011); Qiao et al. (2008a); Rebeiz and Mielich (1995); Wainwright (2002); Wainwright and Boni (1983); Wainwright and Cresswell (2001); Wainwright and Robery (1991); Wainwright and Robery (1997); Wang et al. (2003).

Selection of the appropriate sintering temperature plays a key role in attaining the desired lightweight aggregate properties. The aim is to determine the optimal heating conditions to induce expansion that results in lightweight properties, whilst retaining the balance between satisfactory strength and low water absorption. The properties of the resultant aggregates will vary, with specific sintering condition, depending on the precise composition of mix. As such, establishing the optimal sintering conditions for each mix is initially, to some extent, a trial and error process. Sintering temperatures for MIBA mixes mostly varied from 950°C to 1230°C (Series 1-12), though lower ranges from 600°C to 900°C and 400°C to 600°C were adopted for Series 13 and 14, respectively. Cracks developed in the aggregates when heating the Series 14 MIBA samples above 800°C, and as such, a lower temperature range was selected. However, this cracking did not occur in the other MIBA samples and, in general, should not present a problem. Two additional mix series (Series 15 and 16) did not adopt any heat treatment in the aggregate production, though both contained notable contents of cement alongside MIBA.

Particle density results for manufactured MIBA lightweight aggregates are presented in Figure 5.8 (a). The lightweight aggregate classification limit of 2000 kg/m³ (BS EN 13055, 2016) and typical density of commercial Lytag are also given for comparison purposes. It is evident that the majority of MIBA lightweight aggregates have densities close to the typical Lytag value and well within the lightweight aggregate limit. For MIBA blends ground to a high fineness, dramatic increases in density have been measured at particular sintering temperatures (Series 4). MIBA aggregates produced without thermal treatment (Series 15 and 16) did not experience this densification and had amongst the lowest density values.

Density results for MIBA aggregates are replotted in Figure 5.8 (b), to examine the specific effect of the thermal treatment. Increases in density are visible for each mix series as the temperature increased, though the densification was much more pronounced for the finely ground MIBA blends. Large increases in strength are likely to accompany the increases in density; however, this may not necessarily always be desirable in lightweight applications. The densification progressed only up to a certain temperature, after which large discontinuous pores began to form, resulting in rapid decreases in density. The point at which this occurred

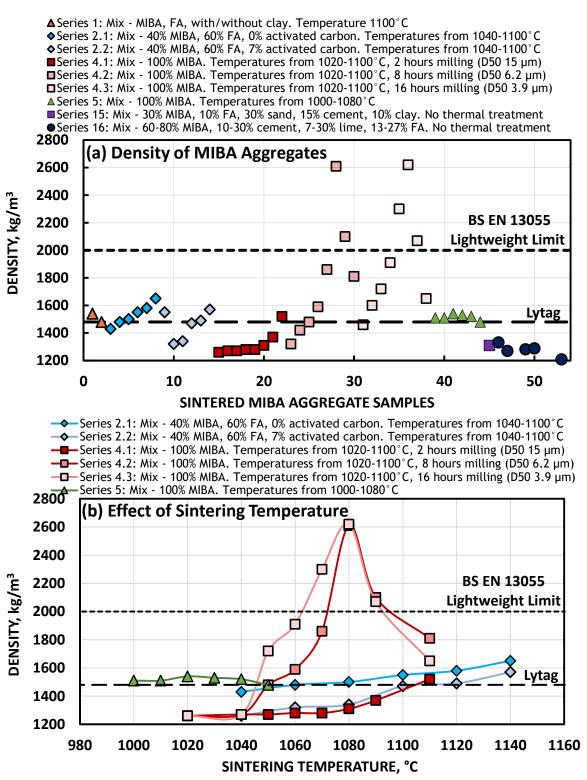


Figure 5.8: Particle density results for (a) MIBA lightweight aggregates and (b) the effect of sintering temperature

FA – coal fly ash, D_{50} - 50% of the sample's mass is comprised of particles < this diameter Data from: Almeida and Lopes (1998); Bethanis (2007); Bethanis and Cheeseman (2004); Bethanis et al. (2004); Cheeseman et al. (2005) and Cioffi et al. (2011)

fluctuated, depending on the chemical composition of the blend. Aggregates made using bulk contents of coal fly ash had higher peak density sintering temperatures, whilst MIBA only blends reached the peak density within the 1000°C–1100°C range.

Additional bulk density values from 820 to 1060 kg/m³ have been measured for MIBA lightweight aggregates (Series 1, 2, 8, 9, 10), which are slightly above the typical Lytag range of 700–800 kg/m³, though well within the BS EN 13055 (2016) lightweight aggregate classification limit of 1200 kg/m³.

Owing to the inherently high porosity of lightweight aggregate, the water absorption is another important property to examine. Results for the manufactured MIBA aggregates are presented in Figure 5.9, and again, the classification limit from BS EN 13055 (2016) for lightweight aggregates and a typical Lytag value are also given as benchmarks.

- △Series 1: Mix MIBA, coal fly ash, with/without clay. Temperature 1100°C
- △Series 2: Mix 40% MIBA, 60% coal fly ash, 7% activated carbon. Temperatures from 1040-1100°C
- △Series 5: Mix 100% MIBA. Temperatures from 1000-1080°C
- Series 8: Mix 82-90% MIBA, 10-18% clay, Temperatures 1100-1230°C
- **★**Series 10: Mix 85% MIBA, 15% clay, Temperature 975°C
- ◆Series 14: Mix 100% MIBA. Temperatures 400-600°C.
- Series 16: Mix 60-80% MIBA, 10-30% cement, 7-30% lime, 13-27% coal fly ash. No thermal treatment

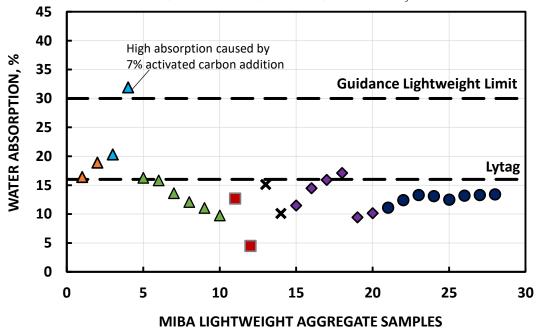


Figure 5.9: Water absorption results for MIBA lightweight aggregates

Data from: Bethanis (2007); Bethanis and Cheeseman (2004), Cheeseman et al. (2005); Cioffi et al. (2011); Wainwright (2002); Wainwright and Boni (1983); Wang et al. (2003)

The MIBA aggregates delivered satisfactory water absorption properties, most comparable to that of Lytag, with values around 15%. The products fall well within the guidance threshold of 30% for lightweight aggregates, though with the exception of one result from Series 2. This high result was caused, by design, by the addition of 7% activated carbon to the aggregate mix of 60% MIBA + 40% coal fly ash. The activated carbon had the desired effect of lowering the density of the resultant aggregate, though it also led to an associated large increase in water absorption due to greater expansive gas formation from the reaction of carbon with oxygen.

Regarding the mechanical performance of MIBA lightweight aggregate, fracture strengths of individual pellets increased from 4.4 to 5.4 MPa, with firing temperatures increasing from 1000°C to 1050°C (Series 5). The results for MIBA pellets were below the Lytag value of 6.9 MPa, indicating that although MIBA lightweight aggregates can match the density and water absorption properties of this commercial aggregate, slightly lower strengths are to be expected. One option to improve strength is to include a binder addition alongside the MIBA. With various combinations of MIBA and cement, lime and coal fly ash, compressive strength values of 1.9 to 4.5 MPa have been measured for the resultant artificial aggregates (Series 16). In order of strongest to weakest, the mix combinations ranked as follows: MIBA + cement, MIBA + lime and MIBA + lime + coal fly ash.

Additional work has been carried out using a different style of test, outlined in ASTM D2166 (1985), intended for measuring the unconfined compressive strength of cohesive soils. Values of 50-52 MPa were recorded for MIBA aggregates produced at sintering temperatures ranging from 400°C to 600°C (Series 14). The low temperatures adopted with this mix are outside the normal approach. The previous analysis shows that MIBA aggregates do not require a great amount of expansive bloating to meet lightweight requirements and these aggregates, with the lower temperature treatment and without major densification, were still sufficiently strong for their target use in permeable blocks (Series 14).

5.2.4 Lightweight Aggregate Concrete

This section covers the performance of lightweight concrete incorporating MIBA as an aggregate component. A number of the MIBA lightweight aggregates dealt with previously were also subsequently used in concrete. An additional mix series used processed, washed, but un-sintered MIBA as a replacement for the 12- to 6-mm sintered coal fly ash aggregate fraction in lightweight concrete (Series 17). Testing carried out on MIBA lightweight aggregate concrete covered consistence, unit weight, absorption, strength, elastic modulus, shrinkage, creep, corrosion, alkali-silica reactivity, corrosion and cracking properties.

Slump results for concrete incorporating MIBA lightweight aggregate are presented in Figure 5.10. It is found that pelletised + sintered MIBA aggregate (including minor clay fractions) greatly improved the concrete consistence when used as a replacement for the natural coarse aggregate (Series 9 and 10). These MIBA mixes also outperformed others using Lytag and indeed another 'pulverised fuel ash aggregate', which is known to have a positive effect on the concrete workability, owing to its spherical particle shape. Slump improvement with MIBA is attributed to the resultant smoothness of the pelletised and sintered aggregate. In contrast, without the pelletising and sintering treatments, mixes using MIBA aggregate as a replacement for Lytag exhibited minor decreases in slump at high replacement levels (Series 17).

As expected, concrete mixes experienced decreases in their unit weight when MIBA lightweight aggregates were used as a replacement for the normal-weight natural aggregate. Reductions in bulk density from 2.1 g/cm³ (with natural aggregate) to 1.71–1.82 g/cm³ (with MIBA lightweight coarse aggregate) and plastic density from 2.4 g/cm³ (with natural aggregate) to 2.0–2.1 g/cm³ (with MIBA lightweight coarse aggregate) were recorded for concrete mix Series 13 and 10, respectively. The extent of the density reduction depends on the ash processing and the specifics of the aggregate manufacturing process, particularly the sintering temperature and material fineness, as is evident from Figure 5.8 (b).

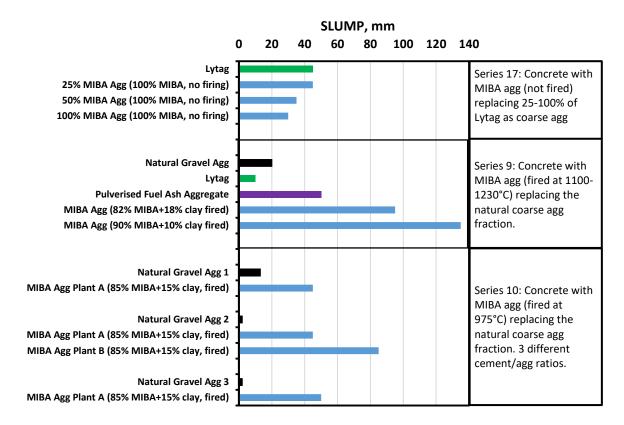


Figure 5.10: Consistence of lightweight MIBA aggregate concrete

Data from: Dhir et al. (2002); Wainwright and Boni (1983); Wainwright and Cresswell (2001)

Compressive strengths, after 28 days curing, are presented in Figure 5.11 for the MIBA lightweight aggregate concrete mixes. These mix series consist of aggregates manufactured from different blends of MIBA, clay and pulverised fuel ash, fired at various temperatures, and used as a replacement for natural aggregate or commercial lightweight aggregates such as Lytag and Leca.

It is evident from Figure 5.11 that the compressive strengths with MIBA fluctuated above and below those of the reference Lytag concrete counterparts, depending on the blend design and firing conditions. Strengths of the MIBA aggregate concrete mixes were well above the expanded clay Leca mix. It is apparent that the firing treatment of MIBA led to a notable improvement in the resultant concrete strength compared to unfired MIBA aggregates. The data indicate that MIBA offers good potential for use as an alternative lightweight aggregate in concrete, though it generally does not achieve the same strength level as natural aggregate mixes.

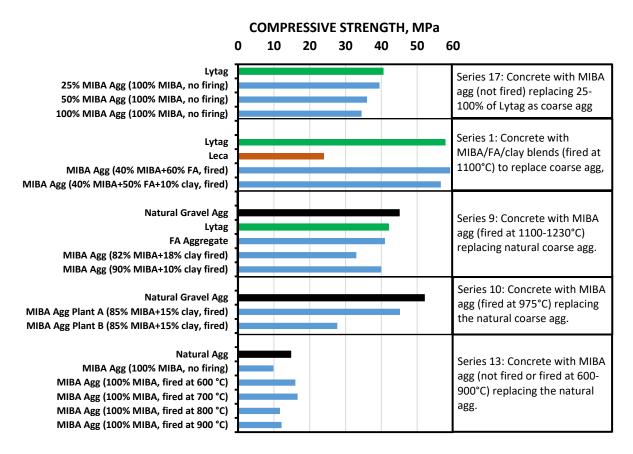


Figure 5.11: Compressive strengths (28 days) for lightweight MIBA aggregate concrete FA – coal fly ash, agg – aggregate, Leca – lightweight expanded clay aggregate Data from: Bethanis (2007); Dhir et al. (2002); Qiao et al. (2008a); Wainwright and Boni (1983); Wainwright and Cresswell (2001)

As has been found previously with MIBA in concrete in section 5.2.1, the tensile strengths for MIBA lightweight aggregate concrete mimics the corresponding trends evident in the compressive strength behaviour. The tensile strengths of MIBA mixes were comparable to those of their Lytag counterparts (Series 17), though below natural aggregate mixes (Series 10). The elastic modulus of MIBA lightweight aggregate concrete mixes also exhibited these same tendencies, achieving values comparable to those of Lytag concrete though below those of the natural aggregate mixes (Series 10, 11, 12 and 17). In line with these findings, shrinkage strains with MIBA were similar to those of Lytag mixes, though 54%–72% higher than those of natural aggregate mixes (Series 8 and 10). The decrease in the elastic modulus when using lightweight MIBA aggregate as a replacement for natural aggregate also resulted in higher concrete creep strains (Series 10), owing to the lower stiffness of the lightweight aggregates.

On the durability of MIBA lightweight aggregate concrete mixes, the following findings emerged:

- Permeation properties: With MIBA replacing 25%–100% of Lytag (Series 17), lower initial surface absorption values of 0.2–0.4 ml/m²s were achieved, compared to 0.7–1.2 ml/m²s with the commercial Lytag mixes.
- Corrosion: After a 12-month testing period, a higher rate of corrosion was measured for the lightweight mix using aggregate produced with a pelletised and fired blend of 85% MIBA + 15% clay, compared to the natural aggregate mix (Series 11). This is explained by the inevitably higher porosity of the lightweight pellets.
- Alkali-silica reactivity: The inclusion of the same Series 11 MIBA lightweight aggregate
 as a replacement for the gravel fraction in concrete showed no negative effects on the
 alkali-silica reactivity potential.
- Cracking: Reinforced concrete beams using MIBA lightweight aggregate (85% MIBA + 15% clay, pelletised and fired at 975°C) exhibited cracks with width and spacing similar to those of commercial Lytag concrete beams, and indeed the overall structural performance was evenly matched (Series 12).

5.3 Use as Cement Component

5.3.1 Raw Feed in Cement Clinker Production

Cement clinker manufacturing involves intimate mixing and heat treatment of a blend of calcareous rocks, such as limestone or chalk, and argillaceous rocks such as clay or shale. The calcium, silicon, alumina and iron oxides in these materials undergo a series of chemical reactions to form a clinker material with main compounds tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A) and tetracalcium aluminoferrite (C_4AF). After cooling and storage, the clinker is ground and gypsum is added to improve the future setting behaviour. Cement clinker production places a high demand on natural resources and large amounts of CO_2 emissions are generated from the energy needed to convert the calcium carbonate ($CaCO_3$) in limestone into calcium oxide (CaO_3). As such, a strong interest has developed to bring alternative sources of the required calcium (without the conversion from $CaCO_3$), silica, alumina and iron.

The chemical composition of MIBA indicates that it can potentially serve as a valuable resource in cement clinker production, with its average SiO_2 , CaO, Al_2O_3 and Fe_2O_3 contents of 37.4%, 22.2%, 10.2% and 8.3% (from Chapter 4). The work undertaken has involved its use in the cement clinker raw feed at relatively low contents up to 15%, alongside the conventional limestone, shale, sand and clay materials and, at times, secondary materials such as copper slag, iron slag and coal fly ash. To ensure that the same main clinker phases are produced when incorporating MIBA, the sum of SiO_2 , CaO, Al_2O_3 and Fe_2O_3 from all the constituents has to be tightly controlled. As such, when MIBA is brought in, the contents of the other constituents have to be altered slightly to meet the target quotas for the main oxides.

Data on the chemical compositions of the MIBA cement clinkers are presented in Table 5.6, covering the oxide composition, the cement compound contents (C_3S , C_2S , C_3A , C_4AF) and the lime saturation factor (LSF), hydraulic modulus (HM), silica ratio (SR) and alumina ratio (AR) parameters. After adjustments of the mix constituents to accommodate MIBA in Series 2, 3, and 4, verified by the very similar LSF, HM, SR and AR values, it is not surprising to find that the oxides and clinker compound contents of the MIBA blends were very similar to those of the control mixes. However, towards the upper end of the tested MIBA contents, around 10%, the levels of minor components, such as P_2O , metallics and organics began to build up to a level that suppressed C_3S formation. This led to reductions in the compressive strength and lengthening of the setting time with MIBA cement clinkers (Series 4). The chloride content of MIBA is another parameter to consider, as its presence in the feed can corrode the equipment in the kiln in the long term, if not properly controlled. One solution to pre-empt this problem is to subject MIBA to a washing treatment before its use, as was done in Series 1 and 4.

A different approach was adopted with Series 1 and 5, which directly replaced certain percentages of the raw feed (up to 15%), without adjusting the contents of the other constituents to preserve the same overall oxide composition. It is evident from Table 5.6 that the LSF, HM, SR and AR values and main oxide contents drifted away from corresponding values for the non-MIBA blends. From subsequent testing of Series 5, it was found that at the 5% replacement level, compressive strengths comparable to that of the control were

Table 5.6: Chemical composition of cement clinker mixes that used MIBA in the raw feed

CEDIEC	NAIDA O/				0	XIDE C	OMPC	SITION	١				CEMI	ENT CC	МРО	UNDS	CLINK	ER PAI	RAMET	ΓERS
SERIES	MIBA, %	SiO ₂	Al_2O_3	Fe ₂ O ₃	CaO	MgO	P ₂ O	SO ₃	K ₂ O	Na₂O	F-CaO	Cl	C₃S	C_2S	C ₃ A	C ₄ AF	LSF	НМ	SR	AR
1	0	20.0	5.7	3.1	65.9	0.8	-	2.33			2.59	n.d.	56.0	15.2	9.8	9.5	101.6	2.3	2.3	1.8
	5	23.3	5.2	3.3	62.8	0.9	-	2.01			0.5	n.d.	30.8	43.9	8.1	10.0	85.3	2.0	2.8	1.6
	10	24.9	5.0	3.3	61.1	1.0	-	2.26			0.48	n.d.	13.2	61.4	7.6	10.2	78.7	1.8	3.0	1.5
2	0	23.3	5.1	3.5	66.2	0.8	0.17	0.27			0.61	0	50.0	27.3	7.6	10.8	89.8	2.1	2.7	1.5
	2	22.6	5.5	3.7	65.0	0.6	0.40	0.13			0.98	0.02	46.4	26.4	8.3	11.4	90.2	2.0	2.4	1.5
	4	22.4	5.5	3.6	64.9	0.6	0.57	0.18			1.04	0.02	46.6	25.6	8.5	11.0	90.5	2.1	2.5	1.5
	6	22.2	5.6	3.7	64.5	0.5	0.94	0.19			1.23	0.02	46.0	24.7	8.4	11.3	90.8	2.1	2.4	1.5
	8	23.0	5.4	3.4	64.8	0.6	1.27	0.23			2.31	0.03	37.9	29.7	8.5	10.4	88.8	2.0	2.6	1.6
3	0	20.6	4.8	3.6	62.6	3.6	0.20	0.19	0.18	0.19							95.5	2.2	2.4	1.4
	9	20.8	5.3	3.3	61.7	4.0	0.32	0.19	0.3	0.82							92.9	2.1	2.4	1.6
4	0	23.1	5.1	3.9	65.5	2.5		0.08			0.18		48.0	30.0	6.9	12.0	89.4	2.0	2.6	1.3
	3.5	23.0	5.4	3.9	65.4	2.5	0.53	0.02	0.32	0.26	0.28		46.0	31.0	7.7	11.8	89.2	2.0	2.5	1.4
5	0																1.0	2.3		
	3																1.0	2.1		
	10																0.8	1.9		
	15																0.8	1.8		
	15 (+CaO)																1.0	2.3		

Series 1: Constituents – limestone, shale, clay and MIBA, oxides not controlled. Series 2: Constituents – limestone, sand, copper slag, coal fly ash and washed MIBA, oxide controlled. Series 3: Limestone, sandstone, coal fly ash, slag and MIBA, oxides controlled. Series 4: limestone, iron slag, clay, sand and washed MIBA, oxides controlled. Series 5: limestone, clay and shale and metal removed MIBA, oxides not controlled. Data from: Krammart and Tangtermsirikul (2003/2004); Lam et al. (2010a/2011), Li et al. (2016); Pan et al. (2008) and Shih et al. (2003).

achieved; however, at the higher 10% and 15% replacements, the MIBA mixes incurred large strength losses owing to the lower CaO in the feed, arising from the replacement of the traditional limestone, clay and shale materials with the ash. With this direct replacement approach, it was subsequently found that a conditioning treatment involving the addition of extra CaO to the MIBA blends (Series 5), to meet the hydraulic modulus and lime saturation factor quotas, was able to correct the problem, and indeed, satisfactory strengths were then achieved using 10% and 15% MIBA.

Based on the overall data, MIBA is suitable for use in cement clinker raw feed, at low contents, mentioned previously as up to around 5%, without any compromising effects on the performance of the resultant products. Higher contents up to 15%, are also possible, depending on the contents of certain minor constituents of the ash, such as P₂O₅, SO₃, Cl and the metallics, and the cement class requirements. Magnetic separation treatment to reduce the metallic fraction and washing to lessen the chloride content are recommended to improve the prospects for MIBA use. Owing to the vast size of the cement manufacturing industry, which produced 250 million tonnes of cement clinker in the European Union countries in 2015 (Cembureau, 2016), it is projected, based on the ash production figures, that close to 80% of the total MIBA generated in these countries could be expended with this use alone, if this 5% content was adopted across the board in cement kilns. This shows the great potential available with MIBA, though much developmental work is needed to realise this.

5.3.2 Paste, Mortar and Concrete

As greater emphasis has been placed on sustainable construction, the incorporation of secondary and recycled materials as cementitious components has become an important part of the cement industry. Indeed, European standard EN 197 (2011) recognises GGBS, silica fume, pozzolana (natural and natural calcined), coal fly ash (siliceous and calcareous), burnt shale and limestone as allowable cementitious components in combination with clinker, and specifies 27 different categories of cements based on the content ranges of these constituents. Up to 5% of a selected minor additional constituent in the cement mixture is also permissible. As such, the term 'cement' is no longer solely reserved for Portland cement. It

can be argued that acceptance of the above listed permissible cementitious constituents is only a start. Indeed, there is an emerging market for additional secondary materials that show potential for use, one of which is MIBA.

This section deals with MIBA as a cement component in pastes, mortars and concrete products. From the material characteristics in Chapter 4, the oxide composition and mineralogy of MIBA indicate a degree of reactivity as a cementitious component. The physical properties dictate that grinding of MIBA would be a prerequisite to convert it into a reactive powder form. The presence of metallic aluminium, which could lead to expansive reactions, is also highlighted as a key issue to address with MIBA as a cement component. In part because of this issue, and also with the aim of generally upgrading the reactivity and mechanical properties of MIBA products, the material has regularly been subjected to various chemical or thermal treatments before its use.

Flow and slump results showing the effects of MIBA, as a cementitious component, on the consistence of mortar and concrete mixes, respectively, are presented in Table 5.7. Both increases and decreases in the flow and slump were evident with MIBA mixes, which can be attributed in part to differences in the processing and fineness of the ash, and also to the impreciseness of the workability test method; however, in most cases the MIBA results were not too far away from the control. Grinding is beneficial in somewhat lessening the irregularity of the MIBA particle shape; therefore, workability losses are milder compared with its use as aggregate. However, as the level of grinding and fineness of the ash increases, the associated water demand rises, which is evident from the consistence results for Series 2, 5 and 6. Additional thermal treatment showed minor improvements in workability (Series 3, 4 and 6), due to a reduction in the absorptive organic fraction, the rounding effect on the particle shape and the reduced porosity associated with the melting and grinding processes.

The effects of MIBA on the initial and final setting times, converted to relative values, are illustrated in Figure 5.12. It is clear that MIBA causes a lengthening of the initial and final setting times, though this is generally expected with all pozzolanic materials. The presence of

Table 5.7: Effects of MIBA as a cement component on mortar/concrete consistence

MIX	MIBA PROCESSING	RESULTS
(A) Mortar		
Series 1: w/c 0.5		Flow, mm
Control		131
10% MIBA	Ground < 0.074mm	123
20% MIBA	Ground < 0.074mm	109
30% MIBA	Ground < 0.074mm	117
40% MIBA	Ground < 0.074mm	119
Series 2 w/c 0.35	<u>5-0.48</u>	
Control		190
10% MIBA	Ground to 0.002 mm	167
40% MIBA	Ground to 0.002 mm	136
Series 3: w/c 0.5		
Control		60
20% MIBA	Vitrified at 1500°C, quenched, ground	75
30% MIBA	Vitrified at 1500°C, quenched, ground	70
<u>Series 4: w/c 0.7</u>	, Varying grinding & with thermal treatment	
Control		170
20% MIBA	Ground < 0.125 mm	175
20% MIBA	Ground < 0.125 mm, heated at 550°C	170
20% MIBA	Ground < 0.125 mm, heated at 750°C	165
20% MIBA	Heated at 550°C, then ground < 0.125 mm	169
20% MIBA	Heated at 550°C, ground for shorter time, sieved < 0.63 mm	160
(B) Concrete		
Series 5: w/c rat	io 0.5, 30% MIBA, Dry vs wet grinding	Slump, mm
Control		60
30% MIBA (dry)	Dry ground, $D_{50} - 0.015$ mm	110
30% MIBA (wet)	Wet ground (1:1 solid/water ratio), $D_{50} - 0.003$ mm	0
<u>Series 6 w/c 0.55</u>	5, 20% MIBA, Varying fineness & with thermal treatment	
Control		162
20% MIBA	Ground < 0.6 mm	178
20% MIBA	Ground < 0.074 mm	167
20% MIBA	MIBA < 0.6 mm melted at 1450°C, water quenched	187
20% MIBA	MIBA < 0.6 mm melted at 1450°C, air-cooled	180
Series 7: w/c 0.5	<u>5, 15% MIBA</u>	
Control		105
15% MIBA	Ground < 0.09 mm	55

Data from: Bertolini et al. (2004); Cheng et al. (2011a); Cheng et al. (2011b); Juric et al. (2006); Saccani et al. (2005); Tang et al. (2014); Tang et al. (2016); Whittaker et al. (2009a); Whittaker et al. (2009b).

organics, phosphorus and zinc in MIBA also contributes to retardation of the cement hydration process. There are certain situations, such as large volume concrete pours, in which the slower setting and lower peak heat release may be desirable.

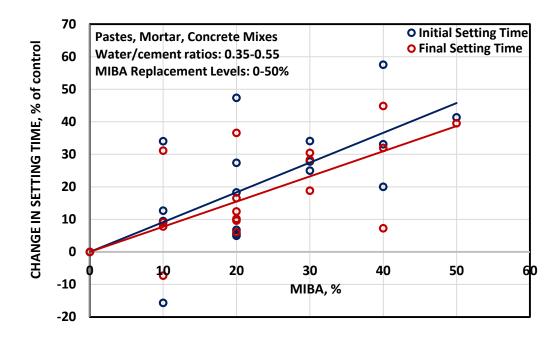


Figure 5.12: Effects of MIBA on the setting of pastes, mortar and concrete mixes

Data from: Cheng et al. (2011a); Cheng et al. (2011b); Cheng (2012); Kim et al. (2016); Lin and Lin (2006a); Lin et al. (2008a); Saccani et al. (2005); Whittaker et al. (2009a); Whittaker et al. (2009b); Zhang and Zhao (2014).

Owing to the lower density of MIBA compared with Portland cement, pastes and mortar mix Series 1 and 4 and an additional Series 8 (Kim et al., 2016), exhibited a slightly lower fresh unit weight with MIBA at replacement levels up to 40%. Corresponding to reductions in the mortar flow evident for Series 1 in Table 5.7, the bleeding was also reduced as the MIBA content increased, owing to the higher water retention of the ash.

Compressive strength results for pastes, mortar and concrete mixes with MIBA are presented in Figures 5.13, 5.14 and 5.15, respectively. For each product, the strength values were split into two groups: (a) standard processed MIBA samples and (b) samples subjected to additional thermal or chemical treatments.

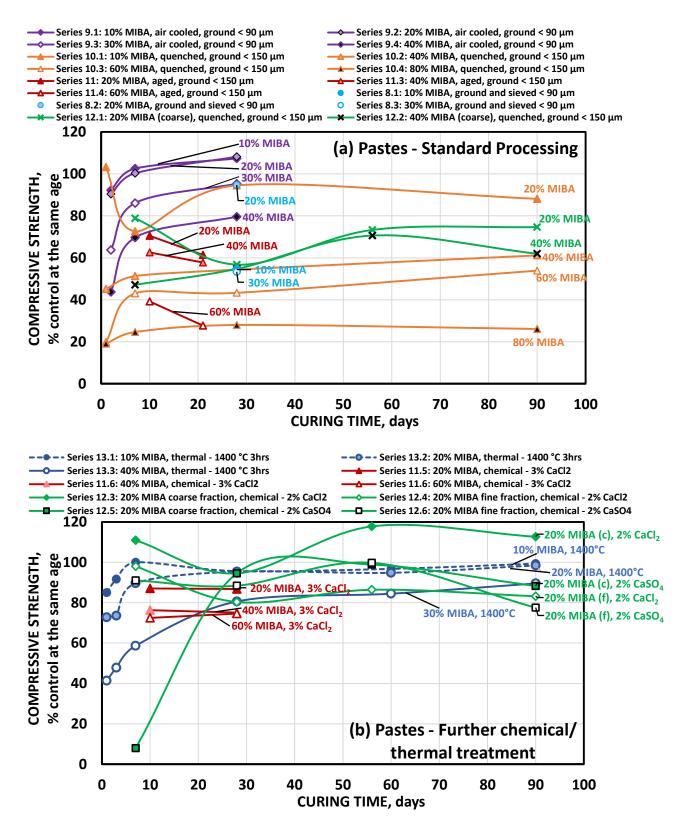
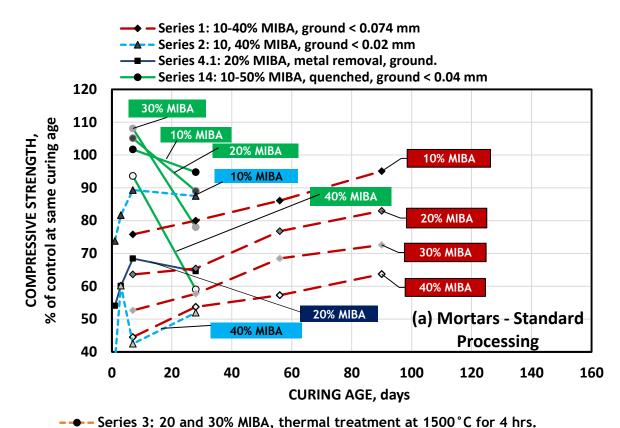


Figure 5.13: Effects on the strength development of cement pastes using (a) standard processed MIBA and (b) further chemically or thermally treated MIBA

Data from: Kokalj et al. (2005); Kim et al. (2016); Lin and Lin (2006a); Lin et al. (2008a); Onori et al. (2011): Polettini et al. (2005); Polettini et al. (2009).



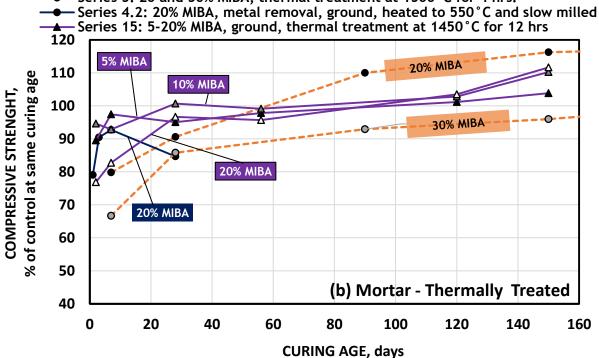


Figure 5.14: Compressive strength of mortars using MIBA subjected to (a) standard processing and (b) thermal treatment

Data from: Cheng et al. (2011b), Cheng (2012); Saccani et al. (2005); Tang et al. (2014), Tang et al. (2016); Whittaker et al. (2009a); Zhang and Zhao (2014).

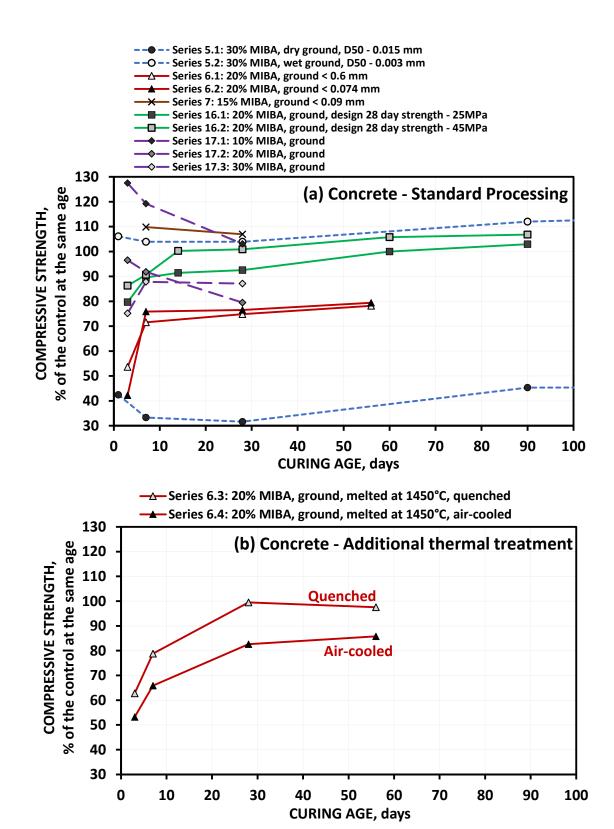


Figure 5.15: Compressive strength of concrete using MIBA subjected to (a) standard processing and (b) additional thermal treatment

Data from: Bertolini et al. (2004); Cheng et al. (2011a); Jaturapitakkul and Cheerarot (2003); Juric et al. (2006); Mangamma (2016)

The typical pozzolanic strength development trend of low early-age strength, but greater strength gain in the medium to late ages, is evident in many of the MIBA mixes in Figures 5.13, 5.14 and 5.15. This finding is also supported by Frattini and lime saturation pozzolanic activity tests measuring the amount of Ca(OH)₂ removed by MIBA, which showed that the ash met the pozzolanic activity requirements for use as a cementitious material and performed comparable to the coal fly ash and natural pozzolan [Series 10 and 11 and additional Series 18 (Fernandez et al., 1998), 19 (Filipponi et al., 2003) and 20 (Giampaolo et al., 2002)].

Compressive strengths with the standard processed MIBA are mostly well below those of the Portland cement mixes, particularly as the replacement level increased, though, surprisingly, the higher-strength concrete products [Figure 5.15 (a)] delivered the most positive results, comparable to the control in Series 5.1, 7 and 16. Furthermore, 28-day strengths similar to that of the control Portland cement paste have been achieved using MIBA produced from a 'light fraction' of municipal solid waste, which contained minimal metals. This demonstrates the capability of MIBA, if the inhibiting elements are taken away. In contrast, significant strength losses arose in some cases, Series 8, 11 and 12 (pastes), 1 and 4.1 (mortars) and 5.1 (concrete). This is primarily due to the damaging expansion behaviour that arose from the reaction of the metallic aluminium fraction in MIBA under alkaline conditions. Indeed, these large strength losses were symptomatic of all-around poor performance, and treatment of these ashes would be necessary prior to their use.

Chemical activation and thermal treatments have been applied to MIBA, though mostly with the aim of improving the mechanical properties of the paste, mortar or concrete mix, rather than specifically to prevent expansive behaviour. Chemical treatment (Series 11 and 12) altered the bottom ash mineralogy to promote greater formation of hydration products. CaCl₂ was the most effective activator, followed by CaSO₄, whilst additional testing showed that Na₂SiO₃·9H₂O, NaOH, Na₂SO₄, KOH and K₂SO₄ were less effective. Thermal processing converts MIBA into an amorphous slag with a higher degree of reactivity, with the additional benefits of decreasing the organic content, improving the material homogeneity and stabilising leaching behaviour. Melting at temperatures of 1450-1500°C enhanced the strength

performance with 20% MIBA to a level comparable to or beyond that of the non-MIBA mixes, at later ages. Quenching produced better results than air-cooling, because of the higher glass content resulting from the rapid cooling process.

As discussed previously, expansive behaviour caused excessive strength losses in some mix series, though it did not always occur with MIBA in cement products. It is of interest to determine which MIBA samples will present this problem. The total aluminium present can be straightforwardly determined; however, it is difficult to precisely measure the reactive metallic aluminium fraction. An indirect method has been developed by Kim et al. (2016) (Series 8), which measured the hydrogen gas generated by MIBA submerged in NaOH solution and then back-calculated the metallic aluminium fraction. Using MIBA and NaOH proportions representing the 10%, 20% and 30% MIBA replacement levels in cement pastes (Series 8), metallic Al contents of 0.02, 0.03 and 0.05 g/cylinder were calculated per MIBA contents of 56.52, 113.04 and 169.56 g in the cylinders. The metallic aluminium fraction and the associated volume of hydrogen gas generated was shown to increase as the MIBA level increased.

Treatment of this expansive behaviour has involved removing the reactive aluminium fraction and dissipating the expansive reactions before use. The methods used are as follows:

- Using a series of grinding + thermal treatments of MIBA (Series 4), it was found that thermal treatment (550°C–750°C) on its own did not significantly reduce the metallic aluminium content. However, when combined with a subsequent slow-speed grinding treatment of the fine (<63 μm) MIBA fraction, owing to the high ductility of the metallic aluminium when heated, these elements formed into larger plate shapes under the action of the ball milling and, as such, could then be sieved out, leading to significant strength improvements (Figure 5.14, Series 4.2 versus 4.1).
- Wet grinding of MIBA has also been effective in preventing damage in cement products
 (Series 5 and an additional Series 21; Carsana et al., 2016). During this grinding, MIBA is
 mixed with water to form a slurry. As the particles fragment, the metallic aluminium
 fractions become more exposed and, owing to the natural alkalinity of MIBA, expansive

reactions (formation of aluminium hydroxide and hydrogen gas) begin to develop. The rate of reaction is slower than that in cement mixes, owing to the lower ash alkalinity; however, with a sufficient wait time (a few days was sufficient for Series 5) before using the ash, the expansive reactions can be fully depleted.

Initial testing on the drying shrinkage and creep of mortar and concrete mixes has been consistent with the previous compressive strength results. When ground MIBA was used as a cement component, at contents up to 40%, increases in drying shrinkage and porosity accompanied the strength reductions (Series 1 and 2). The same trend was also visible in the creep results (series 2). However, after thermal treatment of MIBA, at a maximum temperature of 1450°C for 12 h, the reverse behaviour was recorded (Series 1). As a result of the lower porosity, greater pozzolanic reactivity and associated filling effects, mixes with thermally treated MIBA exhibited less drying shrinkage than Portland cement mixes.

Additional aspects of the mortar/concrete durability, covering porosity, absorption, corrosion, ingress of chlorides and sulphates and alkali aggregate reactivity, have been examined. The effects of MIBA on each property is outlined in Table 5.8. Most of the durability parameters are directly linked to the microstructure of the cement products and match previous trends visible for the compressive strength behaviour. It is found that the standard processed (ground) MIBA mostly had a negative effect on porosity, water absorption, corrosion resistance and chloride and sulphate ingress. However, thermal treatment led to improvements in all cases, and indeed, most of these MIBA mixes compared favourably to reference Portland cement mixes. The wet ground MIBA concrete, which previously exhibited good strength performance, also offered strong corrosion resistance and reduced chloride diffusion. The data indicate that, if strength with MIBA is comparable to that of Portland cement mixes, the additional pozzolanic activity of the ash and the associated filling effects, should ensure that the corresponding durability is comparable or greater. Alkali aggregate reactivity was also reduced with MIBA, arising from the Portland cement replacement and associated dilution of the mortar alkalinity.

Table 5.8: Effects of MIBA on the durability of paste, mortar and concrete mixes

SERIES	MIBA	NEGATIVE EFFECT	NEUTRAL EFFECT	POSITIVE EFFECT	
Series 8: Paste	Ground, 0- 30% MIBA	Increased voids			
Series 1.1: Mortar	Ground, 0- 40% MIBA	Increased gel and capillary porosity			
Series 1.2: Mortar	Thermal, 20% MIBA			Decreased gel and capillary porosity	sity
Series 3: Mortar	Thermal, 20- 30% MIBA			Lower porosity (particularly at late ages)	Porosity
Series 6.1/6.2: Concrete	Ground, 0- 40% MIBA	Increased gel and capillary porosity			
Series 6.3/6.4: Concrete	Thermal, 20% MIBA	Slightly Increased gel and capillary porosity			
Series 8: Paste	Ground, 0- 30% MIBA	Slightly higher than the control at late ages.			uc
Series 6.1/6.2: Concrete Series 6.3/6.4:	Ground, 0- 40% MIBA Thermal,			Decreased initial surface absorption Significantly decreased	Absorption
Concrete	20% MIBA			initial surface absorption	ΙΑ
Series 6.1/6.2: Concrete	Ground, 0- 40% MIBA	Decreased corrosion resistance			
Series 6.3/6.4: Concrete	Thermal, 20% MIBA		Corrosion resistance on par with the control		sion
Series 5.1: Concrete	Dry ground, 30% MIBA		Electrical resistivity comparable to control		Corrosion
Series 5.2: Concrete	Wet Ground, 30% MIBA			Significantly increased electrical resistivity	J
Series 6.1/6.2: Concrete	Ground, 0- 40% MIBA		Chloride permeability similar to control		
Series 6.3/6.4: Concrete	Thermal, 20% MIBA			Significantly decreased chloride permeability	ress
Series 5.1: Concrete	Dry ground, 30% MIBA	Increased chloride diffusion			Chloride Ing
Series 5.2: Concrete	Wet Ground, 30% MIBA			Decreased chloride diffusion	hloric
Series 3: Mortar	Thermal, 20- 30% MIBA			Significantly decreased chloride penetration	0
Series 3: Mortar	Thermal, 20- 30% MIBA			Significantly decreased sulphate penetration	Sulphate Attack
Series 3: Mortar	Thermal, 20- 30% MIBA	205), Chang et al. /2011a		Significantly reduced alkali agg expansion susceptibility	Alkali Agg

Data: Bertolini et al. (2004, 2005); Cheng et al. (2011a, b); Cheng (2012); Kim et al. (2016); Saccani et al. (2005)

5.3.3 Aerated Concrete

Aerated concrete production involves the addition of a foaming agent, such as aluminium powder, to a mix of cement, fine aggregate, water and potentially other constituents such as fly ash, to create expansive reactions from the formation of hydrogen gas, thereby producing a lightweight porous product. Aerated concrete is typically used in autoclaved blocks. The compressive strength of these blocks is low, with commonly available unit strengths from 2.9 to 8.7 N/mm² listed in BS 6073-2 (2008), whilst BS EN 771-4 (2011) stipulates that the declared strength must not be less than 1.5 N/mm². Lightweight properties are essential and a typical net density range from 300 to 1000 kg/m³ is outlined in BS EN 771-4 (2011). Thermal conductivity requirements may also be specified for these products.

Aerated concrete is an ideal outlet for MIBA, as the expansive behaviour arising from its metallic aluminium fraction can contribute positively to the desired foaming reaction and lightweight properties. Its silica content and pozzolanic properties can also contribute somewhat to the strength development.

It was of interest to first examine the hydrogen gas formation that occurs with MIBA in an alkaline environment. This involved the addition of MIBA to varying concentrations of alkaline solutions [0.0016–1 mol/L of NaOH/Ca(OH)₂] at temperatures from 40°C to 70°C and subsequently measuring the volume of hydrogen gas generated (Series 1, 2, and 3; Chen et al., 2014; Song et al. 2015/2016; Wang et al., 2016; Yang et al., 2015). The following findings emerged:

- The volume of hydrogen gas generated increased as the fineness of the MIBA increased.
 It can be inferred from this that for the other concrete-related applications, the use of MIBA as a cement component, in ground form, is likely to present more expansion problems compared with its use as an aggregate.
- The hydrogen gas volume increased with increasing temperature. It is generally recognised that the rate of reaction increases with increasing temperature.

- Hydrogen gas production increased as the conditions became more alkaline (increasing alkaline solution molarity), though the type of solution used, either NaOH or Ca(OH)₂, did not make a significant difference.
- The finest MIBA sample tested, which had an average particle size of 23.2 μm after grinding, was found to produce approximately 1% of the hydrogen gas generated by aluminium powder, per gram, under the same conditions. As such, in the aerated concrete mix design, this equates to replacing 0.01 g of the aluminium powder foaming agent with 1 g of MIBA.

Different approaches have been adopted with MIBA in aerated concrete. The ash has been used as a replacement for cement, coal fly ash/circulating fluidised bed combustion (CFBC) fly ash in mixes also containing other constituents, alkali solutions and water. The effects of MIBA were also compared to equivalent aluminium powder dosages. The mix designs adopted and the resultant density and strength performances of aerated concrete products are presented in Table 5.9.

As a cement replacement, MIBA led to increasing porosity and reductions in density and strength. The same behaviour was evident when the NaOH molarity increased, though the alkaline solution had a greater impact on strength, whilst MIBA had a greater effect on density. The replacement of coal fly ash/CFBC fly ash with MIBA yielded mixes with very low density, owing to the increased expansive reactions and greater porosity, though it also led to decreased strength. The strength-to-density ratios of MIBA mixes were below those of coal/CFBC fly ash mixes, though greater than those using aluminium powder foaming agent (0.01 g of Al used per gram of MIBA). As such, the inclusion of MIBA in combination with coal fly ash/CFBC fly ash in aerated concrete appears to be an ideal combination that offers the flexibility to achieve the desired lightweight properties, whilst retaining a strength-to-density ratio greater than that of mixes using the expensive aluminium powder foaming agent.

Table 5.9: Mix designs and density and strength results using MIBA in aerated concrete

MIX CONSTITUENTS AND PROPORTIONS RESULTS

(A) Series 1: Cement Replaced

Other variables: Alkaline solution type and molarity, L/S ratio

Cement	MIBA	Sand	Alkaline solution	L/S ratio	Density, kg/m ³	Comp Strength, MPa	
4	1	5	Water	0.175	1554	19.5	
4	1	5	0.01 mol/L NaOH	0.175	1512	16.0	
4	1	5	0.1 mol/L NaOH	0.175	1444	12.5	
4	1	5	1 mol/L NaOH	0.175	1324	5.7	
3	2	5	1 mol/L NaOH	0.35	1056	2.7	
2	3	5	1 mol/L NaOH	0.35	932	1.5	

(B) Series 2: Coal Fly Ash Replaced

Other variables: With and without Al powder

Cement	MIBA	Coal fly ash	Al powder	Lime /Gypsum	Water	Density, kg/m ³	Comp strength, MPa
10	0	67	0	20/3	65	1074	13.9
10	5	62	0	20/3	65	846 (↓ 21%)	9.7 (↓ 30%)
10	10	57	0	20/3	65	728 (↓ 32%)	8.4 (↓ 40%)
10	20	47	0	20/3	65	673 (↓ 37%)	7.2 (↓ 49%)
10	30	37	0	20/3	65	637 (↓ 41%)	6.1 (↓ 56%)
10	0	67	0.05	20/3	65	831 (↓ 23%)	7.7 (↓ 45%)
10	0	67	0.1	20/3	65	673 (↓ 37%)	6.5 (↓ 53%)
10	0	67	0.2	20/3	65	603 (↓ 44%)	4.1 (↓ 71%)
10	0	67	0.3	20/3	65	511 (↓ 52%)	2.9 (↓ 79%)

(C) Series 3: CFBC Fly Ash Replaced

Other Variables: None

Cement	MIBA	CFBC fly ash	Lime	Water	Density, kg/m ³	Comp strength, MPa
10	0	70	20	65	1116	18.1
10	5	65	20	65	868 (↓ 22%)	17.1 (↓ 5%)
10	10	60	20	65	799 (↓ 28%)	13.2 (↓ 27%)
10	20	50	20	65	656 (↓ 41%)	8.9 (↓ 51%)
10	30	40	20	65	613 (↓ 45%)	6.8 (↓ 63%)

L/S – liquid/solid, CFBC - circulating fluidised bed combustion, comp - compressive Data from: Chen et al. (2014); Song et al. (2015); Song et al. (2016); Wang et al. (2016); Yang et al. (2015).

Further testing has been carried out on the drying shrinkage of the mixes adopting MIBA as a partial replacement for coal/CFBC fly ashes (Series 2 and 3, refer to Table 5.9 for mix details). It was found that the drying shrinkage of MIBA mixes is higher than that of the control fly ash

mixes, owing to the increased ease of moisture loss associated with the higher porosity and the decreased resistance to movement associated with the lower strength. However, all MIBA mixes were within the targeted Chinese National Standard limit of 0.50 mm/m. For all mixes, the drying shrinkage increased in the early ages and then stabilised, which occurred after 15 and 20 days of drying time for MIBA mix Series 2 and 3, respectively.

5.4 Environmental Assessment

The use of MIBA in concrete offers environmental benefits from conserving natural resources, lowering the carbon footprint and reducing landfilling. Emissions arising from incineration and processing of the ash can be controlled to acceptable levels when adopting standard mitigation protocols. However, of primary concern, as established in Chapter 4, MIBA contains notable quantities of heavy metals. As such, the possible leaching of these elements into surrounding surface water, groundwater and soil must be addressed when assessing its suitability for use.

On the leaching of lightweight aggregates produced from sintered MIBA, sintered MIBA + clay, and MIBA + cement + coal fly ash + lime blends, it was found that in each case, heavy metal concentrations released were very low, at times below detectable limits (Series 5, 9 and 16; Table 5.5). The binder additions of clay and cement are particularly effective in encapsulating the harmful elements in the pellet microstructure, thereby reducing leaching.

On the use of MIBA as raw feed in cement clinker production, there are two concerns: emissions released from the kiln and the leachate released from the cement products. On the former aspect, though there is a scarcity of data on MIBA specifically, the outlook for secondary materials in general in cement production indicates that the high temperatures used, lead to the breakdown of toxic organic components and the immobilisation of non-volatile heavy metals, though volatilisation of elements such as mercury, cadmium and lead may increase the emissions of these components (Jung et al., 2004/2005; Jung and Osako, 2007; Kuo et al., 2003; Reijnders, 2007; Yang et al., 2003). However, cement rotary kilns have been often

retrofitted into MSW incineration facilities, which suggests that emissions from MIBA in cement manufacturing should be manageable.

Reductions in CO_2 emissions are also attained when using MIBA in cement clinker production, owing to the lower conversion from CaO_3 to CaO. Around 25 and 49 kg of CO_2 per 1000 kg of clinker have been saved using 5% and 10% of MIBA, respectively, as raw feed (Series 1; Table 5.6).

Testing the leachability of cement clinker manufactured with 8% MIBA in the raw feed (Series 2; Table 5.6), in accordance with the toxicity characteristic leaching procedure (TCLP) US EPA method 1311 (1990), showed that the released heavy metal concentrations were well within hazardous limits and only marginally higher than those of the control clinker. This demonstrates that the metals have been effectively stabilised within the cement matrix or vaporised in the clinkerisation process.

Results of various leaching tests on paste, mortar and concrete products incorporating MIBA are given in Table 5.10. With MIBA as an aggregate, leached element concentrations were generally comparable to those of the controls. However, there were cases in which the products were classified as non-hazardous, instead of inert, because of the leached concentrations of certain critical elements. This occurred with granular leaching tests on crushed products. In service, mortar and concrete mixes incorporating MIBA as an aggregate can be safely used, though restrictions should be adopted to avoid strongly unfavourable leaching conditions.

The use of MIBA as a cement component in pastes, mortar and concrete, was effective in immobilising its hazardous contaminants. The surface area available for leaching was reduced, whilst the pozzolanic activity of MIBA contributed to a denser microstructure, which further lowered the leachability of the products. Chromium, lead and nickel were identified as critical elements, as their leached concentrations, at times, can be capable of exceeding the limits for inert materials given by the European Community (2003).

Table 5.10: Test methods, mix designs and leaching results for pastes, mortars and concrete

MIX DETAILS	TEST METHOD	RESULTS
(A) MIBA as an Aggregate Componer	nt	
Concrete blocks: Mix proportions - 55% MIBA, 30% sand, 15% cement	Direct analysis of rainwater	All element concentrations were below detection limit except Pb, which exceeded the drinking water limit. However, this was due to contamination, not from MIBA.
Mortar: MIBA replaced 25% of sand, 1:2 cement/aggregate, w/c 0-5-0.6	EN 12457-2 (2002)	Leachate concentrations were within the corresponding inert limits, with the exceptions of Ba and Pb.
Concrete: 25% and 50% MIBA as fine and coarse aggregate replacements	NEN 7341 (1995)	In-service concentrations released were well below the regulatory drinking water limits.
Concrete: MIBA as 100% of mix aggregate, 1:4 cement/aggregate ratio, w/c 0.64	EN 12457-2 (2002); NEN 7375 (2004)	Granular and monolithic samples from concrete mixes with MIBA exhibited similar leachate concentrations to those of the controls. Dynamic leaching tests revealed a high accumulative release of Sb for MIBA samples.
Concrete: MIBA as 23% and 100% of the aggregate, 300 kg/m³ cement content	EN 12457-2 (2002); UNI 10802 (2004)	Granular: Pb and Ba leaching increased with MIBA. Monolithic: greatly reduced Ba with MIBA compared with granular results, but a lesser reduction of Pb.
Concrete: MIBA as a 100% coarse aggregate replacement, w/c 0.65, 350 kg/m³ cement content	NEN 7345 (2004)	Leached concentrations were within respective Flemish limits and at a similar level to the control mix results. Indeed, MIBA mix results were mostly < detectable limits.
Concrete: Unwashed & washed MIBA as 30% of the coarse aggregate, w/c 0.51, 320 kg/m³ cement content	TCLP method 1311 (US EPA, 1990)	Concentrations were lower than TCLP limits, though washing of MIBA led to further reduced releases.
(B) MIBA as a Cement Component		
Paste: MIBA as a 50% cement replacement, w/b 0.45	TCLP Method 1311 (US EPA, 1990)	All leached concentrations for MIBA mix samples were within the GB 5085.3 (2007) hazardous waste limits.
Paste: MIBA as a 30% cement replacement, w/b 0.25-0.30	TCLP Method 1311 (US EPA, 1990)	All leached concentrations with MIBA were within the GB 5085.3 (2007) hazardous waste limits.
Paste: MIBA as 20% and 40% cement replacements, w/b 0.4	CEN/TS-14429 (2005)	Metal immbolisation increased with curing time. Only Cr was released at a rate above the limit, though this was attributed to a sizeable contribution from the cement.
Paste: MIBA as 10% and 20% cement replacements, w/b 0.35	CEN/TS-14429 (2005)	Acid neutralisation tests showed that the buffering capacity decreased with increasing MIBA content.
Paste: 90% MIBA (untreated or thermally treated) with 10% Ca(OH) ₂ , w/b 0.20	NEN 7375 (2004)	Except for As and Cu, the mix leachate concentrations were below the detection limit (0.01 mg/L) after 64 days.
Mortar: 30% MIBA (with varying treatments) with 70% cement, w/b 0.7	NEN 7383 (2004)	Mortars with MIBA showed much lower mobility that original MIBA; thermal treatment of MIBA reduced the Cu leachability.
Concrete: MIBA as a 15% cement replacement, w/b 0.55	DIN 38414-4 (1984)	Leached contents of As, Ba, Cd, Co, Cu, Hg, Mo, Sb, Se and Zn were within the respective Slovenian inert waste limits. Cr, Ni and Pb were above these inert limits.

Data from: Dhir et al. (2002); Gines et al. (2009); Juric et al. (2006); Kokalj et al. (2005); Li et al. (2010); Li et al. (2012); Onori et al. (2011); Polettini et al. (2000); Qiao et al. (2008c); Roethel and Breslin (1995); Saikia et al. (2008); Sorlini et al. (2011); Tang et al. (2016); Van den Heede et al. (2015); Zhang and Zhao (2014);

5.5 Case Studies

After promising initial development work, the use of MIBA in concrete-related applications has progressed to the stage at which case studies have been undertaken in certain areas, to

provide valuable insight into its performance under real-world conditions. Case studies carried out using MIBA are described next, based on information from Bouchelaghem et al. (1997), Chandler et al. (1997), Cresswell (2007), Dhir et al. (2002), Environment Agency (2002), IEABioenergy (2000), Kikuchi (2001), Plumley and Boley (1990), Cosentino et al. (1995b), Roethel and Breslin (1995) and Wiles and Shepherd (1999).

<u>Lightweight aggregate production</u>

- Project by ABB Resource Recovery, Connecticut, USA: MIBA aggregates exhibited suitable lightweight properties and satisfied leaching requirements.
- Commercial process started in 1989 by Rolite Inc., New York, USA: over 400,000 tons
 of MIBA were used over 8 years. Aggregates were used in many applications, including
 landfill cover, fill material and subbase in road pavements.
- Pilot project setup at Aveley landfill, Essex, UK: three sizes of aggregate produced, 10
 mm for concrete blocks, 10–20 mm for coating and 40 mm for bulk fill.
- Project by RTAL, Tilbury, Essex, UK: artificial aggregates produced with up to 50% MIBA
 exhibited performance similar to that of the conventional coal fly ash + clay aggregate.

Raw feed in cement clinker manufacturing

- Pilot-scale project in Japan: MSW incinerated ash included as part of the raw feed in the production of 50 tons/day of cement clinker.
- Full-scale manufacturing beginning in 1991 in Tacoma, Washington, USA: fine fractions of MSW incinerated ash incorporated into the raw feed as a shale replacement.
- Full-scale manufacturing before the 1980s in Charleston, South Carolina, USA: MSW
 incinerated ash was used in an old wet process cement manufacturing plant, though it
 was discontinued, for undisclosed reasons, when the system was replaced by a modern
 dry process.

Mortar and Concrete

- Full-scale project at the Beaulieu Mines, France: plant setup for 1 year (1996–1997) to
 process MIBA to produce a stabilised mortar backfill material for reinforcement of the
 mines. The MIBA backfill material was used successfully, whilst treatment costs were
 offset by savings from acquiring and transporting natural materials and from
 landfilling.
- Full-scale demonstration on ready-mixed concrete in Dundee, UK: MIBA used as 25% and 50% of the coarse aggregate in concrete. Adjustments in the water content were required to satisfy the workability and mechanical requirements.
- Full-scale demonstration on precast concrete in Dundee, UK: MIBA was used as partial
 and complete aggregate replacements in full-size concrete blocks and lightweight
 thermal blocks.
- Artificial reef construction in 1988 in Conscience Bay, New York, USA: constructed with blocks made with 85% MIBA + 15% cement. No harmful environmental impacts or strength losses were recorded when re-examined.
- Raw material supplied by Ballast Phoenix to block producers around the UK: over
 15,000 tonnes of MSW incinerated ash were distributed between 1998 and 2000.
- Paving blocks used in a project in 1984 in Keilehaven, The Netherlands: use of 300,000 blocks containing MIBA as up to 40% of the coarse aggregate. Paving blocks with MIBA exhibited physical and environmental performance comparable to that of conventional blocks, when re-examined after 5 years of traffic loading.
- Blocks used in two buildings constructed in the early 1990s in Montgomery County,
 Maryland, USA: MIBA was used as aggregate in external non-load bearing wall blocks.
 An initial spalling problem on the block surface was rectified by effective ferrous metal separation treatment of MIBA.
- Full-scale boathouse constructed in Peekskill, New York, USA: 250 tons of MIBA and
 100 tons of mixed ash (MIBA + municipal incinerated fly ash) used as aggregate in the

production of the boathouse block walls. No negative environmental impacts or decline in mechanical performance was observed as a result of the MIBA or mixed ash.

Case studies incorporating MIBA in lightweight aggregate production demonstrated that the resultant aggregates were suitable as an alternative to commercial lightweight aggregate products for use in a range of construction applications. The ash has been successfully used as part of the raw feed in cement clinker production in full-scale operations in the past, although, with limited recent work, there are questions to answer regarding the economic viability of this use of MIBA in modern cement kilns. Case studies on mortar and concrete applications were mostly related to the production of masonry blocks. These MIBA blocks have been successfully used in a range of construction applications including in artificial reefs, non-load-bearing walls, paving units, residential foundations, external and internal walls of a boathouse and as shoreline revetment blocks.

5.6 Summary

This chapter analyses and evaluates the results on the use of MIBA as aggregate and cement components in a variety of applications covering cement clinker production, cement pastes, mortar, concrete, blocks, lightweight aggregate production, lightweight concrete and aerated concrete.

As an aggregate, MIBA leads to an increased water demand, which must be accounted for in mortar and concrete mixes. This effect is less when used as coarse compared to fine aggregate. Reductions in the mix density were incurred owing to the lower specific gravity of MIBA. Treatment of the bottom ash beyond sieving may be needed for some ash samples to limit negative effects on the strength and durability, by preventing damaging hydrogen gas expansive reactions, minimising organics and lowering the content of chlorides. The elastic modulus-to-compressive strength ratio decreased with MIBA, though it remained within the Eurocode 2 range, with lower replacement levels. Drying shrinkage and absorption increased for MIBA mixes, owing to higher porosity and lower stiffness. Heavy metals in MIBA were mostly effectively stabilized within the cement matrix, though strongly unfavourable leaching

conditions should be avoided. Overall, the ash can be suitable as a low-content aggregate component in mortar and concrete.

MIBA as an aggregate in concrete blocks reduced the unit weight, though generally to the medium-weight classification rather than lightweight. Spalling and staining arose at times, though they can be overcome by removing the ferrous and non-ferrous fractions in the ash. Despite some strength reductions, MIBA blocks satisfied the requirements for use in a range of applications: load-bearing/non-load-bearing, paving and interlocking blocks. Many full-scale projects have also been successfully undertaken with MIBA blocks.

Aggregates manufactured using MIBA achieved the target lightweight properties and exhibited water absorption comparable with that of Lytag, though with marginally lower compressive strengths. Their subsequent use as a natural aggregate replacement in lightweight concrete led to improvements in consistence and reductions in unit weight. Concrete compressive strength with MIBA lightweight aggregate fluctuated above and below that of the counterpart Lytag mixes, though all were below natural aggregate mixes. Elastic modulus and shrinkage strains showed the same trends. The higher porosity of MIBA lightweight concrete, compared with natural aggregate mixes led to high corrosion susceptibility. However, MIBA blends exhibited lower initial surface absorption values than Lytag mixes, and showed no negative effects on alkali silica reactivity.

As part of the raw feed in cement clinker manufacturing, MIBA can be incorporated at low contents around 5%, contributing to the required quotas of SiO_2 , CaO, Fe_2O_3 and Al_2O_3 and delivering performance comparable to that of the control cement clinker. At higher MIBA contents, the build-up of minor constituents of P_2O_5 , organics and metallics begins to suppress the formation of C_3S , thus affecting the setting behaviour and strength development. The use of MIBA also reduced the CO_2 emissions, whilst the leaching behaviour of the resultant MIBA cement clinkers was comparable to that of the controls.

When MIBA was used as a cement component in pastes, mortar and concrete, thermal and chemical treatments effectively enhanced its reactivity, leading to strength comparable to, and at times greater than that of Portland cement mixes, at later ages. In cases in which hydrogen gas expansive reactions arose, special thermal treatment + slow speed grinding or wet grinding techniques could be adopted to specifically remove the problematic metallic aluminium, which led to significantly enhanced performance with regard to mechanical strength, density, absorption, shrinkage and durability. The grinding required for its use as a cement component has a beneficial shaping effect which lessens the workability losses compared to those incurred as an aggregate. After further thermal treatment, MIBA mortars also exhibited greater flowability than the control. MIBA products showed expected lower early age strength and longer setting times. After appropriate treatment, products containing the ash performed comparable to or better than the control, at equivalent strengths, with regard to porosity, absorption, corrosion and chloride and sulphate ingress. Alkali aggregate reactivity was reduced with MIBA.

Aerated concrete is an ideal outlet for MIBA, as the hydrogen gas expansion arising from the material under alkaline conditions, can contribute towards the desired low-density, thus serving as a cheaper alternative to aerating agents, whilst its pozzolanic activity can support strength development. The material has been incorporated in varying mix designs as a replacement for cement, coal fly ash/CFBC fly ash, and delivered the desired reductions in density. Mixes containing MIBA as a partial coal fly ash replacement (with no aerating agent) also exhibited strength-to-density ratios that were superior to those of control fly ash + aluminium powder mixes.

CHAPTER 6

USE OF SEWAGE SLUDGE ASH IN CONCRETE

6.1 Introduction

The use of SSA in concrete is at a similar stage of development compared to MIBA, given that it has exhibited positive characteristics for potential utilisation, though its application has not yet been effectively exploited. As a potential cementitious material, the material again is not specifically named in standards such as EN 197 (2011). However, encouragement can be taken from the progress in recent years in permitting other secondary materials such as GGBS and coal fly ash as cement admixtures. Recycled aggregate has also been permitted for use as sand and gravel replacements, though the finer particle size distribution of SSA, as shown in Figure 4.1, dictates that its use will be directed towards filler and fine aggregate size fractions. When natural resources are used, the environmental assessment is generally not a pressing concern, though with a material such as SSA, its acceptance in concrete, must be predicated on ensuring that no adverse leaching of harmful constituents arises from its use.

Owing to the smaller quantity of SSA produced worldwide compared with MIBA, the overall capacity for incorporation of the former incinerated ash as a construction material is less. Nevertheless, there is a need to develop sustainable construction materials from all available secondary and recycled sources, and concrete applications appear to provide an outlet whereby the suitable characteristics of SSA can be taken advantage of.

This chapter analyses and evaluates the data on the use of SSA, predominantly as a cementitious component after grinding, and also as a filler and fine aggregate, in a range of concrete related applications. The outlets covered include cement clinker production, lightweight aggregates, mortars, normal-weight concrete, blocks, aerated concrete and foamed concrete. Environmental impacts arising from these uses of SSA and relevant case studies are also examined.

6.2 Use as a Cement Component

6.2.1 Raw Feed in Cement Clinker Production

The oxide composition of SSA, with average contents of 33% SiO₂, 14% CaO, 14% Al₂O₃ and 11% Fe₂O₃ (Chapter 4), indicates that the ash can contribute positively to the formation of the main clinker phases during cement manufacturing. This use also offers the accompanying benefits of reduced consumption of natural resources and CO_2 emissions released.

As part of the raw feed for cement clinker production, SSA has been explored at low contents from 1% to 11%, as outlined in Table 6.1, alongside various combinations of the traditional limestone, clay and sand components and secondary materials including coal fly ash, copper slag, ferrate waste, water purification sludge ash (WPSA) and industrial wastewater sludge ash (IWSA). The constituent contents in each blend were controlled, guided by recommended limits for the lime saturation factor (LSF), hydraulic modulus (HM), silica ratio (SR) and alumina ratio (AR) parameters, to ensure the main clinker phases formed were consistent with conventional Portland cement clinker.

Table 6.1: Constituent contents of blends using SSA in cement clinker production

		CONSTITUENTS, %													
MIX SERIES	SSA	Limestone	Clay	Ferrate	Copper Slag	Sand	Coal Fly ash	MIBA	WPSA	IWSA					
1.1	1.1	61.8	6.0		0.3			30.4							
2.1	2.0	75.7			1.3	15.7	5.3								
2.2	4.0	74.8			0.8	15.0	5.3								
2.3	8.0	73.0			0.2	14.3	4.5								
3.1	6.8	80.0		2.0					9.7	1.5					
3.2	8.5	80.1		1.7					8.7	1.0					
3.3	9.3	79.4		2.1					6.2	3.0					
4.1	4.2	81.5		1.9					12.5						
4.2	4.7	80.4		1.9					13.0						
4.3	9.0	79.6		2.1					9.3						
5.1	4.9	80.6		2.2					12.4						
5.2	6.5	81.3		1.9					10.3						
5.3	11.4	79.2		2.3					7.2						

Data from: Kikuchi (2001); Lam et al. (2010a); Lin and Lin (2004, 2005, 2006b); Lin et al. (2005b)

Results on the oxide composition, clinker parameters (HM, LSF, SR and AR) and clinker compounds are presented in Table 6.2, for clinkers incorporating SSA, along with control Portland cement clinkers. Similar to the behaviour evident with MIBA, at the lower end of the tested SSA contents, the clinkers exhibited chemical compositions comparable to those of the controls. However, despite the main oxide contents being regulated, the build-up of minor elements in the blend, in particular the high P₂O content of SSA, began to alter the formation of the clinker compounds. This led to the suppression of C₃S, which is the clinker phase primarily responsible for setting behaviour and early strength development.

Trends evident in the data on setting behaviour, compressive strength and hydration products for SSA clinker series 3, 4 and 5 align with the P_2O_5 and C_3S results in Table 6.2. With the exception of series 4.1, with the lower 4.2% SSA content, the cement mixes exhibited lengthened setting times, and indeed, the final setting times for series 3.3 and 4.3, containing higher SSA contents of 9% and 9.3%, were approximately double that of the control Portland cement clinker. Apart from mixes with the highest tested SSA contents of 9.3%, 9.0% and 11.4% (series 3.3, 4.3 and 5.3), the rest of the later age compressive strength results were comparable to those of the controls, though with lower early age strengths.

This performance suggests that the content of SSA in the cement clinker raw feed should be limited to reduce excessive negative effects on setting and strength development. A limit of around 5% SSA is recommended, though this will generally be controlled by the phosphorus content in the feed, whilst the SO₃, organics and metal contents may also need to be considered. The American ASTM C150 (2017) standard on cement limits the allowable MgO and SO₃ contents, and indeed, with SSA cements, it is recommended to adopt an additional P_2O_5 limit in the range from 0.5% to 1%. Assessment of the P_2O_5 , SO₃ and loss on ignition of SSA samples should be undertaken when evaluating the ash and samples that greatly exceed the mean contents of 15% P_2O_5 , 3% SO₃ and 4.3% LOI (from Chapter 4) would not be favourable for this use in cement clinker production.

Table 6.2: Chemical composition of cement clinkers produced using SSA in the raw feed

MIX	CCA 0/		CEMENT CLINKER OXIDE COMPOSITION, %								CLINKER PARAMETERS			CEMENT COMPOUNDS, %						
SERIES	SSA, %	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	P ₂ O	SO₃	Alkalis	F- CaO	Cl	LOI	НМ	LSF	SR	AR	C₃S	C ₂ S	C ₃ A	C ₄ AF
1.1	1.1	17.7	9.4	3.3	62.4	1.80	1.90	1.40	-	1-5	0.5	-	2.1	1.0	1.4	2.8	60.0	60.0	-	-
2.0 (PC)	0	23.3	5.1	3.5	66.2	0.83	0.17	0.27	0.23	0.6	0	-	2.1	0.9	2.7	1.5	50.0	27.3	7.6	10.8
2.1	2	23.7	4.9	3.2	65.2	0.83	0.58	0.18	0.52	2.2	0.01	-	2.1	0.9	2.9	1.5	38.9	31.4	7.5	9.8
2.2	4	23.8	4.8	3.5	65.2	0.74	0.92	0.30	0.34	3.8	0.01	-	2.0	0.9	2.9	1.3	30.9	32.6	6.6	10.8
2.3	8	23.0	4.6	2.8	65.6	0.87	1.58	0.93	0.61	8.8	0.01	-	2.2	0.9	3.1	1.6	19.3	22.4	7.3	8.5
3.0 (PC)	0	20.0		3.4	63.2	2.31	nd		0.56	0.2	-	-	2.2	1.0		1.6	51.0	23.2	8.2	10.3
3.1	6.8	23.1		3.5	63.2	1.17	0.50	0.41		0.3	-	-	1.9	0.8	2.2		26.7	46.1	13.4	10.5
3.2	8.5	21.1	7.6	3.6	64.6	1.28	0.48	0.38	0.24	0.3	-	-	2.0	0.9	1.9	2.1	45.2	26.6	14.0	10.9
3.3	9.3	22.5	8.3	4.6	31.2	1.11	0.85	0.45	0.18	0.2	-	-	0.9	0.4	1.7	1.8	14.0	54.1	14.2	14.1
4.0 (PC)	0	20.0	5.4	3.4	63.2	2.31	nd	2.03	0.56	0.2	_	0.85	2.2	1.0	2.3	1.6	51.0	23.2	8.2	10.3
4.1	4.2	20.9	6.6	3.4	65.2	1.17	0.21	3.51	0.56	0.4	-	0.93	2.1	1.0	2.1	2.0	56.9	17.1	11.8	10.2
4.2	4.7	21.2	7.1	3.6	64.8	1.28	0.46	3.24	0.72	0.3	-	0.94	2.0	0.9	2.0	2.0	48.7	24.2	12.8	10.8
4.3	9	23.2	6.4	3.7	63.1	1.11	0.75	3.27	0.26	0.2	-	0.96	1.9	0.8	2.3	1.7	31.7	42.5	10.7	11.2
5.0 (PC)	0	21.7	6.5	3.2	63.7	1.90	nd	2.20	0.30	0.3	-	0.93	2.0	0.9	2.2	2.0	46.7	27.3	11.8	9.7
5.1	4.9	21.2	7.1	3.6	64.8	1.28	0.46	0.34	0.32	0.3	-	0.96	2.0	0.9	2.0	2.0	48.7	24.2	12.8	10.8
5.2	6.5	20.9	6.6	3.4	65.2	1.17	0.21	0.14	0.56	0.4	-	0.93	2.1	1.0	2.1	2.0	56.9	17.1	11.8	10.2
5.3	11.4	23.2	6.4	3.7	63.1	1.11	0.75	0.37	0.26	0.2	-	0.94	1.9	8.0	2.3	1.7	35.6	38.4	10.7	11.2

nd - not detected. F-CaO - free CaO

Alternatively, an integrated approach involving the pre-treatment of SSA to extract the phosphorus fraction, which can be utilised as a valuable agricultural resource, and the subsequent use of the ash in cement clinker production appears to be a complementary option to explore. However, questions around the economic and practical viability of this combined approach would have to be answered.

6.2.2 Paste, Mortar and Concrete

As a potential cement component in pastes, mortar and concrete, the oxide composition and amorphous content of SSA (from Chapter 4) indicate that the material may possess a degree of reactivity when combined with cement. Grinding of SSA is required, though to a lesser extent than for MIBA, to convert the material to the required fineness, whilst the ash does not initially appear to present expansion problems associated with a reactive metallic aluminium fraction. Full assessment of the pastes, mortars and concrete products made with SSA, in their fresh and hardened states, is given below.

Consistence (Workability)

The effects of SSA, in ground form, as a partial cement replacement on the flow and slump values of mortar and concrete mixes are presented in Figure 6.1 (a) and (b), respectively, along with details on the water/binder ratio (w/b) and cement type. Decreases in the mortar flow spread and concrete slump, corresponding to reduced workability, are evident with the inclusion of SSA. The high water absorption of SSA (approximately 18%, from Chapter 4) is considered to be the main cause, whilst its irregular particle shapes with rough surface texture also contribute to this behaviour.

Reduction rates in the mortar flow and concrete slump on the order of 7% and 14%, respectively, per 10% replacement level, have been calculated from the linear trend lines in Figure 6.1. Regarding the difference in the calculated rates of decrease between the two methods, it must be considered that the tests in question are used to broadly categorise the consistence of the product, rather than for an exact measurement. As such, a small disparity between the rates calculated for the slump and flow is not unexpected.

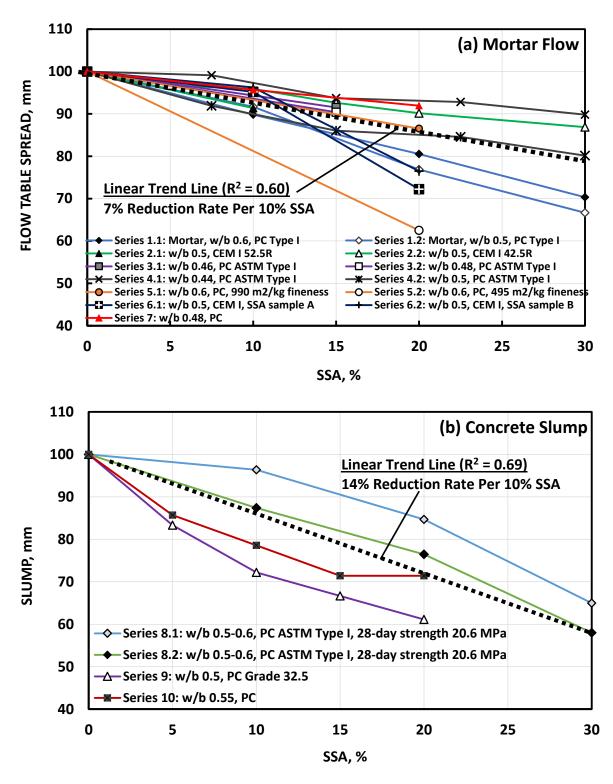


Figure 6.1: Effects of SSA as a cement component on (a) mortar flow and (b) concrete slump w/b – water/binder, with binder including cement plus SSA.

Data from: Chang et al. (2010); Garces et al. (2008); Halliday et al. (2012); Ing et al. (2015); Monzo et al. (1996); Monzo et al. (2003); Pan et al. (2003b); Pinarli (2000).

These data provide a useful benchmark of the performance with SSA as a direct cement replacement; however, in practice the mix should be adjusted to accommodate the characteristics of the new material, perhaps by using a water-reducing admixture. Indeed, in case studies undertaken as part of a large project in Denmark (Damtoft et al., 2001; Svensson et al., 2008 and Jensen, 2008), this approach of using superplasticiser alongside SSA was successfully adopted to achieve the desired concrete slump. An alternative option is to incorporate SSA alongside coal fly ash, which is known to benefit the workability, owing to the ball bearing effect of its spherical particles. Flow results showing the effect of coal fly ash, with respect to the control, on mortars containing up to 10% SSA as a cement component, are shown in Figure 6.2. It is evident that with SSA in combination with coal fly ash, the flow losses are counteracted, resulting in a mortar with workability greater than that of the control Portland cement mix, albeit less than with the coal fly ash replacement alone.

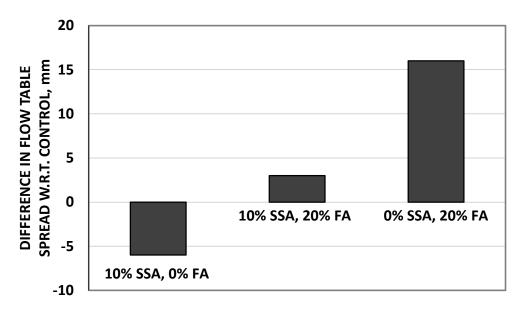


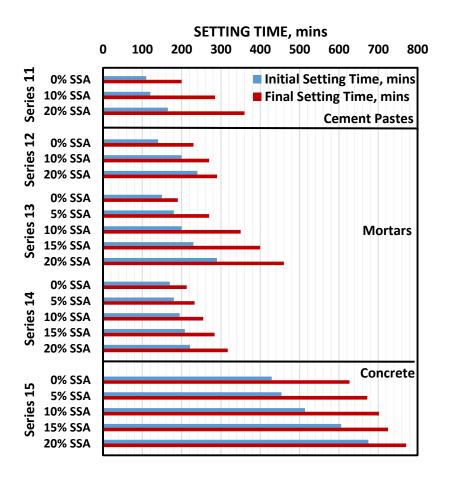
Figure 6.2: Consistence of mortars containing a combination of SSA and coal fly ash as cement components, with respect to the control Portland cement mix

FA – coal fly ash. Data from: Paya et al. (2002)

Setting Time

Setting time results for mixes containing SSA as a cement replacement are presented in Figure 6.3. This shows that the ash increases both the initial and the final setting times in all mixes. Average rates of increase of 34% and 43% for the initial and final setting times were calculated

for 10% SSA. Whilst pozzolanic materials are generally known to be slower to set, an additional direct comparison of separate cement pastes using SSA and coal fly ash (Series 11) showed that SSA causes greater retardation in setting time, attributed to its high phosphorus content, which interferes with the hydration products formed. Whilst engineers should be aware of this retardation effect and evaluate its positives and negatives in specific applications and climate conditions, the adoption of SSA per se is unlikely to affect the requirements of EN 197 (2011), specifying an initial setting time of greater than 45 min.



Cement pastes: Series 11 - w/b 0.25, CEM I 42.5R. **Mortars**: Series 12 - Blend of 1:1 SSA: coal fly ash used as cement replacement, w/b 0.7, PC ASTM C150 Type I; Series 13 - w/b 0.5, Ordinary PC; Series 14 – w/b 0.5, CPJ45 PC (includes limestone). **Concrete**: Series 15: w/b 0.5, PC grade 32.5R.

Data from: Lin et al. (2008c), Naamane et al. (2016), Piasta and Lukawska (2016), Pinarli (2000) and Pinarli and Kaymal (1994).

Figure 6.3: Effects of SSA as a cement component on the setting behaviour of pastes, mortar and concrete mixes

Pozzolanic Activity

The pozzolanic activity of SSA has been assessed using Frattini and saturated lime tests and thermogravimetric analysis, by directly measuring the quantity of Ca(OH)₂ fixed. Indirect strength activity index (SAI) tests have also been used to compare the strengths achievable with SSA under specific conditions with corresponding control Portland cement results. Strength requirements for coal fly ash in concrete, in European (EN 450, 2012) and American (ASTM C618, 2015) standards, are specified based on the SAI method.

Strength activity index results for SSA obtained according to methods in BS EN 196-1 (2005) and ASTM C311 (2013) are shown in Figure 6.4 (a) and (b), respectively, and values obtained from other mortar and concrete mixes are shown in Figure 6.4 (c) and (d), respectively. Accompanying details on the coal fly ash SAI test specifications, covering the replacement level and SAI requirements, are also provided, as a point of reference.

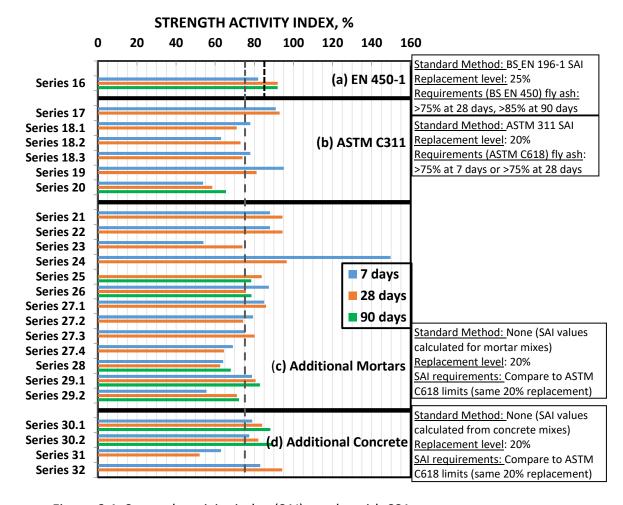


Figure 6.4: Strength activity index (SAI) results with SSA as a cement component

Data from: Baeza et al. (2014); Baeza-Brotons et al. (2015); Chang et al. (2010); Chen and Poon (2016); Coutand et al. (2006); Donatello et al. (2010a); Fontes et al. (2004); Garces et al. (2008); Halliday et al. (2012); Ing et al. (2015); Luo et al. (2009); Pinarli (2000); Pinarli and Kaymal (1994); Saikia et al. (2006); Tempest and Pando (2013); Tseng and Pan (2000).

Figure 6.4 shows that with the standard tests, SSA generally satisfied the reactivity requirements for coal fly ash, with SAI values greater than 75% and 85% at 28 and 90 days, respectively, given in EN 450 (2012) and greater than 75% at 7 or 28 days given in ASTM C618 (2015). For the additional mortar and concrete mixes with 20% SSA, the majority satisfied the equivalent ASTM C618 (2015) limit, though the lower results achieved at times suggest that SSA is somewhat less reactive than coal fly ash. EN 197 (2011) allows up to 35% coal fly ash as a cementitious component, though for SSA, a lower maximum limit should be adopted. The higher strength development at later ages that generally occurs with pozzolanic materials is also evident in many SSA mixes in Figure 6.4, from the increasing SAI values at later curing times.

Additional Frattini, saturated lime and thermogravimetric analysis tests are not referred to in the standards, though they offer a more direct measure of pozzolanic activity. The quantities of Ca(OH)₂ fixed by SSA place the material at a level comparable to that of coal fly ash (Baeza et al., 2014; Baeza-Brotons et al., 2014; Donatello et al., 2010a and Naamane et al., 2016).

Compressive Strength

The compressive strength performance of mortar and concrete mixes, using 28-day results, is presented in Table 6.3. Relative values as a percentage of the Portland cement mix at the same age are also given, with colour added to highlight the trends occurring. Table 6.3 shows that the strength of mortar and concrete mixes decreased as the ash content increased. An accompanying graph combining the 28 day results for all the mix series is given in Figure 6.5 On average, a rate of strength loss reaching around 1% per 1% SSA replacement level, at 28 days curing, is expected, though the lower R² indicates that there is a degree of scatter present in the measured strength results.

Table 6.3: Effect of SSA on 28 day mortar/concrete compressive strength

MIV	/o	28-DAY COMPRESSIVE STRENGTH, MPa (% Control)								
MIX	w/c	5% SSA	10% SSA	15% SSA	20% SSA	30% SSA				
Series 33.1	0.5		57.5 (93%)		50 (81%)	42 (68%)				
Series 33.2	0.7		35.5 (86%)		29.5 (71%)	23.5 (57%)				
Series 34	0.5		33.5 (88%)		31.9 (84%)	23.5 (62%)				
Series 35.1	0.5		37 (95%)		32 (82%)	27 (69%)				
Series 35.2	0.5		35 (95%)		28 (76%)	24 (65%)				
Series 36	0.5				37.4 (92%)					
Series 37.1	0.5		33.3 (95%)							
Series 37.2	0.5		35.5 (101%)							
Series 38	0.77				16 (62%)					
Series 39	0.5		39 (95%)	40.6 (99%)	39.5 (97%)	37.1 (91%)				
Series 40	?		50 (95%)		39 (74%)	25.8 (49%)				
Series 41.1	0.5		47 (78%)		43 (71%)					
Series 41.2	0.5		49.5 (82%)		44 (73%)					
Series 41.3	0.5		48 (79%)		44.5 (74%)					
Series 42	0.55	33.9 (110%)	25.2 (84%)	23.2 (77%)	15.7 (52%)					
Series 43	0.5-1.0		33 (77%)		27 (63%)	21 (49%)				
Series 44	0.6		40 (85%)		37 (79%)					
Series 45	0.5	29.7 (107%)	26.7 (96%)	24.4 (88%)	26.2 (94%)					
Series 46	0.5	44.7 (103%)	43.7 (101%)	41.9 (97%)	40.9 (95%)					
Series 47	0.6		35.9 (103%)	13.9 (40%)						

Green - >100% of control, Brown 90-100%, Orange - 80-90%, Red < 80%.

Data from Alcocel et al. (2006); Baeza et al. (2014); Chang et al. (2010); Chen and Poon (2016); Chin et al. (2016); Dhir et al. (2002); Fontes et al. (2004); Garces et al. (2008); Halliday et al. (2012); Ing et al. (2015); Maozhe et al. (2013); Piasta and Lukawska (2016); Pinarli (2000); Pinarli and Kaymal (1994); Yu et al. (2016).

Data on the effect of SSA on strength development with age, up to 90 days, are presented in Table 6.4. The results are given as a range of the control mix, along with the mean strength in parentheses, e.g., with 10% SSA at 3 days, the strength varied from 70% to 113% of the control, with an average value of 90% of the control. As is typical with pozzolanic materials, the disparity between the SSA and the control Portland cement mixes tends to be greater at early ages, though SSA mixes achieve greater late age-strength development.

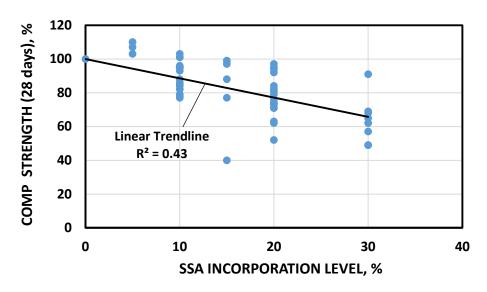


Figure 6.5: Combined data on the effect of SSA content on mortar/concrete strength

Table 6.4: Effects of SSA on mortar/concrete strength development with age

SSA, %	COMPRE	SSIVE STREN	GTH RANGE (MEAN), % C	CONTROL
	3 days	7 days	28 days	56 days	90 days
10	70-113 (90)	85-113 (97)	79-103 (92)	90-93 (92)	85-105 (95)
20	55-100 (73)	61-97 (81)	55-97 (78)	78-95 (85)	79-98 (84)
30	35-93 (66)	41-91 (66)	49-91 (66)	71-73 (72)	59-76 (68)

Note: Values in parentheses represent the mean of the range, as a percentage of the control

As a direct cement replacement, long-term compressive strengths approaching that of the control mix are achievable with SSA contents up to 10%. With higher contents, modifications to the mix design are recommended, to achieve more effective performance. This can include the use of superplasticiser, which allows the w/c to be lowered, to achieve improved strength whilst maintaining the same workability. With this approach, 28-day strengths similar to those of Portland cement mixes have been achievable in many cases with SSA (Coutand et al., 2006; Cyr et al., 2007a; Fontes et al., 2004; Monzo et al., 1996 and Monzo et al., 1999a).

Alternatively, using a combination of SSA + coal fly ash, at contents of 20% each (per mass of cement) (Paya et al., 2002), the compressive strength, at 28 days cured at 40°C to accelerate the gain, was comparable to that of the Portland cement mixes, which was a noteworthy improvement from SSA as the sole cement replacement. The use of SSA and coal fly ash

together is also promising from the sustainability point of view. The aforementioned mix, with secondary materials accounting for 40% of the cement, would fit into the CEM IV or CEM V category in EN 197 (2011), if SSA became a recognised cementitious component.

Similar positive results are expected with GGBS and SSA combinations. Cement kiln dust (CKD) has also been used as an activator of coal fly ash to improve strength (Daous, 2004) and similar benefits may be gained from this approach with SSA. Silica fume or metakaolin with SSA is an alternative option for high strength applications. Of these two, silica fume is better matched chemically with SSA, as the alumina in the ash would compensate for the shortage in silica fume. Minor additions of nano-materials have been effective in upgrading SSA products, due to improvements in the paste density and microstructure (Lin et al., 2008b; Lin and Tsai, 2006 and Luo et al., 2014). Calcination of SSA, at temperatures from 1000°C to 1200°C (Tseng and Pan, 2000), to enhance its amorphous content and reactivity, has also been effective in delivering SAI values similar to those of Portland cement with SSA, though it is a less environmentally favourable option because of the energy requirements.

Flexural Strength

Analysis of the data from a wide range of mix designs with SSA contents from 0% to 60% (by mass of cement) and curing ages from 3 to 90 days revealed that flexural strength mirrored the compressive strength behaviour. Reductions were evident as the SSA content increased, though the difference compared to the control became proportionally less with age.

Further comparison of the relationship between the flexural and compressive strength of SSA mixes and that of the control Portland cement mixes is presented in Figure 6.6. The compressive-to-flexural strength ratio for SSA mixes matches quite well with that of the non-SSA mixes, and indeed, the power trend lines plotted for the two datasets were very similar. As such, for practical purposes, existing empirical relationships between compressive and flexural strength should remain valid with SSA.

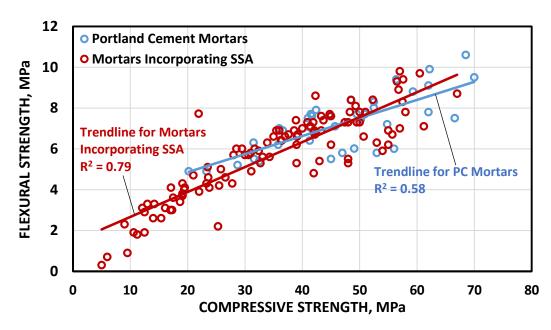


Figure 6.6: Comparison of the relationship between compressive and flexural strength for mixes with SSA and the control Portland cement mixes

Data from: Alcocel et al. (2006); Cyr et al. (2007a); Garces et al. (2008); Maozhe et al. (2013); Monzo et al. (1996, 1997, 1999a, 1999b); Paya et al. (2002) and Pinarli and Kaymal (1994).

Elastic Modulus

Initial data obtained on the elastic modulus of a concrete mix series using SSA as a cement replacement at contents from 0% to 20%, at 5% intervals, with w/c ratios ranging from 0.5 to 1.1 (Geyer et al., 2002), are somewhat inconsistent. With the remarkably high w/c ratio of 1.1, SSA increased the concrete elastic modulus, whilst at the more reasonable w/c ratio of 0.5, the opposite effect was observed. The elastic modulus and the strength are affected by many of the same factors and given the previous strength reductions with SSA, combined with the porous characteristics of the material, a drop in the elastic modulus is anticipated, despite the aforementioned varying performance.

<u>Shrinkage</u>

Regarding the effect of SSA as a cement component on the shrinkage of mortar and concrete mixes, a concise description of this work and the emerging findings are presented in Table 6.5. Both minor increases and decreases in the shrinkage strain have been recorded in the SSA mixes. However, for practical purposes, low SSA contents up to 20% are not expected to have any major effects on this aspect of the load-independent deformation properties.

Table 6.5: Results on the shrinkage of mortar and concrete mixes containing SSA

MIX	WORK UNDERTAKEN AND RESULTS
Series 48	Tests: Mortar. PC. 20% SSA. w/c 0.48. Curing at 23°C at 50% RH for up to 112 days. Results: Drying shrinkage ratios up to 820 x10 ⁻⁶ (0% SSA) and 1020 x 10 ⁻⁶ (20% SSA) Verdict: SSA increased the drying shrinkage
Series 49	Tests: Mortar. CEM 1 52.5R. 10% SSA. w/c 0.5. 80 days. Immersion cured. RH 65%, 100%. Results: Change in length for SSA and control mixes is comparable in both RH conditions. Verdict: SSA did not cause a significant difference to the mortar shrinkage.
Series 50	Tests: Concrete. PC. 5, 10, 15, 20% SSA. w/c 0.6. Immersion cured. Up to 28 days. Results: Shrinkage strains of 855, 839, 815, 820, 765 x 10^{-6} with 0, 5, 10, 15, 20% SSA. Verdict: Very slight shrinkage reduction with SSA, but not a major effect.
Series 51	Tests: Mortar. CEM 1 52.5 R. 2 mixes: 75% PC 25% MK, 75% PC 22.5% MK 2.25% SSA. Curing: 20°C at 50% RH for 1-110 days. Results: Total shrinkage of 950 μ m/m (0% SSA) and 1070 μ m/m (2.25% SSA) Verdict: Minor increase in total shrinkage with low SSA replacement level.

Data from: Chen and Poon (2016), Cyr et al. (2007b), Garces et al. (2008), Tay (1987)

RH – relative humidity, MK – metakaolin, PC – Portland cement

Durability

Durability testing of SSA-mortar and concrete mixes has dealt with the permeation properties (absorptivity, permeability and porosity), corrosion resistance, susceptibility to carbonation and sulphate attack. Based on the same approach adopted previously with MIBA (Table 5.8), the impacts of SSA as a cement component, on each property, have been categorised in Table 6.6.

Increases in the porosity of the SSA mortar and concrete mixes were evident, which can be traced back to the porous characteristics of the ash. Testing mostly focused on the total porosity, with the exception of mix series 52, in which the distribution of different pore sizes was measured using mercury intrusion porosimetry. This test revealed that, whilst SSA increased the volume of large-capillary pores ($0.05-10~\mu m$), which play an important role in strength development, the small capillaries ($0.0025-0.01~\mu m$) made up a higher proportion of the overall pores in the ash concrete mixes, compared to the control Portland cement mix. It was established from Series 55 that CEM II-B/M (V-LL) (Portland composite cement) was more favourable to porosity, compared with CEM I (Portland cement), which further strengthens the case for the use of secondary materials such as coal fly ash alongside SSA.

Table 6.6: Effect of SSA on the durability of mortar and concrete mixes

SERIES	SSA	NEGATIVE EFFECT	NEUTRAL EFFECT	POSITIVE EFFECT
				2 2 2 2 2 2 7
Porosity Series 52: Concrete.	5, 10%	Large capillaries increase:		
w/c 0.5. PC+LS	3, 10%	22% (control), 28-31% (SSA)		
Series 53: Mortar. w/c	10, 20,	Increased porosity. Relative		
0.5, 0.7. CEM I 52.5.		rise of 7-82% (10-60% SSA)		
Series 54: Concrete.		Minor porosity increase:		
w/c 0.5. PC+LS	3, 10%	5.1% (control) to 5.4% (SSA)		
Series 55. Mortar. w/c	10, 20,	Relative increase of 10, 16,		
0.5. CEM I & II.	30%	25% with 10, 20, 30% SSA		
Absorption/Absorption		2570 With 15, 25, 5570 5570		
Series 56: Concrete.	5, 10,			Decreased absorption:
w/c 0.5. PC 32.5.	15, 20%			6.2% (control) to 5.0-5.6%
Series 57 - Concrete.	5, 10,		Absorption ≈ 6% with	
w/c 0.5. PC	5, 10, 15, 20%		0-15% SSA	reduction with 20% SSA
Series 58: Concrete.	10%		0 13/0 33A	Decrease in absorption of
w/c 0.5. CEM II/B-L	10/0			appr. 60% with SSA
Series 52: Concrete.	5, 10%		Minor absorptivity	Minor decreases in
w/c 0.5. PC+LS	3, 1070		fluctuations with SSA	
Series 54: Concrete.	5, 10%		Tractadions With 557	Absorptivity with SSA ≈
w/c 0.5. PC+LS	3, 1070			half the control value
Permeability				
Series 56 - Concrete.	5, 10,			Decreased permeability
w/c 0.5. PC 32.5.	15, 20%			constant: 1.5 x10 ⁻⁸
.,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,				(control), 1-7 x10 ⁻⁹ (SSA)
Chloride Ingress				
Series 52: Concrete.	5, 10%			Chloride pen. (current)
w/c 0.5. PC+LS	,			reduced: 1656 C (control)
·				to 891-1161 C (with SSA)
Corrosion Resistance				
Series 53: Mortar. w/c	10, 20,	Increased Icorr with 30 & 60%	I _{corr} ≈ control value	
0.5, 0.7. CEM I 52.5	30, 60%	SSA	with 10%, 20% SSA	
Carbonation				
Series 53: Mortar. w/c	10, 20,	Increased carbonation	Carbonation	
0.5, 0.7. CEM I 52.5		corrosion with 20%+ SSA	corrosion ≈ control,	
0.0, 0 0 0	00,007	20,000,000,000,000,000,000,000,000,000,	with 10% SSA	
Sulphate Attack				
Series 55: Mortar. w/c	10, 20,		No sulphate	
0.5. CEM I & II	30%		expansion with SSA	
Alkali Aggregate Read	ctivitv			
	10, 20%			Expansion 0.27% (control)
Series SS, Midital, W/C	,,			[
0.48. PC.				dropped to 0.12% and 0%,

PC – Portland cement, LS – Limestone, pen. – Penetration, I_{corr} - Instantaneous corrosion rate. Data from: Alcocel et al. (2006); Baeza-Brotons et al. (2014); Chen and Poon (2016); Fontes et al. (2004); Fontes et al. (2016); Garces et al. (2008); Pinarli (2000); Tay (1987).

The aforementioned pore size distribution changes associated with SSA and the pozzolanic reactions may explain the reductions in absorption and permeability evident in the concrete, which are in contrast to the higher absorption of SSA itself. Average absorption reduction rates of 6% and 10% were calculated for the 10% and 20% SSA replacement levels, respectively.

As shown in Table 6.6, SSA had a positive effect on the resistance to chloride ingress, measured as current passing through the concrete (Series 52). This may to be linked to alterations in the pore size distribution and bodes well for durability, given that the 5% and 10% SSA mixes were categorised as exhibiting "low" and "very low" chloride penetration, respectively.

The corrosion resistance of mortars with embedded steel bars, under immersed conditions, was comparable to that of the controls, with SSA as a low-content cement replacement, based on the instantaneous corrosion rate (I_{corr}) measured using a polarisation resistance technique (Series 53). This can be attributed, in part, to the pore refinement from the pozzolanic activity of SSA, and possibly to the high alumina content in SSA (average Al₂O₃ of 14%, compared to the typical 5% in Portland cement, given by Jackson and Dhir, 1996) that leads to higher C₃A, which binds with chlorides in the formation of calcium chloro-aluminates. However, at higher SSA replacement levels of 30% and 60%, these positive effects were overcome by the progressive weakening of the mortar microstructure, resulting in a net negative effect.

Carbonation-induced corrosion increased with SSA, though the effect was not significant (neutral in Table 6.6), with cement replacements up to 10%. This increased corrosion is caused by the lowered alkalinity resulting from the reduced cement content and the pozzolanic reactions with lime. Indeed, increased susceptibility to carbonation in concrete is a recognised issue with all pozzolanic materials (Lye et al., 2015 and Lye et al., 2016), not just with SSA.

Despite initial reservations due to the higher Al_2O_3 in SSA, no sulphate expansion occurred in mortars using up to 30% ash as a cement component. It appears that sulphates present in SSA are not in soluble form and do not react with the tricalcium aluminates to produce the

damaging secondary ettringite. Expansion from alkali aggregate reactivity was also reduced with SSA, owing to the lower Portland cement content and the associated dilution in alkalinity.

6.2.3 Aerated Concrete

The use of SSA in aerated concrete appears to be a particularly suitable application for a number of reasons:

- The average aluminium content of SSA, at 15%, is much greater than that of Portland cement (typically around 5%). Although SSA has not shown the same intense expansive reactions in alkaline cement mixes as MIBA, the aluminium may be able to contribute somewhat to the expansion and lessen the required aluminium foaming agent dosages. However, as high alkalinity is required for the foaming reaction to take place, Portland cement is still essential to provide this pH condition.
- The pozzolanic activity of SSA can contribute to a modest strength development.
- The irregular particle size and porosity of SSA favour the desired lightweight and low thermal conductivity properties.

The work involved the use of ground SSA as a bulk component, at contents of from 60% to 80% by mass of cement, in the manufacturing of aerated concrete cubes by a process of casting, de-moulding, curing and subsequently cutting away the excess bulging (Chen et al., 2006a, Wang et al., 2005a; Wang et al., 2005b and Wang and Chiou, 2004). Water/binder ratios (with the binder consisting of cement + SSA) ranged from 0.5 to 0.8 in these mix series, with aluminium powder foaming agent dosages from 0.1% to 1.3%.

Of the aforementioned mix parameters, an increase in the water/binder (w/b) ratio caused the greatest increase in the foaming reaction intensity, leading to better lightweight properties and lower strengths. The magnitude of any positive contribution of SSA to the expansion is unclear, as at the high ash contents used (60% to 80%), significant dilution of the mix alkalinity ended up having a net negative effect with increasing SSA contents. Upping the foaming agent dosage led to the expected increase in the foaming reaction intensity.

Bulk density results for the aerated concrete with 60%–80% SSA ranged from 610 to 1070 kg/m³, which is in the upper end of the 300–1000 kg/m³ range for autoclaved aerated concrete

outlined in BS EN 771-4 (2011). The lightweight properties, closely linked to the porosity, improved with increasing SSA content, foaming agent content and w/b ratio. Pores generated during foaming were mostly larger than 10 μ m, which is significantly bigger than the cement hydration pores. As such, the foaming creates a high volume and a loose and open pore structure. The porous nature of SSA also contributes to the light weight.

Compressive strength results are presented in Figure 6.7, for two aerated concrete mix series adopting cement/SSA ratios of 40:60 (blue-shaded area), 30:70 (red) and 20:80 (green); w/b from 0.5 to 0.8 and foaming agent dosages [aluminium powder/binder ratio (a/b)] from 0.1% to 0.3%. Mirroring the density results, it is evident that strength reduces as the w/b ratio, a/b ratio and SSA content increase. The effect of both the a/b ratio and the SSA content on the strength performance is much greater at lower w/b ratios, though in all cases, the minimum strength requirements of greater than 1.5 N/mm² were satisfied.

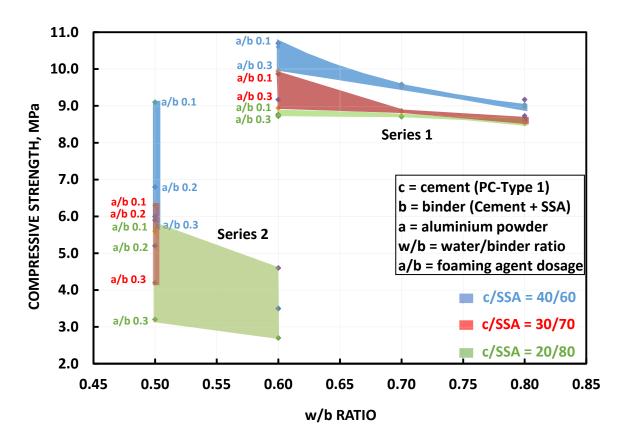


Figure 6.7: Compressive strength performance of aerated concrete mixes with SSA Data from: Chen et al. (2006a), Wang et al. (2005a); Wang et al. (2005b) and Wang and Chiou (2004).

Thermal conductivity values from 0.076 to 0.247 W/m K have been achieved with the same mix series. The higher porosity attained with increasing w/b ratio, SSA content and aluminium powder dosages correlated with improved thermal performance. Thermal conductivity requirements of 0.12–0.15 W/m K for autoclaved lightweight concrete and 0.034–0.173 W/m K for heat insulation materials are specified in the Taiwanese Standards. The latter more stringent constraints can be met with the following mix proportions: w/b ratio greater than 0.4, aluminium powder content greater than 0.1% and SSA content greater than 60%.

6.2.4 Blocks

Data on the performance of two precast concrete block mix series, incorporating SSA as a cement replacement and addition, at contents up to 20%, are presented in Table 6.7. It is evident that the replacement of cement with SSA led to a minor decrease in the density, attributable to the lower specific gravity of the ash, though the addition led to a minor density increase, possibly due to filling effects arising from the SSA pozzolanic activity. Nevertheless, in both cases the changes were very minor, varying from 96% to 102% of the control.

Opposing trends of both increasing and decreasing water absorption and capillary water absorption results were also evident in the two mix series. The reason for the inconsistency is uncertain, though it was shown previously in section 6.2.2 that, despite the high absorption of SSA itself, the pozzolanic reactions can lead to refinement of the pore structure, resulting in reduced absorption and permeation properties.

The block compressive strengths were found to initially increase with the lowest SSA contents, but then they reduced to below the control at high ash contents. This pattern was evident previously in some mortars and concrete in Table 6.3 and again may be linked to the initial refinement of the pore structure, followed by the progressive dilution of the cement and weakening of the concrete block microstructure at higher SSA contents. Strength requirements for blocks are generally less onerous and there are broad strength quality levels; as such, SSA can be a suitable option as an effective cement component at low contents. Table 6.7 also shows that blocks with SSA exhibited slightly improved insulation properties.

Table 6.7: Performance of concrete blocks incorporating SSA as a cement component

	SERIES 1: CEMENT F	REPLACEME	NT, w/b 0.67,	CEM I 52.5R	SERIES 2: CEMENT ADDITION, w/b 0.68, CEM II BM (S-LL)-42.5R							
<u>-</u>	PARAMETER	CONTROL BLOCKS	10% SSA	20% SSA	PARAMETER	CONTROL BLOCKS	5% SSA	10% SSA	15% SSA	20% SSA		
sity	Density, kg/m³	2120	2070 (-2%)	2040 (-4%)	Density, kg/m ³	2058	2059 (0%)	2087 (+1%)	2096 (+2%)	2101 (+2%)		
Density	Pearson Coefficient	0.004	0.006	0.008	CV, %	0	0.6	0.5	0.2	0.3		
Absorption Properties	Absorption, %	8.0	8.7 (+8%)	8.3 (+4%)	Absorption, %	8.7	8.9 (+3%)	7.9 (-9%)	7.3 (-16%)	7.3 (-16%)		
Abso Prop					CV, %	0.6	3.5	4.8	2.8	0.4		
Water	Capillarity, C _{10mins} , g/m ⁻² s ^{-0.5}	87	87 (0%)	101 (+16%)	Capillarity, kg/m²min ^{0.5}	0.61	0.65 (+6%)	0.48 (-22%)	0.51 (-33%)	0.38 (-38%)		
Capillary Wat Absorption	Capillarity, C _{final} , g	245	277 (+13%)	239 (-2%)	CV, %	3.1	1.9	2.1	5.5	2.3		
Comp Strength	Mean Strength, MPa	4.44	5.59 (+26%)	4.17 (-6%)	28-Day Mean Strength, MPa	7.0	7.1 (+2%)	6.8 (-2%)	6.5 (-6%)	5.5 (-21%)		
Co	Char. Strength, MPa	3.88	4.58 (+18%)	3.77 (-3%)	CV, %	2.7	14.7	6.6	3.6	2.8		
Thermal Conductivity	$\lambda_{10,dry}$ (p= 50%), W.mK ⁻¹	1.136	1.077 (-5%)	1.04 (-8%)								
Ther	$\lambda_{10,dry}$ (p=90%), W.mK ⁻¹	1.324	1.267 (-4%)	1.23 (-7%)								

Note: Values in brackets represent the percentage change from the control blocks, green - increase & red - decrease

CV = coefficient of variation. Char – Characteristic, Comp – compressive, C_{10mins} – coefficient after a time period of 10mins, C_{final} – coefficient at the test end time (EN 771-3), $\lambda_{10,dry}$ (p= 50%) – thermal conductivity in dry blocks, 50th percentile, $\lambda_{10,dry}$ (p= 90%) – thermal conductivity in dry blocks, 90th percentile. Data from: Perez-Carrion et al. (2013); Baeza-Brotons et al. (2014)

Data on the variability of block performance are also given in the Table, as in block manufacturing, retaining a high degree of reliability and repeatability is particularly important, when producing large volumes of standard units. It is evident that the coefficient of variation and the Pearson coefficient for the SSA blocks are generally small and within close proximity to the control values. In addition, no adverse effects on the dimensional stability or configuration of the precast block units were recorded with SSA.

6.3 Use as an Aggregate Component

6.3.1 Mortar and Concrete

The particle size distribution of SSA, in as-produced form (Figure 4.1), points towards its suitability as a filler or fine aggregate. The results of the work undertaken using mortar and concrete mixes, involving its use as a fine aggregate replacement and addition, and filler addition, are presented in Table 6.8.

The slump results show large reductions in the concrete consistence, reducing from 180 to 0 mm at 15% SSA addition. Though the irregular morphology and porosity of SSA is expected to somewhat decrease the workability, the effect is exacerbated by the increased overall aggregate content, from the SSA addition. Mix adjustments should be adopted to accommodate SSA, which can include extra water or superplasticiser additions, as in Series 5 (Table 6.8).

The density of the mixes was generally reduced, owing to the lower specific gravity and bulk density of SSA compared to sand and cement. The contrasting increase evident for Series 2.2, with SSA as a sand replacement, points towards an improvement in the particle packing of this concrete, though the ash did not consistently achieve this desired pore refinement when used as filler in other mix series.

Table 6.8 shows that SSA had a varying effect on the compressive strength performance. Remarkable increases were evident in Series 1 and 2.2, and to a lesser extent Series 5. Improvements in the mix grading and particle packing can contribute to noteworthy increases

Table 6.8: Results on the performance of mortar/concrete with SSA as aggregate

SERIES DETAILS	RESULTS				
Consistence					
Series 1: Concrete. SSA as a sand	SSA, % (sand)	0	5	10	15
addition. Constant Water.	Slump, mm	180	125 (-31%)	30 (-83%)	0 (-100%)
Density					
Series 2.1: Mortar. 0-4mm sand	SSA, % (sand)	0	10		
replaced. Extra water	Density, kg/m ³	2094	1923 (-8%)		
Series 2.2: Concrete. 0-4mm sand	SSA, % (sand)	0	10		
replacement. Extra Water	Density, kg/m ³	2058	2204 (+7%)		
Series 3.1: Concrete. Filler	SSA, % (cement)	0	5	10	20
addition. Constant w/c 0.45	Density, kg/m ³	2292	2202 (-4%)	2190 (-4%)	2161 (- <mark>6%</mark>)
Series 3.2: Concrete. Filler	SSA, % (cement)	0	5	10	20
addition. Constant w/c 0.55	Density, kg/m ³	2231	2196 (-2%)	2161 (- <mark>3%</mark>)	2099 (- <mark>6%</mark>)
Series 4. Concrete. 0-4mm	SSA, % (sand)	0	10	25	50
replaced. 50% SSA – extra water	Density, kg/m ³	2277	2216 (-3%)	2179 (-4%)	1900 (-17%)
Series 5: Concrete. 0/1mm sand	SSA, % (sand)	0	10	20	30
replaced. Constant water with SP	Density, kg/m ³	2350	2340 (-0.4%)	2340 (-0.4%)	2330 (-0.9%)
Compressive Strength					
Series 1: Concrete. Sand addition.	SSA. % (sand)	0	5	10	
Constant Water.	Comp 28 days, MPa	16.2	25.0 (+55 %)	28.9 (+78%)	
Series 2.1: Mortar. 0-4mm sand	SSA, % (sand)	0	10	, ,	
replacement. Extra Water	Comp 28 days, MPa	38.1	32.4 (-15%)		
Series 2.2: Concrete. 0-4mm sand		0	10		
replacement. Extra Water	Comp 28 days, MPa	7	14.4 (+106%)		
Series 3.1: Concrete. Filler	SSA, % (cement)	0	5	10	20
addition. Constant w/c 0.45	Comp 28 days, MPa	36.8	34.4 (-6%)	32.8 (-11%)	28.8 (-22%)
Series 3.2: Concrete. Filler	SSA, % (cement)	0	5	10	20
addition. Constant w/c 0.55	Comp 28 days, MPa	32.2	31.7 (-1%)	29 (-10%)	24.3 (-25%)
Series 6: Concrete. Sand	SSA, % (sand)	0	10	20	30
replacement. Constant water	Comp 28 days, MPa	28.5	24.3 (-15%)	21.9 (-23%)	22.6 (-21%)
Series 4. Concrete. 0-4mm	SSA, % (sand)	0	10	25	50
replaced. 50% SSA – extra water	Comp 28 days, MPa	39.4	36.0 (-9%)	33.8 (-14%)	9.3 (-76%)
Series 5: Concrete. 0/1mm sand	SSA, % (sand)	0	10	20	30
replaced. Constant water with SP.	Comp 28 days, MPa	29.2	31.2 (+7%)	32 (10%)	34.7 (+18%)
Water Absorption Properties					
Series 1: Concrete. Sand addition.	SSA, % (sand)	0	5	10	
Constant Water.	Absorption, %	4.47	4.19 (-6%)	4.38 (-2%)	
Series 2.1: Mortar. 0-4mm sand	SSA, % (sand)	0	10		
replacement. Extra Water	Absorption, %	8.3	13.2 (+59%)		
Series 2.2: Concrete. 0-4mm sand	SSA, % (sand)	0	10		
replacement. Extra Water	Absorption, %	8.7	6.0 (-30%)		
	Capillary, kg/m ² min ^{0.5}	0.61	0.26 (-57%)		
Series 3.1: Concrete. Filler	SSA, % (cement)	0	5	10	20
addition. Constant w/c 0.45	Capillary, kg/m ² h ^{0.5}	0.549	0.554 (+1%)	0.539 (-3%)	0.900 (+67%)
Series 4. Concrete. 0-4mm	SSA, % (sand)	0	10	25	50
replaced. 50% SSA – extra water	Absorption, %	4.43	4.73 (+7%)	5.37 (+21%)	8.72 (+97 %)

Green – Increase, Red – Decrease. SP – superplasticiser, Comp – Compressive strength, Relative effect compared with the control given in brackets.

Data from: De Lima et al. (2015); Baeza-Brotons et al. (2015); Jamshidi et al. (2012); Khanbilvardi and Afshari-Tork (1995); Kosior-Kazberuk (2011); Paraschivoiu and Vlad (2016);

in strength, though not to the huge extent evident in these mix series. As such, these results appear to be questionable. Decreases in strength occurred in Series 2.1, 3.1, 3.2, 4 and 6, using SSA as a filler addition and sand replacement, as the ash seemed to have the opposing effect of reducing the density, with increased volume of pores, thus demonstrating the sensitivity of the strength performance to the aggregate/filler grading. Notwithstanding this, the use of SSA would be more favoured as a minor component, rather than a bulk or complete natural aggregate replacement. Flexural strength results were found to mirror the compressive strength performance.

The mixed effect of SSA on the mortar and concrete mixes is also reflected in the absorption results in Table 6.8. Increases in water absorption tended to correlate with reductions in compressive strength and vice-versa, which again can be attributed to the particle packing behaviour and the associated effects on the pore distribution. Increases and decreases were also evident in the capillary water absorption with SSA; however, even the least favourable result of $0.9 \text{ kg/m}^2 \text{ h}^{0.5}$, with 20% SSA addition, is still within the normal range for conventional concrete mixes.

6.3.2 Lightweight Aggregate Production

The manufacturing process with SSA involved sintering treatment to bring about the expansive behaviour, to create a lightweight aggregate that retains a strong outer surface layer. Key parameters to consider in the production of the SSA lightweight aggregates are:

- 1. Sintering temperature: temperatures from 900 to 1150°C have been adopted.
- 2. Sintering duration: times from 10 to 30 min have been used.
- 3. Admixtures: sewage sludge, clay, boric acid (H₃BO₃), glass cullet powder, aluminium oxide and municipal solid waste fly ash have been incorporated into various mixes to lower the melting temperature or enhance strength properties.

Data showing the relationship between the sintering temperature and the lightweight properties, specifically SSA bulk density results, are presented in Figure 6.8. The effects of the sintering time (green colour) and the inclusion of sewage sludge (orange) and H₃BO₃ (red) admixtures are also highlighted in the figure.

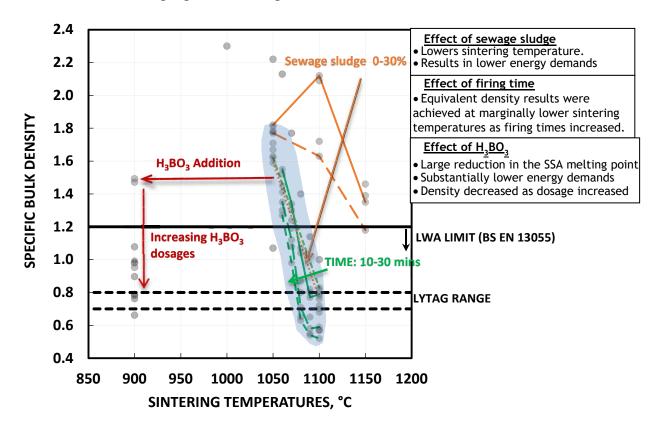


Figure 6.8: Specific bulk density of SSA lightweight aggregates

LWA – lightweight aggregate

Data from: Bhatty and Reid (1989b); Chiou et al. (2006); Hu et al. (2012b); Lin (2006); Tsai et al. (2006).

It is evident from Figure 6.8 that the majority of the density results fall within the temperature range 1050–1100°C, in which the expansive processes develops, leading to the formation of large discontinuous pores. This results in a rapid decrease in the density, as is highlighted in the blue-shaded region in Figure 6.8. To satisfy the bulk density limit of 1.2 for classification as a lightweight aggregate in BS EN 13055 (2016), a peak sintering temperature of around 1060°C is required. Lightweight properties similar to those of commercially available Lytag (bulk density of 0.7–0.8 from Gunning et al., 2011) were achieved with SSA at around 1080°C.

The effect of increasing sintering times, from 10 to 30 min (green lines in Figure 6.8), was much less pronounced compared with the temperature change, though the longer duration resulted in a slightly steeper decrease in density with increasing temperature. Additions of sewage sludge and in particular H₃BO₃ and glass cullet powder have been effective in reducing the melting temperature of SSA aggregate, which reduces the energy demands by allowing the same expansive behaviour and density reductions to be achieved at lower peak temperatures.

Reductions in density as the pores develop also have to be balanced with associated strength decreases. It is found from an additional SSA lightweight aggregate series (data from Cheeseman and Virdi, 2005) that the strength is maximum at the sintering temperature of 1050°C, immediately prior to the instigation of the expansive behaviour. Compressive strengths from 3 to 5 MPa were achieved when SSA had a bulk density comparable to that of Lytag aggregate. This places the SSA lightweight aggregate at the lower end of the expected Lytag strength range. However, data from additional mix series (Cheeseman and Virdi, 2005 and Tsai et al., 2006) showed that clay, aluminium oxide and municipal solid waste fly ash can be effectively combined with SSA to enhance the mechanical properties of the sintered mixture.

6.3.3 Lightweight Aggregate Concrete

The work undertaken and the resultant performance of concrete mixes incorporating lightweight aggregates manufactured from SSA are described in Table 6.9. A wide variety is evident in both the aggregate production process and the concrete mix designs adopted. The SSA lightweight aggregate has been used mainly as a coarse aggregate and, in one mix series, as a combined coarse + fine aggregate. Certain mix series have added sewage sludge and clay alongside SSA to improve the cohesiveness before sintering. The work also includes concrete mixes containing lightweight aggregates produced from the sintering of the original sewage sludge, instead of SSA.

WORK UNDERTAKEN AND DATA OBTAINED

MIX SERIES

<u>Series 1:</u> SS sintered at 1050°C. Concrete A: SSA LWA as coarse agg, w/c 0.5, fine:coarse agg ratios from 1:1.5 to 1:2.48. Concrete B: SSA LWA as coarse + fine agg, w/c 0.5 - 1.04, fine:coarse ratios from 1:0.9 to 1:1.15.

<u>Series 2:</u> SSA pelletized (≈12 mm), sintered at 1050°C, 30mins. Concrete: SSA LWA as 0, 10, 20, 35, 50, 70, 100% of the coarse agg. An additional reference mix was produced with commercial LWA as 35% of the coarse agg.

<u>Series 3:</u> Blend A: 63% SSA+10% clay+27% SS. Blend B: 64% SSA+20% clay+16% SS. Pelletized & sintered at 1090°C to produce LWA. Concrete using SSA LWA as coarse agg, w/c 0.55, fine:coarse ratio of 0.99 and 1.04 for LWA A & B.

<u>Series 4:</u> SSA pelletized or slabbed, then sintered at 1060-1100°C, 10-30 mins. SSA LWA split into heavier (sg 1.1-1.4) and lighter fractions (sg 0.8-1.1) and used as coarse agg, making up 18-40% of the concrete mixes.

Series 5: SS sintered at 1050°C for 6 hours. Used as coarse agg in concrete, 1:1.5 fine:coarse agg ratio, w/c 0.6.

Series 6: Concrete panel with SSA LWA as coarse agg & no fine agg. Cement-to-coarse agg ratio of 1:6 and w/c 0.5.

Series 7: Concrete panel with SSA LWA as coarse agg.

CONSISTENCE

Series 1: Mix A slumps 10-20mm, Mix B slumps 20-50mm, minor increases with decreasing fine:coarse agg ratio.

Series 2: Slump - Control 70mm. 10-35% SSA LWA - 80mm. 50-100% SSA LWA - 90mm. Commercial LWA - 70mm.

Series 3: Concrete slumps - Control 20mm, with LWA A 20mm, with LWA B 50mm, with Lytag 10 mm.

DENSITY

<u>Series 1:</u> Air-dry densities from 1716-1949 kg/m³ and 1545-1831kg/m³ for concrete A & B. Density increased with increasing fines. Concrete with SSA LWA was fit for medium to high strength applications.

<u>Series 4:</u> Concrete with SSA LWA were denser & stronger than the commercial clay agg concrete. With coarse agg:sand:cement ratios of 40:60:10, densities of 1345-1583 kg/m³ with SSA vs. 1243 kg/m³ with expanded clay.

Series 5: Air-dry density of SSA LWA mix - 1706kg/m³ < LW concrete limit of 1850kg/m³, > Leca mix (1546kg/m³)

COMPRESSIVE STRENGTH

Series 1: Concrete mix A (13-31 MPa) and mix B (6-27 MPa) could be fit for structural use, when using high fines %.

<u>Series 2:</u> 28 day compressive strengths from 84-104% of the control with SSA LWA. Strength generally decreased with increasing SSA%. SSA mixes had strengths > corresponding 35% commercial agg mix.

<u>Series 3:</u> 28 day strength. Blend A 33MPa - 84% of the natural agg mix, 90% of Lytag mix. Blend B - 51 MPa - 112% of the natural agg, 120% of the Lytag mix.

<u>Series 4:</u> Pellet agg concrete was stronger than slabs. Lighter fraction SSA LWA pellets (9.3 MPa) and slabs (5.7 MPa) met moderate strength concrete limits with coarse agg:sand:cement ratios of 40:60:10.

Series 5: 28 day strength. SSA LWA concrete 23.6MPa > Leca mix 21.6 MPa. Met limit for structural use.

WATER ABSORPTION

<u>Series 1:</u> Absorption of SSA LWA of 10.3-13.8% for mix A and 12.1-14.8% for mix B. Decreasing absorption with increasing cement % and fine aggregate proportion.

Series 5: Absorption of SSA LWA agg concrete of 10.6% is lower than the Leca mix value of 13.5%.

THERMAL CONDUCTIVITY

Series 6: Thermal conductivity with SSA = 0.27 W/m°C, < ASTM limit of 0.43 W/m°C for LW agg insulating concrete.

<u>Series 7:</u> Thermal conductivity of the SSA LWA panel = $0.49 \text{ W/m}^{\circ}\text{C}$, marginally above ASTM limit for LW agg concrete ($0.43 \text{ W/m}^{\circ}\text{C}$) and the corresponding Leca concrete panel value of $0.3 \text{ W/m}^{\circ}\text{C}$.

Agg – aggregate, LW – lightweight, LWA – lightweight aggregate, SS – sewage sludge

Data from: Bhatty and Reid (1989b); Bhatty et al. (1992); Tay and Yip (1989); Wainwright and Cresswell (2001); Yip and Tay (1990).

The slump results in Table 6.9 reveal that SSA lightweight aggregates had a positive impact on the concrete consistence, as a replacement of the coarse natural aggregate. This is attributed to the pelletisation and sintering in the aggregate production, which lead to more rounded and smoother particles, with lower water demands.

From varying the concrete mix design (e.g., increasing fine/coarse aggregate ratios, using clay additions), there was great flexibility achievable with SSA lightweight aggregate. Densities from 1345 to 1950 kg/m³ and compressive strengths from 5 to 30 MPa were achieved, though SSA lightweight aggregate mixes generally exhibited higher density and corresponding strength results than the equivalent mixes using commercial lightweight aggregate. Dividing SSA into light and heavy fractions (series 4) was effective in targeting a higher or lower strength level, as the density-to-compressive ratio remained constant for both fractions. Strength-to-weight ratios for SSA lightweight aggregate concrete mixes ranged from 74×10^{-3} to 88×10^{-3} MPa/g, compared with 77.7×10^{-3} MPa/g for natural aggregate and 78×10^{-3} MPa/g with the commercial lightweight aggregate mixes (Series 2). This indicates that SSA lightweight aggregate can provide performance, in terms of strength for a given density, comparable to that of these established materials.

Water absorption values, ranging from 10.3% to 14.8% for concrete mixes containing SSA lightweight aggregate, were at a similar level compared to concrete containing the commercially available lightweight aggregate Leca (13.5% absorption). High water absorption properties are typical for lightweight aggregate concrete and values of approximately 10% are considered normal.

Thermal conductivity has been shown to increase with increasing mix porosity and reducing density and, as outlined in Table 6.9, concrete using SSA lightweight aggregate can be designed to meet the thermal conductivity requirements for insulating concrete specified in ASTM C332 (2009).

6.3.4 Foamed Concrete

Foamed concrete, as another potential outlet for SSA, is a relatively new concept that developed because of health and safety concerns (white finger scenario), relating to using vibrators in trench filling for gas, electricity and water utility companies. Essentially, it is a slurry (made from cementitious materials, fine aggregate and a large quantity of water), into which foam is pumped, creating a product in which the solid particles are not in contact with one another and as such, the quality of the solid particles is of less consequence. The key properties for this application are high flowability, self-compacting, self-curing and lightweight properties and low strength.

In the initial work undertaken, SSA replaced 50% and 100% of the fine aggregate in foamed concrete mixes. The effects, relative to the control sand mix, on the tested flowability, compressive strength, permeability and thermal conductivity are presented in Figure 6.9. The magnitude of each property, at 50% and 100% SSA contents, is also provided in the adjacent labels.

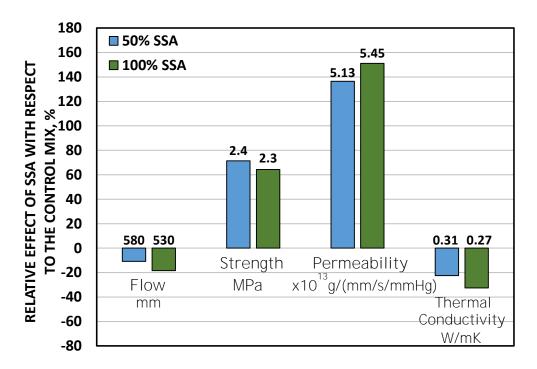


Figure 6.9: Effect of SSA as a fine aggregate replacement on foamed concrete properties Data from Halliday et al. (2012).

It is apparent from Figure 6.9 that SSA decreased the mix workability, with flow values 11% and 18% lower than that of the control, with 50% and 100% SSA, respectively. However, the reductions are significantly less pronounced compared with the previous use as an aggregate in normal-weight concrete. Indeed, the SSA foamed concrete mixes still retained the desired high flowability and self-compacting traits.

The compressive strength was found to increase with SSA. This was attributed to the greater quantity of fine aggregate required to achieve the target concrete plastic density, because of the lower particle density of SSA. Nevertheless, with modest strength requirements in foamed concrete, any effects of SSA on this parameter should not dictate its suitability for use.

The increase in permeability and decrease in thermal conductivity evident in Figure 6.9 with SSA are both attributed to the porous nature of the ash particles. As such, this equates to improved insulation properties with the SSA foamed concrete, though with reduced durability. When used as a natural sand replacement SSA can also contribute to the desired lightweight properties. It is noteworthy that the inherent porosity of the ash, which is normally perceived solely as a negative, may in fact increase the value of the material in this concrete application.

6.4 Environmental Assessment

6.4.1 SSA Leaching

Similar to MIBA, the environmental assessment undertaken with SSA addresses the leaching of its heavy metals. From the initial classification of the ash, leached element concentrations using the American toxicity characteristic leaching procedure (TCLP), given in Table 6.10, were below the US EPA hazardous waste classification limits, with the exception of series 18, which exhibited remarkably high releases of Cd, Cr and As (4, 11 and 5 times higher, respectively, than the next highest from all other samples).

Table 6.10: Leaching behaviour of SSA using the TCLP

NAIV CERIES			TCLP LEAC	CHING (CONCENT	RATION	S, mg/L	
MIX SERIES	Cd	Cr	Cu	Ni	Pb	Zn	As	Hg
Series 1	<0.2	<0.2	7.94	-	<0.2	11.3	0.42	nd
Series 2	nd	0.1	3.81	-	0.017	-	-	-
Series 3	0.02	0.1	3.81	nd	0.1	17.66	nd	nd
Series 4	0.06	< 0.01	-	-	<0.1	-	0.16	<0.0002
Series 5	0.16	0.01	-	-	<0.05	-	0.12	<0.2
Series 6	0.009	0.009	0.008	0.046	0.061	0.019	-	-
Series 7	0.03	nd	0.51	-	nd	7.04	-	-
Series 8	0.03	0.06	0.82	1.97	0.37	8.14	-	-
Series 9	0.03	0.06	8.24	1.97	0.37	8.14	-	-
Series 10	0.03	0.24	1.95	6.52	0.54	16.75	-	-
Series 11	0.02	0.034	8.725	0.033	0.027	1.7	0.084	-
Series 12	0.03	0.01	11.9	0.06	0.02	2.07	-	-
Series 13.1	0.04	0.28	1.02	-	0.2	0.65	-	-
Series 13.2	0.04	0.53	11.5	-	0.28	0.56	-	-
Series 13.3	0.01	0.1	0.17	-	0.12	0.68	-	-
Series 13.4	0.02	0.42	1.78	-	0.12	1.21	-	-
Series 14	0.07	nd	0.63	-	nd	6.05	-	-
Series 15	0.293	0.1367	-	0.217	0.0122		0.1217	-
Series 16	<0.01	< 0.01	0.06	-	0.39		< 0.01	-
Series 17	-	1.36	4.64	1.22	2.43	3.16	-	-
Series 18	1.05	15.2	3.34	0.45	0.28	4.1	0.77	<0.002
Min SSA, mg/L	nd	nd	0.17	nd	nd	0.65	nd	nd
Max SSA, mg/L	1.05	15.2	11.5	6.52	2.43	16.75	0.77	nd
TCLP limits, mg/L	1	5	15 (ROC)	-	5	25	5 (ROC)	0.2 (ROC)
SSA Samples exceeding TCLP limits	1	1	0	-	0	0	0	0

Red – exceeding TCLP hazardous waste classification limits. nd – not detected.

Data from: Chen and Lin (2009a); Chen and Poon (2016); Chen et al. (2006a); Huang and Li (2003); Khanbilvardi and Afshari-Tork (1995); Khanbilvardi and Afshari-Tork (2002a); Latosinka (2014); Lin (2006); Lin and Weng (2001); Lin et al. (2005a); Lin et al. (2005c); Lin et al. (2008b); Lin et al. (2008c); Lin et al. (2009a); Tsai et al. (2006); Wang et al. (2015); Weng (2002); Zhang et al. (2015b).

Note: ROC – Republic of China. These additional limits are regulated by NIEA R355.00C (2005) and NIEA R201.13C (2007) set by the Chinese EPA.

With the European EN 12457-3 (2002) test, the mean leached concentrations calculated for SSA (Table 6.11) were generally categorised as "inert" or "non-hazardous" for most elements, according to the Council Decision 2003/33/EC limits (European Community, 2003). The

exceptions were Se and Mo, classified as hazardous, though these elements are not limited in the equivalent US EPA classification. Maximum Hg and Sb values also exceeded the non-hazardous limits.

Table 6.11: Leaching behaviour of SSA using the EN 12457 (2002) method

ELEMENT	LEACHII	NG, mg/k	g SSIA		LANDFILL LIMITS, I	mg/kg	MEAN	
ELEIVIEINI	Min	Max	Mean	Inert	Non-Hazardous	Hazardous	CLASSIFICATION	
Pb	0.002	0.24	0.04	0.5	10	50	Inert	
Zn	0.001	0.6	0.32	4	50	200	Inert	
Ni	< 0.01	0.13	0.04	0.4	10	40	Inert	
Se	0.32	3.31	1.17	0.1	0.5	7	Hazardous	
Cu	< 0.01	0.19	0.05	2	50	100	Inert	
Cr	0.02	0.45	0.19	0.5	10	70	Inert	
Cd	< 0.001	0.008	0.002	0.04	1	5	Inert	
As	0.008	0.71	0.24	0.5	2	25	Inert	
Hg	0.009	0.212	0.048	0.01	0.2	2	Non-hazardous	
Мо	5.8	19.5	11.3	0.5	10	30	Hazardous	
Sb	0.004	1.14	0.36	0.06	0.7	5	Non-hazardous	
Cl	102	1320	452	800	15000	25000	Inert	
F	11	39.1	24.3	10	150	500	Non-hazardous	
SO ₄	6536	13427	9342	1000	20000	50000	Non-hazardous	

Green - Inert, Brown - Non-Hazardous, Red - Hazardous

Note: In cases where the leached concentration was below the detection limit, this detection limit value was conservatively used as the value to calculate the mean leached concentration. Data from: Cyr et al. (2012); Donatello et al. (2010b), Maozhe et al. (2013) (9 samples)

Findings on the effects of the incineration temperature and pH conditions on leaching behaviour are given as follows:

- The leachability of Cd, Cr, Pb and Ni was largely unaffected by incineration temperature; however, the mobility of Cu and Zn increased significantly with rising temperatures in the tested 850°C–1000°C range (Latosinka and Gawdzik, 2012). As such, the lower end of this range should be more favourable for SSA future use in construction applications.
- SSA has a low acid-neutralisation capacity. Its overall leachability is greatest under acidic conditions. Released concentrations of Cd, Zn, Ni, Pb, Cu, Co, Mg and Ca

decreased with increasing pH from acidic to neutral to alkaline conditions, whilst Zn exhibited amphoteric properties, with high leachability in both acidic and alkaline conditions. The leachability of Na and K was not strongly pH dependent (Cheeseman et al., 2003 and Johnson, 2003).

6.4.2 Raw Feed in Cement Clinker Production

As covered in Section 6.2.1, SSA has been tested at low contents (up to 11%) as part of the raw feed for cement clinker production. Leaching results for Series 2, 3, and 5 from Table 6.1, obtained using the TCLP, are presented in Table 6.12, along with data for control mixes and corresponding US EPA hazardous limits.

Table 6.12: Leaching results (TCLP) of cement clinkers incorporation SSA in the raw feed

MIV CEDIEC	CCA 0/		TCL	P LEACHED	CONCE	NTRATIO	N, mg/L		
MIX SERIES	SSA, %	Cu	Cr	Cd	Pb	Ni	Zn	Ва	TI
2.0	Control	nd	nd	nd	nd	nd	0.065	1.327	0.056
2.3	8.0	nd	nd	nd	1.121	nd	nd	0.6	0.431
3.0	Control	< 0.02	< 0.016	< 0.014	0.72	_	0.33	_	
3.1	6.84	< 0.02	< 0.016	< 0.014	0.72	-	0.32	_	-
3.2	8.51	< 0.02	< 0.016	< 0.014	0.61	-	0.28	-	-
3.3	9.29	< 0.02	< 0.016	< 0.014	0.68	-	0.46	-	-
5.0	Control	<0.02	<0.016	<0.014	0.7	<0.014	0.3	_	_
5.1	4.86	<0.02	<0.016	<0.014	0.6	<0.014	0.3	_	-
5.2	6.47	<0.02	<0.016	<0.014	0.6	<0.014	0.3	-	-
5.3	11.39	<0.02	<0.016	<0.014	0.6	<0.014	0.3	-	-
USEPA Haz. Limits	-	15	5	1	5	-	25	100	-

nd – not detected, Haz – Hazardous.

Data from: Lam et al. (2010a); Lin and Lin (2004, 2005)

Table 6.12 shows that as SSA varies, and concurrently, other constituents fluctuate to satisfy the oxide requirements, the leaching behaviour is not meaningfully affected. Cement clinkers with SSA performed similar to the controls. All results were considerably lower than the EPA hazardous limits and many were below the detectable limits. This suggests that the heavy

metals in SSA have been effectively bound within the cement matrix. It should be noted that the TCLP is intended to classify the leaching of granular wastes and may not provide a complete evaluation of the aforementioned monolithic cement products. Leaching of crushed specimens containing SSA would also be useful to consider.

6.4.3 Lightweight Aggregate

Limited leaching tests on SSA lightweight aggregates have been undertaken, using the USEPA TCLP (Bhatty et al., 1992). Pellets were produced after pelletisation and sintering of SSA at 1050°C. Released concentrations of As, Ba, Cd, Cr, Pb, Hg and Se were all considerably lower than the EPA hazardous classification limits and indeed, Cd, Pb and Hg values were below the more stringent EU drinking water limits (European Community, 1998). The pelletisation and sintering processes, and the resultant hard outer surface layer formed, appear to inhibit the release of harmful constituents from SSA lightweight aggregates.

6.4.4 Mortar and Concrete

Leaching results for mortar and concrete mixes using SSA as a cement component are given in Table 6.13, covering the concentrations and cumulative releases from both monolithic and crushed specimens. It is evident that the leaching behaviour for the monoliths was very low, frequently below detectable limits, and comparable to the controls. These SSA results were below the respective limits for Danish Category 1 residues and soils construction materials (Danish EPA, 2000), Dutch building materials (Dutch Government, 1995), Brazilian inert materials (NBR 10004, 1987) and World Health Organisation (WHO, 2004) and European Community drinking water (European Community, 1998). In service, the elements can be effectively encapsulated within the mortar and concrete products.

Leaching from crushed specimens was substantially higher, ranging from three to six times greater than corresponding monolithic results, attributed to the increased available surface area of the fragments. However, the same trend is evident with non-SSA mixes. With 25% and 50% SSA as cement replacement, the total leached element concentrations increased by 6% and 51%, respectively, compared with the control crushed mortars, mainly due to higher Ti,

Table 6.13: Leaching results for concrete using SSA as a partial cement component

MIX SERIES	TEST	TYPE	RESUL	ΓS												
Series 1: Mo	ortar, cement rej	olacement	Leache	d concer	tration, u	g/L										
	,		As	Cd	Cr	Cu	Ni	Sb	Sn	Pb	Ti	V	Zn	рН	_	
Control 2.25% SSA	NF X31-211	Monolithic	0.04 0.04	0.02 0.02	3.1 3.41	2.6 1.02	0.5 0.31	0.1 0.14	1.1 0.23	0.3 0.22	0.3	1.4	2 1.36	11.6 11.68	_	
25% SSA 50% SSIA			0.25 0.3	0.05 0.08	6.5 7	2.6 4	2.5 3	0.7 1.2	0.7 1.3	0.3 0.35	0.5 0.5	1.9 5	2.4 3	11.6		
Control 2.25% SSA	NF X31-211	Crushed	0.7 0.18	0.16 0.11	35 64.15	5 6.92	3 2.98	0.1 0.1	2 0.45	1.6 1.21	8	0.5	5 8.34	12.55 12.39		
25% SSIA 50% SSIA			0.4 0.2	0.13 0.25	35 35	8 6	7 0.8	0.3 1	1 1	1.6 2.7	1.5 1.4	0.42 0.5	7 40			
Limits (WHO	/EU drinking wa	ter <u>)</u>	10	3/5	50	2000	20	20 / 5	-	10	-	-	3000	6.5-9.5	_	
Series 2: Cor	ncrete, cement r	eplacement	Leache	d concer	tration, m	ng/L										
			Ag	Al	Ва	Cl	Cu	F	Fe	Mn	Na	Pb	Se	S	Zn	Hardness
10% SSA 10% SSA	NBR 10006	Monolithic Crushed	<0.03 <0.03	<0.05 <0.05	<0.01 <0.01	5.63 13.5	<0.002 <0.002	<0.1 <0.1	<0.02 <0.02	<0.002 <0.002	107 104	<0.015 <0.015	<0.01 <0.01	9.52 7.47	<0.002 <0.002	0 560
Brazilian NBI	R 10004 Inert Ma	aterial Limits	0.05	0.2	1	250	1	1.5	0.3	0.1	200	0.05	0.01	400	5	500
Series 3: Cor	ncrete, cement r	eplacement	Cumula	ative Lea	ching after	r 36 days,	mg/m²		- -							
			Cr	Zn	Cl-	SO ₄ ²	Мо	Se	_							
Control 10% SSA	NF EN 15863	Monolithic	3.0 2.1	3.4 4.6	383 107	2874 2348	< d.l. <d.l< td=""><td>< d.l. <d.l< td=""><td>_</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></d.l<></td></d.l<>	< d.l. <d.l< td=""><td>_</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></d.l<>	_							
Dutch Buildi	ng Materials Lim	<u>its</u>	120	800	100000	160000	-	-								
Series 4: Cor	ncrete, FA replac	<u>cement</u>	Cumula	ative Lea	ching after	r 64 days,	mg/m ²									
			As	Ва	Cd	Cr	Cu	Мо	Ni	Pb	S	Sb	Se	Sn	V	Zn
Control	NEN 7345	Monolithic	1.42	0.59	0.43	0.94	0.33	0.71	0.63	0.67	433.5	1.01	3.03	1.01	6.25	1.16
50% SSA		Monolithic	0.81	4.91	0.43	3.05	0.3	0.87	0.48	0.25	738.9	0.82	1.72	0.82	10.7	0.98
	gory 1 residues a		41	600	1.1	140	51	14	50	120	107000	29	1.4	29	230	200
	gory 2 residues a alues – exceed lir		140	2000	3.8	480	170	48	170	400	320000	95	4.8	95	760	670

Zn and Sb concentrations. Leachate results for crushed SSA mixes were generally within the corresponding stringent limits shown in Table 6.13. The exception was the Cr release with 2.25% SSA (shown in red) which was also significantly higher than the control, though this was in contrast to higher 25% and 50% SSA, which were both below the EU drinking water limit. This suggests that the high Cr leaching result was not caused by SSA, and that there may have been some contamination during this test. The hardness value for crushed concrete with 10% SSA also was exceeded the pertinent Brazilian limit for inert materials.

An additional concrete mix series using SSA as a 25% fine aggregate replacement (Kosior-Kazberuk, 2011) showed negligible heavy metal leaching, well below the inert classification limits. Overall, when SSA is used at the expected low replacement level in mortar and concrete that is practical from a mechanical performance standpoint, the data suggest that the environmental impacts, even under non-favourable testing conditions, can be manageable, at a level close to the control mixes, in service and after destruction.

6.4.5 Blocks

The environmental assessment of SSA in blocks focused on the leaching behaviour under representative in-service conditions. Monitoring was undertaken over 3 months on concrete pavers using SSA as 20% by mass of the mix, using the Dutch NEN 7375 (2004) monolithic tank testing procedure, set up to simulate natural exposure conditions (Environmental & Water Technology Centre of Innovation Ngee Ann Polytechnic, 2012). Further monitoring of an offshore marine structure in Great South Bay, New York, using blocks with SSA as 40% of the fine aggregate, was also carried out over a 12 month period (Khanbilvardi and Afshari-Tork, 2002b).

The key points to emerge are:

• For pavers with 20% SSA, the leached concentrations of many of the more toxic heavy metals, As, Fe, Hg, Mn, Ni, Pb, Zn and Co, were below the detection limits at all times during monitoring. Concentrations of Cd, Ba and Mo were below the Dutch target values (Dutch Ministry, 2000) for soils/groundwater to be considered unpolluted.

Leached Cr was above the Dutch unpolluted target, though below the intervention value that would demand a clean-up operation.

- For the SSA blocks in the offshore structure, the TCLP results for the tested elements Ba and Cd were comparable to those for the control blocks over the 12 months of monitoring. The ash had no significant effect on these released concentrations, though complete data on the leaching of other heavy metals would also be of interest.
- The element leaching behaviour generally remained consistent over both the 3- and the 12-month monitoring periods. This suggests that there had been little depletion of the elements present in the SSA concrete blocks and that the solubility of the constituents was restricted to a certain extent within the concrete matrix.

6.5 Case Studies

Case studies have been undertaken using SSA in Portland cement clinker production, normal-weight concrete, blocks and aerated concrete. The emerging findings are presented in Table 6.14.

The outcomes of the case studies have been mainly positive, as the SSA products have generally been successfully produced to meet the performance requirements. Modifications to the mix designs have been required at times, including lower w/c ratios with superplasticiser addition, higher cement contents or higher water contents. The ash has been used effectively as raw feed in cement clinker production, though when accounting for the full life-cycle and the energy use during incineration, the original sewage sludge is a more economically favourable option overall, despite higher transport costs. Suitable use as cement, filler and fine aggregate components has been achieved in concrete and masonry blocks. Its use as a filler, rather than a cement component, was favoured in the Danish project, because of the lower pozzolanic activity of those ash samples.

CONCRETE RELATED CASE STUDIES FINDINGS

CEMENT CLINKER MANUFACTURING

<u>Case Study 1 – Japan, c. 2001:</u> Cement manufacturer accepted both dewatered sludge and SSA into a full scale plant. Sludge is more economically favourable than SSA, due to its energy contribution, despite the cake having higher transport costs.

<u>Case Study 2 – USA, c. 2004:</u> At the time of this study, SSA was being used as part of the raw feed in Holham Cement manufacturing plant in Mason City, Iowa, USA.

CONCRETE

<u>Case Study 3 – UK, c. 2002:</u> SSA replaced 10% and 20% of the cement. Strengths equal to the control were achieved using superplasticiser and lowering the w/c. SSA mixes exhibited increased shrinkage.

<u>Case Study 4 – Denmark, c. 2006-2008:</u> 1900t of SSA was used. The ash was more suited as filler than as a cement component. Ash from a fluidised bed incinerator was usable in its as-produced form, though multiple hearth ash required grinding. At the concrete plant, SSA was compatible with standard equipment, though a SSA only silo was needed. With SSA replacing coal fly ash, target strengths were attained with a marginally higher cement %. Recommendation is for SSA to replace <50% of the fly ash.

<u>Case Study 5 – Japan, c. 2007:</u> SSA was recycled in eco-friendly high flow concrete that was used in a shield tunnel, in a project undertaken in partnership with Yokohama City, Japan.

BLOCKS

<u>Case Study 6 – UK, c. 2007:</u> Trial with SSA as fine aggregate in walling blocks. The variability of SSA, inconsistent production and negative public perception were identified as potential barriers.

<u>Case Study 7 – UK, c. 2002:</u> SSA replaced 10% of the coal bottom ash in medium density blocks. Problems occurred during the production and the resultant blocks were not fit for testing.

<u>Case Study 8 – Singapore, c. 2011:</u> SSA replaced up to 20% of the sand in pavers. Mixes satisfied strength and leaching requirements, though marginal cement increases were needed for equal strength.

<u>Case Study 9 – USA, c. 1996:</u> SSA as 40% of the fine aggregate in blocks used in an erosion control structure. No weathering/damage with SSA blocks after 1 year, performed similar to control blocks.

AERATED CONCRETE

<u>Case Study 10 – UK, c. 2007:</u> SSA was used as a coal fly ash substitute by 2 commercial producers. Fit for use products were produced with SSA, though higher water demands had to be managed.

c. – circa. Data from: Avedoere Wastewater Services (2008); BRE (2007); Danish Technological Institute (2006); DEFRA (2007); Dhir et al. (2002); Dunster (2007); Environmental & Water Technology Centre of Innovation, Ngee Ann Polytechnic (2012); Jensen (2008); Khanbilvardi and Afshari-Tork (2002b); Kjersgaard (2007); Okuno et al. (2004); Sato Kogyo Co. (2007); URS Corporation (2004).

6.6 Summary

This chapter deals with the performance of SSA in concrete-related applications. As raw feed in cement clinker production, SSA can be incorporated at limited contents, up to around 5%, without compromising performance. At the higher tested SSA contents, up to 11%, the build-

up of P_2O_5 , SO_3 and organics suppresses the formation of C_3S , which negatively affects the setting properties and early strength development, though this aspect could be used as an advantage in hot weather concreting. For SSA cements, it is suggested to adopt a P_2O_5 limit in the range from 0.5% to 1%. From leaching testing, the element concentrations released from SSA cement clinkers were all below detectable limits.

As a cement component in mortar and concrete, the direct substitution of cement with ground SSA led to reductions in consistence and strength; though with mix design adjustments, such as superplasticiser to lower the w/c ratio, higher cement contents, coal fly ash or nanomaterial additions, performance comparable to that of controls has been achieved. Pozzolanic activity testing on SSA indicated a comparable degree of reactivity to coal fly ash, whilst the mixes exhibited the recognised trend of greater late age strength gain. Increases in the setting time are incurred with SSA. The effects on the mix shrinkage are not significant, at low replacement levels. Despite increased porosity, SSA had positive impacts on absorption properties, permeability and chloride ingress, due to refinements in the pore size distribution. Susceptibility to carbonation increased with SSA, though the effect was very minor with cement replacements up to 10%. No sulphate-induced expansion was evident with SSA. Alkali aggregate expansion was also reduced owing to the diluted alkalinity in SSA mixes. From the environmental standpoint, heavy metals in SSA have been encapsulated effectively within the mortar and concrete matrix.

As fine aggregate and filler components in mortar and concrete mixes, SSA had varying effects on density and the associated compressive strength, due to the sensitivity to the mix grading. Improvements in the particle packing have been achieved in some mix series, resulting in enhanced density and strength performance. Increased water demands were incurred with SSA. At low SSA contents, the water absorption of mortar and concrete mixes was largely unaffected.

Aggregates manufactured using SSA achieved the desired lightweight properties after pelletisation and sintering at 1060°C–1080°C. Corresponding compressive strengths of 3–5

MPa were at the lower end of the expected range for Lytag. Using SSA lightweight aggregates in concrete had a positive effect on workability, due to the smoothness of the pellets, and provided strength-to-density ratios comparable to those of commercial lightweight aggregate concrete mixes. Water absorption of concrete using sintered SSA aggregates was similar to that of mixes containing commercially available Leca. Thermal conductivity requirements for insulating concrete can also be met using SSA lightweight aggregates. The hard outer layer, formed from the pelletisation and sintering processes, inhibited the leaching of heavy metals from SSA lightweight aggregates.

As a cement component in concrete blocks, fluctuating effects on the density, absorption properties and compressive strengths were incurred with SSA. This was linked with an initial refinement of the pore structure caused by the pozzolanic reactions, followed by the progressive dilution of the cement and weakening of the microstructure as SSA increased. The dimensional stability and configuration of the precast units were not adversely affected, whilst minor improvements in the insulating properties were evident. The coefficients of variation for the tested parameters for SSA blocks were small and close to the control values, indicating good reliability. As an aggregate component, successful case studies have been undertaken using SSA blocks in an erosion control structure (40% of the fine aggregate) and the production of SSA concrete pavers (20% of the fine aggregate).

The high porosity, irregular particle shape and high Al_2O_3 content of SSA lends itself towards suitability for autoclaved aerated concrete. With ground SSA as a bulk component, at contents from 60% to 80% by weight of cement, densities in the upper end of the typical range for autoclaved aerated concrete were achieved. Compressive strength decreased with increasing SSA, though the water/binder ratio was the strongest influencing factor and the modest strength requirements were satisfied with all SSA mixes. Thermal performance improved with increasing SSA content. Units produced using SSA as a partial coal fly ash replacement in pilot-scale trials by two commercial producers were fit for use, though higher water demands were incurred.

The use of SSA in foamed concrete, as a complete fine aggregate replacement, led to improved insulating properties. Strengths with SSA were higher than that of the control, though the requirements for this application are very low. Despite a decrease in workability, the reductions were significantly less compared to those with SSA in normal-weight concrete, and the foamed mixes still retained the high flowability and self-compacting traits.

CHAPTER 7

MODELLING OF CONCRETE STRENGTH WITH INCINERATED ASHES

7.1 Introduction

This chapter presents the final stage of the thesis and deals with modelling the strength performance of concrete and mortar mixes incorporating MIBA and SSA as cement and aggregate components. In doing so, it is envisioned that the model can also be further used with all incinerated ashes. Chapters 5 and 6 provided a thorough analysis and evaluation of the effects of the ashes on various aspects of the concrete performance, for many application types. Building on this, the objective of the modelling work was to attain a further in-depth understanding of one specific parameter, the compressive strength, and develop a procedure to predict the performance with MIBA and SSA. As a key property of concrete, compressive strength was the clear-cut starting point for the modelling work. Strength is generally amongst the first properties to be examined when incorporating recycled and secondary materials (RSMs) into the mix, whilst, in addition, because of its importance, a large amount of data was available on this parameter.

An assessment of current procedures available for prediction of concrete performance with MIBA and SSA was undertaken first, though it was already somewhat apparent from the analysis and evaluation sections that the information available in this area was limited. As such, this work is timely given the ever-increasing emphasis being placed on sustainability and absorbing RSMs within the construction sector, and can be useful for researchers and concrete designers. In contrast, a huge amount of work has been devoted to developing compressive strength models for normal concrete mixes over the years, building from Feret's Power model, way back in 1892, right up to the present. Although these models do not have provisions to specifically account for MIBA and SSA as cement or aggregate components, they were still considered closely, to inform the subsequent formation of the ash models.

In the prediction of ash-concrete compressive strength, priority was given to ensuring that the model could be practically usable, retaining a degree of simplicity and familiarity to what is already known. The aim was to develop a model for using incinerated ashes as a cement

component and another for use as an aggregate component, taking into account the ash's physical and chemical characteristics and providing an estimate of the 28-day strength when using either MIBA or SSA. These could also be later extended to become generic incinerated ash models. The final part of the modelling work is to provide a demonstration of how the model-estimated compressive strengths can be used as a tool to inform for concrete strength design, for mixes incorporating incinerated ashes from around the world.

7.2 Current Status of Modelling Work on Concrete using Incinerated Ashes

Modelling work undertaken on certain aspects of incinerated ashes, particularly related to their environmental impacts such as incinerator plant emissions and material leaching behaviour, has received significant interest. However, progress regarding their use in concrete-related applications, remains, at this point, in the early stages.

It is evident from Chapters 5 and 6 that the use of MIBA/SSA into concrete products, though achievable, is not without its challenges, which include questions regarding their reactivity, porosity and potential expansive behaviour. There has also been notable experimentation with how the ashes are processed prior to their use in concrete. These factors, combined with the high variability in the ash characteristics, make it difficult for individual researchers to obtain a sufficiently diverse and populous database to develop models that are adequately robust to be widely usable for estimating ash-concrete strength and for concrete design.

The initial modelling work that has been undertaken with MIBA and SSA is described in Table 7.1, and consists of their use in pastes, mortars, blocks and aerated concrete, as aggregate and cement components. This work mostly focused on establishing the correlation between various aspects of the product performance and the p-value significance from analysis of variance (ANOVA). Strong correlations between absorption and density (Series 1.1 and 1.2), compressive strength and ultrasonic pulse velocity (Series 1.1 and 1.2) and strength and porosity and bulk density (Series 2) of mixtures have been established. Regarding the mix proportions, Pearson correlation coefficients (measuring the linear correlation between two parameters) in Series 4 surprisingly demonstrated strong correlation between strength and

cement/aggregate ratio, and low correlation between strength and water/cement ratio. This finding makes the reliability of this work appear questionable, as it has been well established that the w/c ratio has a much greater impact on the strength, compared with the cement/aggregate ratio, and this perhaps suggests that the products in question, incorporating MIBA, were not performing effectively, or that there was a flaw in the model.

Table 7.1: Modelling work undertaken with SSA/MIBA in concrete-related applications

SERIES	PRODUCT	USE	WORK UNDERTAKEN
Sewag	e Sludge As	<u>sh</u>	
1.1	Pastes, Mortar, Blocks	5-20% cement addition. 10% sand replaced	Statistical summary, correlation matrix (with Pearson's product-moment correlation and p-value significance) and ANOVA. Variables considered: density, water absorption, capillary absorption, flexural & compressive Strength
1.2	Pastes, Mortar	10-30% cement replaced	Same procedure and variables examined as in Series 1.1, with new data from pastes and mortar mixes.
2	Aerated Concrete	60-80% cement replaced	Regression Analysis. Formulas for foaming ratio, density, absorption, compressive strength, from the cement/binder ratio, water/binder ratio and the aluminium powder content.
Munic	ipal Inciner	ated Bottom Ash	
3	Pastes	20-60% cement replaced	Main and interaction effects from orthogonal contrast coefficients. ANOVA. Polynomial response surface to predict compressive strength, based on the MIBA content and activator dosage, using linear regression.
4	Mortar paving units	60-80% cement replaced	Pearson correlation analysis. Correlation between cement/agg ratio, w/c ratio and various parameters: strength, absorption, permeability, abrasion, porosity and ultrasonic velocity.

ANOVA – analysis of variance, solids – cement + SSA, agg – aggregate. Information from Baeza et al. (2014); Baeza-Brotons et al. (2014), Wang et al. (2005a), Polettini et al. (2009); Kuo et al. (2015).

Formulas for foaming ratio, density, water absorption and compressive strength (f_c, shown in Equation 7.1) were developed in Series 2 (Wang et al., 2005a) for aerated concrete mixes containing SSA, based on the cement/binder ratio (c/b), water/binder ratio (w/b) and the aluminium powder (a/b) contents. Though useful, these formulas could be applicable only for the specialised aerated concrete mixes.

$$f_c = 0.481 \frac{c}{b} - 0.477 \frac{w}{b} - 0.184 \frac{a}{b}, \quad R^2 = 0.70$$
 (Eq. 7.1)

In addition, a polynomial expression developed in Series 3, presented as a surface response on a contour plot, illustrated the response of the compressive strength performance of cement pastes to the MIBA content and activator dosage. Although useful for its intended purpose of establishing the effectiveness of various activators, the model could not be widely applied for strength estimation of concrete-related products. Indeed, from the current work on the whole, it is clear that a model of this nature, which is suitable for estimating strength with the use of various MIBA and SSA samples, and potentially incinerated ashes in general, as both cement and aggregate components for varying mix designs, has not yet been developed.

7.3 Strength Models for Normal Concrete Mixtures

This section provides an overview of the strength models developed for normal concrete mixes. These models have also been expanded in recent years, to account for accepted RSMs such as coal fly ash, GGBS, silica fume, metakaolin and recycled aggregate. The latest models have broadened out to include different machine learning techniques such as artificial neural networks, logistic model trees and support vector machines, in addition to the regression analysis, as a statistical algorithm, that was adopted for the past models.

The first concrete strength model was developed by Feret (1892). The expression, shown in Equation 7.2, relates the compressive strength to the ratio of cement volume to total volume of cement + water + air.

$$f_c = A \left(\frac{V_c}{V_c + V_w + V_a} \right)^B$$
 (Eq. 7.2)

Where:

 V_c - volume of cement, V_w - volume of water, V_a - volume of air, A & B – coefficients.

Abrams (1919) proposed the somewhat related expression that connected compressive strength performance to the water/cement ratio by a power relationship (Equation 7.3). This expression has become extremely well renowned in concrete design owing to its simplicity and is generally referred to as Abrams' Law.

$$f_c = \frac{A}{B^{w/c}} \tag{Eq. 7.3}$$

Lyse (1932) (Equation 7.4) and Bolomey (1935) (Equation 7.5) later proposed further simplified linear forms of Abrams' model.

$$f_c = A + B. c/w \tag{Eq. 7.4}$$

$$f_c = A \left(c/w - B \right) \tag{Eq. 7.5}$$

Although the straightforwardness of these early models encouraged their adoption by engineers and designers, at the same time, there were also accuracy drawbacks associated with these simplified expressions. As such, building on the foundation of these equations, the subsequent work undertaken began to develop more multifaceted models, to achieve better representation of the various factors that determine concrete strength. In forming a picture of the history of the concrete strength models, the work of Chidiac et al. (2013), Moutassem and Chidiac (2016), Yeh (2006) and Neshat et al. (2012) served as useful references.

Powers and Brownyard (1946) developed a modified version of Feret's model that related strength to the gel/space ratio (X) (defined as the cement gel volume divided by the gel volume + capillary pores) (Equation 7.6). The gel/space ratio is expressed as a function of the water and cement contents, the volume of air and a new factor, α , which represents the degree of cement hydration, i.e., how much of the original cement is hydrated. Though not covered in detail in this project, a further series of models have also been developed to establish a value for α , taking into account factors such as the curing conditions (temperature, moisture and age), cement properties (composition and fineness) and mix proportions (w/c ratio and admixtures).

$$f_c = A(X)^B = A(\frac{0.66\alpha}{w + V_a + 0.32\alpha})^B$$
 (Eq. 7.6)

X - gel space ratio, α - degree of cement hydration

Karni (1974) later developed a modified expression for strength, based on the combination of the Bolomey (1935) and Powers and Brownyard (1946) models (shown in Equation 7.7).

$$f_c = A \left[\frac{\alpha x \, 100}{1.53 \left(\frac{w + V_a}{c} \right) + \frac{\alpha}{2.06}} - B \right]$$
 (Eq. 7.7)

A subsequent model was proposed by de Larrard (1999) (Equation 7.8) that took into account the contribution of the aggregate fraction to the concrete strength and the effect of the aggregate-cement bond.

$$f_c = KR_{c28}[A\log\left(\frac{t}{28}\right) + \left(\frac{V_c}{V_c + V_w + V_a}\right)^B]MPT^m$$
 (Eq. 7.8)

with MPT =
$$D_{max}(\sqrt[3]{\frac{\phi*}{\phi}-1})$$
 (Eq. 7.8.1)

Where:

K - paste to aggregate bond strength constant, R_{c28} - standard cement 28 day strength, t - age in days, MPT - maximum paste thickness, D_{max} - maximum aggregate size, ϕ - volumetric fraction of aggregate, ϕ^* - maximum aggregate packing density. Note: m is an additional model coefficient.

As a follow-on from the work of Karni (1974) and Powers and Brownyard (1946), Tango (2000) developed a model focusing on the effect of the cement proportion, though based on the activation energy, w/c ratio and curing age, rather than the α factor (Equation 7.9).

$$f_c = \frac{A}{B^{w/c} E^{w/c (t^{-0.5})} D^{t^{-0.5}}}$$
 (Eq. 7.9)

E - Activation energy, D - mean aggregate diameter

In 2008, as another variation of Abrams' Law, Popovics and Ujhelyi (2008) developed a model that took into account the cement content, separate from the w/c ratio, as they postulated that for a given w/c ratio, mixes with higher cement contents would exhibit lower strengths (Equation 7.10). Note: m is an additional model coefficient.

$$f_c = \frac{A}{B(\frac{w}{c} + mc)} \cdot 10^{-0.38 \, V_a}$$
 (Eq. 7.10)

Mechling et al. (2009) continued the adjustment of the strength estimation expression. In addition to parameters on aggregate packing and aggregate-cement bonding effects, the chemical composition of the cement was also considered, in the form of the C₃S content (Equation 7.11).

$$f_c = KR_{c28} \left(0.2 C_3 S - 1.65\right) \left[A \log\left(\frac{t}{28}\right) + \left(\frac{V_c}{V_c + V_w + V_a}\right)^B\right] MPT^m$$
 (Eq. 7.11)

Where:

C₃S - tricalcium silicate content of cement

Bringing together many parameters from previous models, Chidiac et al. (2013) developed an expression taking in the cement hydration factor, along with parameters on particle packing, cement-aggregate bond and cement type strength (Equation 7.12).

$$f_c(t) = K R_{c28} \left(\frac{APT}{D}\right)^A B^{\left(w + \frac{V_a}{c}\right)} \left(\alpha(t) - \alpha_{cr}\right)$$
 (Eq. 7.12)

With
$$\alpha(t) > \alpha_{cr}$$
 (Eq. 7.12.1)

$$\alpha_{cr} = m \cdot w/c$$
 (Eq. 7.12.3) $\alpha = \alpha_u \cdot \exp(-\left[\frac{\tau}{t_e}\right]^{\beta})$

$$\tau = 66.78\,p_{C3A}^{-0.154}p_{C3S}^{-0.401}F^{-0.804}.p_{SO3}^{-0.758} {\rm exp}(2.187p_{SLAG} + 9.5p_{FA}p_{FA-CaO}) \ \ ({\rm Eq.\ 7.12.4})$$

$$\beta = 181.4 \, p_{C3A}^{0.146} p_{C3S}^{0.227} F^{-0.535} \, . \, \, p_{SO3}^{0.558} \, \exp(-0.647 \, p_{SLAG}) \tag{Eq. 7.12.5}$$

$$\alpha_u = \frac{1.031 \, w/c}{0.194 + w/c} + 0.50 \, p_{FA} + 0.30 \, p_{SLAG} \le 1.0 \tag{Eq. 7.12.6}$$

$$t_e = \sum_{0}^{4} \exp\left(\frac{E}{R} \left(\frac{1}{293} - \frac{1}{T + 273}\right)\right) \Delta t$$
 (Eq. 7.12.7)

 α_{cr} - critical degree of cement hydration, α_u - ultimate degree of cement hydration, τ - hydration time parameter, β - hydration shape parameter, p_{C3A} , p_{C3S} , p_{SO3} , p_{SLAG} , p_{FA} , p_{FA-CaO} - weight ratio of C_3A , C_3S , SO_3 , slag, coal fly ash, coal fly ash-CaO per total cement, F - Blaine fineness, t_e - equivalent age, R - universal gas constant, T - concrete temperature.

As mentioned previously, the latest models began to depart from the conventional statistical regression analysis, to explore various machine learning techniques. Although these algorithms can be very useful in learning from the data and subsequently making predictions, the drawback is that they produce a system for estimating strength, rather than a single equation that can be readily transferred and used by engineers. A flavour of the work undertaken with these models for concrete mixes is given in Table 7.2.

Regarding the inclusion of RSMs in the concrete mix, some of the previously discussed models can be suitable for use: Chidiac's (2013) model has specific inputs for the weight ratio of coal fly ash and slag, whilst the machine learning algorithms of Deshpande et al. (2014) were designed based on the inclusion of recycled aggregate. Alternatively, an efficiency factor can be adopted to essentially convert coal fly ash to an equivalent cement fraction, as proposed by Babu and Rao (1996) (Equation 7.13). However, the challenge with this approach is in determining an appropriate value for K_{eff}, as experimental data tend to show a high degree of fluctuation. A further example is the work undertaken by de Brito and Saikia (2013), which developed a method for classifying recycled aggregate, to subsequently estimate the resultant strength performance of recycled aggregate concrete.

Table 7.2: Selection of machine learning modelling work on concrete strength

PUBLICATION	MODEL TYPE	INPUT PARAMETERS		
Bilgehan and Turgut (2010)	Artificial Neural Network	Ultrasonic pulse velocity		
Geet et al. (2016)	Artificial Neural Network	Cement grade, curing conditions		
Chou et al. (2014)	Multilayer perceptron neural network			
	Support vector machine	Mix proportions, admixtures,		
	Classification and regression tree	age, entrapped air content		
	Linear regression			
Neshat et al. (2012)	Fuzzy logic	w/c ratio, slump, aggregate type and maximum size, age		
Saridemir (2009)	Artificial Neural Network	Age, mix proportions, admixture usage		
	Artificial neural network	Mix proportions including		
Deshpande et al. (2014)	Model Tree	recycled aggregate		
	Non-linear regression	replacement level		

$$w/c = \frac{w}{c + K_{eff}.FA + GGBS}$$
 (Eq. 7.13.1)

 K_{eff} - efficiency factor, FA – coal fly ash content, GGBS - ground granulated blastfurnace slag content.

7.4 Development of Strength Models with Incinerated Ashes

Based on the assessment of strength models for normal concrete, a number of options were available for developing models incorporating incinerated ashes. However, a balance should be struck in that the model should deliver satisfactory accuracy, yet avoid becoming overly intricate to a point that would discourage its adoption in practice. It was also envisaged that the model would not contain parameters that are difficult to obtain or that fall outside of the normal testing for concrete mix designs in practice. Indeed, the nature of the data obtained for incinerated ash mixes, originating from various countries worldwide, with no fixed testing

methodology, automatically requires that any variables used in the model must be commonly available.

The collective data considered for modelling the strength performance of MIBA and SSA in concrete applications consisted initially of more than 450 mixes, produced in over 20 countries. This involved their use as cement and as fine and coarse aggregate components, though owing to its finer particle size distribution, SSA is more commonly utilised as a cement replacement and to a lesser degree as aggregate, whilst the reverse is true for MIBA. With the aim of developing models that could potentially be applied to incinerated ashes in general, based on performance, rather than to a specific material, the combination of the data for the two ashes somewhat balances out the split between the aggregate and the cement sample sizes, and most importantly, there are adequate results in each dataset to develop both cement and aggregate models. The data selection comprised normal mortar and concrete mixes. Other application types covered in Chapters 5 and 6, such as raw feed for cement clinker production, lightweight aggregate concrete, aerated concrete and foamed concrete were not included.

Of the models covered in section 7.3, the power relationship between the strength and the w/c ratio given in Abrams' law stands out as the one that engineers are very familiar with and is widely used in mix design. As such, when weighing the pros and cons of various model types, the concept of utilising Abrams' law as the foundation for developing the incinerated ash model became an obviously appealing choice.

The selection of a multiple regression statistical modelling approach offered the control to structure the model in the form of Abrams' law and subsequently build in further parameters to best fit the strength performance of the incinerated ash-concrete. Implementing the concept of Abrams' law as the starting point for the statistical model also somewhat lessens the manual fine-tuning required, though clearly this is an aspect in which the computation power of machine learning is superior. Indeed in one case of modelling the strength of recycled aggregate concrete (Deshpande et al., 2014), it emerged that the accuracy of the

strength estimation was greater for the artificial neural network compared to the nonlinear regression model.

Overall, both modelling approaches have their advantages, and both were seen as viable options, though for these particular data, taking the concept of developing from Abrams' strength to w/c ratio relationship, the statistical regression analysis modelling was clearly the preferred choice. MATLAB was selected as the software package to undertake this work.

The first stage of the statistical modelling was the preparation of the data. The collective strength data for MIBA and SSA mixes were organised, along with the associated information on the ash processing and characteristics, replacement levels, mix proportions, curing conditions and specimen type. A summary of these data is presented in Table 7.3. It should be noted that for the cement model, the focus was more on the chemical properties of the ash in its ground reactive form, whilst the physical properties were more relevant for the aggregate model.

It is evident that there is a wide spread in almost all aspects of the model data (mix details, ash processing and ash characteristics). The fact that the data population is so diverse should contribute to greater reproducibility of the model, which ultimately should encourage greater uptake in practice. At the same time, the looser control is also likely to lead to greater variance in the results, which could limit the level of precision that is achievable. Managing incompleteness in the data was another major challenge in developing the model. Although the model was aiming to consider only key parameters, it is still not uncommon to find that, for example, for a series of mixes, the cement grade class and curing conditions may not be specified, or that the ash water absorption may not have been measured. At times, this limited how certain parameters could be incorporated into the model, whilst in other cases, average values could be filled in as a workaround.

Table 7.3: Summary of initial data for the incinerated ash aggregate and cement models

PARAMETER	AGGREGATE MODEL	CEMENT MODEL
Incinerated Ashes	MIBA, SSA	MIBA, SSA
Products	Mortar, Concrete	Mortar, Concrete
Specimens	Cubes, cylinders, prisms	Cube, cylinders, prisms
Ash Use	Fine and Coarse Aggregate	Cement component
Ash Processing	Pre-treatment, Sieving, chemical, thermal	Pre-treatment, sieving, grinding, chemical, thermal
Replacement level, %	0-100	0-60
Curing conditions	Water, air. RH >90-100%. Temperature: 19°C, 20°C, room temperature	Water, saturated lime. RH 100%. Temperature: 20, 23, 40°C, room temperature
Age, days	1-180	1-360
w/c	0.21-0.86	0.36-0.77
Strength (28 days), MPa	8-60	5-66
Cement types	CEM I 52.5 N/R, CEM I 42.5 N/R, CEM I 32.5 N, CEM II/BL 32.5R, Type I PC, OPC, PC type 425, PC+FA, SPC 42.5 N	CEM I 52.5R/N, CEM I 42.5R/N, CEM I 32.5N, CEM II/A-LL 42.5R, CEM II/B-L 32.5N, CEM II/B-M(V-LL) 42.5R, OPC, PC+FA, MS 522
Admixtures	Superplasticiser	Superplasticiser
Ash Oxide Composition	-	SiO ₂ 10.7-58.1%, CaO 3.7-39.0%, Al ₂ O ₃ 5.1- 26.3, Fe ₂ O ₃ 1.9-21.8%
Ash SiO ₂ /CaO ratio	-	0.36-13.2
Ash fineness after grinding	-	D ₅₀ 2-130 μm, D _{max} 2-600 μm, Blaine fineness 330-1578 m ² /kg
Ash LOI, %	0.67 - 12.1	1.8-24.8
Ash Grading	$D_{60}/D_{10} = 2.1-19.7$ $D_{30}^2/(D_{10} \times D_{60}) = 0.5-2.6$	-
Ash Absorption, %	0.7-50	-
Ash Specific Gravity	1.0-2.8	-

At the beginning of the model building, the following decisions on the model constraints and assumptions were made:

- The model would be for 28 day compressive strength. This is the curing age that is
 used in concrete specification and design, and is also where the bulk of the strength
 results lie.
- The model focused solely on the mortar and concrete mixes containing MIBA or SSA.
 As shown in section 7.3, there have been many models developed for normal concrete mixes and the focus of this work was very much on the performance with incinerated ashes.
- The incinerated ashes were incorporated as a percentage of cement or aggregate replacement, rather than as a mass per cubic meter. The model would be designed to establish the overall effects of the incinerated ash, relative to the control mix strength, and as such, data on the base performance of the control mix are required.
- Cylinder strengths were converted to cube strengths. Table 12 of BS EN 206 (2013) adopts cylinder/cube strength ratios varying from 0.78 to 0.83, for strength classes up to C70/85. Based off this, a conversion factor of 0.8 was adopted in this study.
- As established in chapter 5, certain mixes with MIBA were prone to exhibiting
 damaging expansive behaviour, brought about by the reaction of the metallic
 aluminium in the alkaline cement mix. As strength and indeed all aspects of
 performance were greatly diminished, these concrete mixes would not be suitable
 for use and as such were removed from the model data.

In fleshing out the theory on the factors affecting the ash-concrete strength, three key parameters were identified:

- i. The w/c ratio, which has a determining effect on the mix pore structure
- ii. The strength of the control mix, which provides positioning from which the effect of the ash is evaluated.
- iii. The ash replacement level, which dictates the magnitude of the effect of the ash.

There are a large number of additional factors that can have an impact on the strength, but to a lesser extent, most of which have been referred to in Table 7.3. Using these parameters, along with an understanding of concrete technology, the ash characteristics and their

performance in concrete, the strategy for building the strength model was to add in the parameters, one by one, starting from Abrams' law with w/c and working through the important factors. To restrain the model from becoming overly bulky and developing overfitting problems, the parameters used in the equation were limited to those that had a significant effect on the strength estimation accuracy and, at the same time, exhibited behaviour that was consistent with the principles of concrete technology.

In undertaking the regression analysis, the function 'fitnlm' was utilised in the MATLAB script. Fitnlm is a statistical toolbox function that uses a Levenberg-Marquardt least squares algorithm to minimize the errors between the observed and predicted values of a model. This function provides a wide-array of useful statistical measures and was identified as a suitable tool to undertake the regression analysis required to develop the models. Values were obtained for the regression coeficients and the associated measures of the model fit, including the R² value, root mean square error (RMSE), t-statistic and p-value, as well as further diagnostic information on the standardised residuals, Cook's distance and leverage. The R² value was calculated from the total sum of squares (SST), which itself is calculated from the sum of squared errors (SSE) + the regression sum of squares (SSR), as shown in Equation 7.13. The adjusted R², which subsequently takes into account the number of coefficients, was the value referred to when evaluating the fit of the incinerated ash models.

$$R^2 = \frac{SSR}{SST} = 1 - \frac{SSE}{SST} \tag{Eq. 7.13}$$

The RMSE provides a measure of the standard deviation of the model error (Equation 7.14).

$$RMSE = \sqrt{Mean((Measured_Strength - Estimated_Strength)^2)}$$
 (Eq. 7.14)

The t-statistic and p-value were used together to offer a measure of the statistical significance of the variables. The higher the t-value and the lower the p-value, the greater the likelihood that the variable is significant. Typically a 5% significance level is adopted, and as such, p-values < 0.05 indicate that the null hypothesis is rejected and, in effect, the variable in question is deemed to have a statistically significant effect on the strength. An additional plot of the residual vs fitted values is useful to check if the error is randomly distributed. Any patterns

emerging in this plot suggest that the model is not appropriate in its current form and needs to be adjusted.

Cook's distance measures the change in the model when a data point is removed from the dataset. Leverage measures how far away the independent (predictor) variable value is from the other values in the dataset. The standardised residual is the ratio of the residual divided by the standard deviation. Plots of these three parameters were used in the model diagnosis to identify the influential points and potential outliers.

For the model validation, the approach taken was to set aside a random 15% of the data population for solely this purpose and use the other 85% to build the model. It was subsequently decided that this process would be repeated several times, taking multiple random selections of 85% of the data and validating with the remainder, as an improved validation process.

7.5 Cement Model

The developed model estimates the compressive strength of concrete/mortar using SSA and MIBA as cement components. The model takes the form of Abrams' law, with ash characteristics and additional mix design parameters. Thus, the model outline format is presented in Equations 7.15.1 and 7.15.2:

$$f_c = \frac{A}{B^{w/c}}$$
 (Eq. 7.15.1)

where,

$$A = A_1 (f_c'^{0.9}) \times A_2 \frac{w/c'}{w/c} \times (1 - A_3.r) \times \left(1 + [A_4.F_{High}.r] + [A_5.\frac{SiO_2}{CaO}.r]\right) \times \left(1 + [A_6.P_{Fur}.r] + [A_7.W_{Control}.r]\right)$$
(Eq. 7.15.2)

 A_1 , A_2 , A_3 , A_4 , A_5 , A_6 , A_7 , B, are coefficients.

 $f_c = Ash mix strength (28 days) in MPa,$

f_c'= Control mix strength (28 days) in MPa,

w/c = Ash mix water cement ratio,

w/c' = Control water cement ratio,

r = Cement replacement level (as a fraction),

 F_{High} = Ash classified as high fineness,

 P_{Fur} = Ash subjected to further processing (washing, chemical, thermal treatment),

 $W_{Control}$ = Ash mixes, where the water content is controlled.

Note: F_{High} , P_{Fur} , and $W_{Control}$ are categorical variables which = 1 if true, or otherwise = 0.

For fineness, ash is classed as high fineness if any of the following conditions are satisfied:

- $D_{50} < 30 \mu m$
- $D_{max} < 75 \mu m$
- Blaine Fineness > 400 m²/kg

The above equation shows the full form of the strength model using incinerated ashes as cement components. However, Equation 7.15.2 contains three categorical dummy variables used to accommodate the ash fineness, processing and water control of the mix.

Examining the form of the equation, it is apparent that the bottom line of Equation 7.15.1 remains unchanged from Abrams' law, which is simply a constant to the power of the w/c. On the top line, the first part of Equation 7.15.2 brings in the 28-day compressive strength of the control mix. The power of 0.9 signifies that MIBA/SSA causes greater relative reductions from the control strength as the absolute strength of the mix increases.

The second term of Equation 7.15.2 $(A_2^{\frac{w/c'}{w/c}})$ accounts for adjustments in the w/c ratio when partially replacing the cement with MIBA or SSA. The most likely example of this is when the designer decides to slightly lower the w/c ratio when using the ash, to target a strength equal to the control mix.

The third term $(1 - A_3.r)$ accounts for the ash replacement level, independent of the properties of the ash. It should be noted that the replacement level is inserted as a fraction, i.e., 10% replacement is inserted as 0.1.

The next part of the equation factors in the fineness and chemical composition of the ash, both of which are connected to the reactivity. Measures such as the CaO/SiO₂ and Al₂O₃/SiO₂ ratios, for GGBS, and SiO₂+Al₂O₃+Fe₂O₃ for coal fly ash are commonly used to assess the reactivity of these RSMs. After various combinations were experimented with, the SiO₂/CaO ratio of the ash, which is inversely proportional to the strength, was found to exhibit the best correlation with strength, though its influence is less than that of the other factors in the model.

The reactivity of cement components, and the associated strength are known to increase with increasing fineness. To account for this, the ash samples were categorised into two groups, low and high fineness, based on criteria determined for the maximum particle size after grinding (D_{max}), the D_{50} value (50% of the ash particles pass this size) or the Blaine fineness. The fineness levels were set according to what is achievable in present-day practice and produced a close to even split in the number of ash samples in each. Expected higher strengths in the higher fineness category were well reflected in the data. However, as the quantity of data on this aspect rises, some tweaking of these fineness limits may be desirable in the future.

The last two terms in Equation 7.15.2 relate to further processing of the ash and regulating the water demand of the mix. Beyond grinding of the ash, further washing, chemical or thermal treatments have commonly been implemented, mostly with MIBA, to improve its reactivity and/or to diminish negative effects, which has led to enhanced performance. As the data on ash-processing also continues to develop, the model can evolve in future to separately account for the effects of the individual washing, chemical and thermal treatments.

Regulation of the water content of the mix, achieved usually, though not necessarily always, by using water-reducing admixtures, also indirectly improved the strength of the ash-concrete. It has been established that both SSA and MIBA increase the water demand, owing to their morphology and pore structure (water absorption). The admixture becomes useful in reducing any interference of the ash with the moisture movement and the hydration process, and, as such, contributes to greater strength.

Other factors such as the amorphous fraction and porosity of the ash are likely to have played a role in the concrete strength development. Data available on these parameters did not lend themselves to their inclusion, though in any case, with the aim of encouraging the use of the model as much as possible, it would be counterproductive to include parameters that are not readily available in most cases.

As discussed previously, a random sample of 85% of the total data was selected to build the model and the remainder was kept for validation. This process was repeated for six iterations, using different random selections of the data each time. This produced a series of regression coefficient estimates, standard errors, t-statistics and p-values for each of the iterations. These data are presented in Table 7.4, though the t-statistic results were omitted to keep the table neater. Associated data on the RMSE, R² and adjusted R² value for each iteration of the model building are given in Table 7.5, along with the RMSE determined from the residual validation data (15% of the total).

Plugging the attained regression coefficients back into Equations 7.15.1 and 7.15.2, an approximate understanding of the contribution of each of the factors can be attained. With control strength values reaching up to 66 MPa, and associated coefficients ranging from 0.79 to 0.98 (six iterations of A_1) it is clear that this part plays a key role. Coefficients for A_2 fluctuated above and below 2, and as such, when the ash mix w/c ratio differs from the control w/c ratio, there will be a notable shift in the model prediction. Values close to 1 were attained for A_3 , which accounts for replacement level, which suggests that if the ash fineness was low, with no further processing and the water was not regulated with a water reducing admixture, there would be roughly a 1:1 ratio of strength loss per ash replacement percentage. For the three dummy variables on the ash fineness, processing and use of superplasticiser, coefficients from 0.54 to 0.73, 0.62 to 0.81 and 0.50 to 0.67 were determined for A_4 , A_6 and A_7 , respectively. Regarding the SiO₂/CaO ratio, the associated coefficient values for A_5 ranged from -0.15 to -0.17. These negative values match the expected inverse relationship between SiO₂/CaO and strength. Values for the remaining B coefficient, which is raised to the power of the w/c, ranged from 1.44 to 1.63.

Table 7.4: Coefficient estimates, standard error and p-values for six iterations with the cement model

PARAMETER	COFF	FACTOR	RESULTS								
TANAMETER	COLI.	TACTOR	Iteration 1	Iteration 2	Iteration 3	Iteration 4	Iteration 5	Iteration 6			
Coefficient	В	/s	1.438	1.575	1.534	1.508	1.621	1.633			
Estimate		w/c		0.922	0.980	0.872	0.873				
Estimate	A ₁	f _c '	0.792					0.965			
	A ₂	w/c′	2.378	2.117	1.975	2.236	2.244	2.065			
	A ₃	r	1.121	1.073	1.130	1.121	1.030	1.110			
	A_4	F _{High}	0.726	0.729	0.538	0.712	0.589	0.607			
	A ₅	SiO ₂ /CaO	-0.171	-0.160	-0.155	-0.168	-0.158	-0.149			
	A_6	P_{Fur}	0.712	0.723	0.810	0.641	0.619	0.753			
	A ₇	W _{Control}	0.629	0.497	0.826	0.519	0.665	0.657			
Standard	В	w/c	0.282	0.305	0.304	0.328	0.301	0.320			
Error	A_1	f _c '	0.204	0.230	0.237	0.242	0.204	0.238			
	A_2	w/c'	0.538	0.428	0.392	0.490	0.436	0.414			
	A_3	r	0.073	0.074	0.072	0.073	0.076	0.074			
	A_4	F_{High}	0.145	0.135	0.143	0.142	0.133	0.139			
	A_5	SiO ₂ /CaO	0.025	0.024	0.026	0.028	0.024	0.026			
	A_6	P_{Fur}	0.181	0.168	0.184	0.191	0.169	0.174			
	A ₇	W_{Control}	0.186	0.171	0.206	0.206	0.184	0.190			
P-Values	В	w/c	1.31E-06	9.84E-07	1.60E-06	1.08E-05	3.83E-07	1.30E-06			
	A_1	f_c	1.69E-04	1.05E-04	6.80E-05	4.53E-04	3.87E-05	8.78E-05			
	A_2	w/c'	2.23E-05	2.51E-06	1.75E-06	1.25E-05	1.08E-06	2.11E-06			
	A_3	r	6.42E-30	8.25E-28	1.60E-30	5.51E-30	5.11E-26	5.15E-29			
	A_4	F_{High}	2.11E-06	3.33E-07	2.74E-04	1.82E-06	2.20E-05	2.87E-05			
	A_5	SiO ₂ /CaO	2.45E-10	7.41E-10	2.11E-08	1.53E-08	1.33E-09	5.71E-08			
	A_6	P_{Fur}	1.46E-04	3.37E-05	2.37E-05	1.05E-03	3.60E-04	3.02E-05			
	A_7	W_{Control}	9.89E-04	4.42E-03	1.08E-04	1.31E-02	4.48E-04	7.39E-04			

COEF - Coefficient

Table 7.5: RMSE and R² values for cement model and validation data RMSE

		RESULTS							
PARAMETER	Iteration 1	Iteration 2	Iteration 3	Iteration 4	Iteration 5	Iteration 6			
Model Building (r	andom 85% of t	he data, 126	observation	s)					
RMSE	5.23	5.08	5.24	5.39	4.99	5.19			
R^2	0.852	0.861	0.866	0.845	0.87	0.862			
R ² adjusted	0.843	0.853	0.858	0.836	0.862	0.854			
Model Validation	(remaining 15%	6 of the data,	23 observat	ions)					
RMSE	5.02	5.74	5.16	3.96	6.46	5.09			

As a measure of the deviation of the coefficients, it is evident from Table 7.4 that the standard error was the largest for the A_2 term, ranging from 0.39 to 0.54. This is not surprising as the coefficient values of A_2 were also the largest. Indeed, in relative terms, the errors for A_7 , A_6 and A_1 were larger. The variation in the coefficient values across the six iterations provides additional insight into this aspect. It is apparent that although some fluctuations are present, there are no wild swings or major realignments of the model coefficients.

The p-values shown in Table 7.4 are all below the 5% significance level (0.05 value). Results for A_7 were the only ones that came close to this limit, reaching a maximum p-value of 0.013 on the fourth iteration.

The adjusted R² value was the main parameter used in judging and subsequently fine-tuning the fit in the model building stage. As shown in Table 7.5, values from 0.84 to 0.86 were obtained for the completed model. As an additional measure of the magnitude of the error, the model yielded RMSE values of approximately 5 MPa.

As part of the validation process, additional RMSE values were calculated using the remaining 15% of the data, yielding values from 3.96 to 6.46. It is evident that, for a particular iteration, as the R² improved and RMSE values for the main model building moved upwards, the validation data RMSE value tended to move slightly downwards, depending on which samples were randomly selected for each part. Overall, the ranges of RMSE values determined for the main model and the validation data are comparable, which endorses the validity of the model, in this regard.

Although the six iterations of the model were useful in the analysis and validation of the data, the coefficients needed to be fixed to single values in the final model to be adopted by others. Rather than selecting the coefficients from the iteration that produced the highest adjusted R² value, the decision was made to try to match the RMSE of the model building data and the validation data, whilst minimising these errors and maximising the adjusted R² value for both

sets of data. Based on this, the coefficients from iteration 6 were selected. As such, the final model is given in Equations 7.16.1 and 7.16.2:

$$f_c = \frac{A}{1.63^{w/c}} \tag{Eq. 7.16.1}$$

With:

$$A = 0.97. (f_c'^{0.9}) \times 2.07^{\frac{w/c'}{w/c}} \times (1 - 1.11.r) \times \left(1 + [0.61. F_{High}.r] - [0.15. \frac{SiO_2}{CaO}.r]\right) \times \left(1 + [0.75. P_{Fur}.r] + [0.66. W_{Control}.r]\right)$$
(Eq. 7.16.2)

Diagnostics

During the process of building, fine-tuning and validating the model, additional diagnostics were also being undertaken concurrently, examining the raw residuals, leverage, standardised residuals, Cook's distance and actual vs predicted strengths. As the coefficients from the sixth iteration were selected for the final model, the associated diagnostic information for this iteration is presented in this section.

Part of the process of fine-tuning the model was managing potential outliers. This aspect was assessed using leverage, Cook's distance and standardised residuals determined using the MATLAB function 'fitnlm' with the model building data. The results for these parameters are presented in Figures 7.1, 7.2 and 7.3, respectively,

Although there are no cast-iron rules for classifying and removing outlier data points, there are certain guidelines that can be adopted when evaluating the leverage, Cook's distance and standardised residuals. It is common to consider leverage values exceeding $2 \times L_{sum}/n$ as high (where L_{sum} is the sum of the leverages, n is the number of samples). A Cook's distance threshold of 3x mean Cook's distance is also commonly adopted, though it has also been postulated by Weisberg (2005) that the removal of points with values markedly less than 1 should not greatly affect the model. Standardised residual values outside the range from -2 to +2 have been recommended as outlier limits (Sheather, 2009).

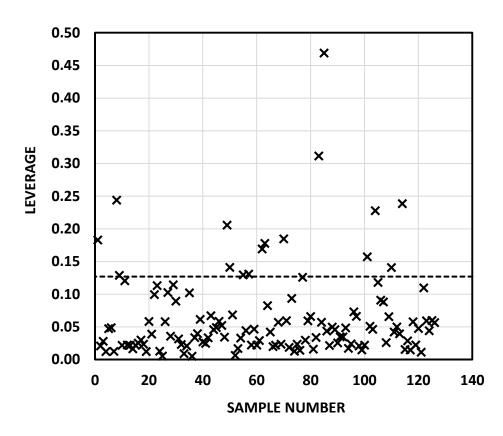


Figure 7.1: Leverage values for the cement model building data

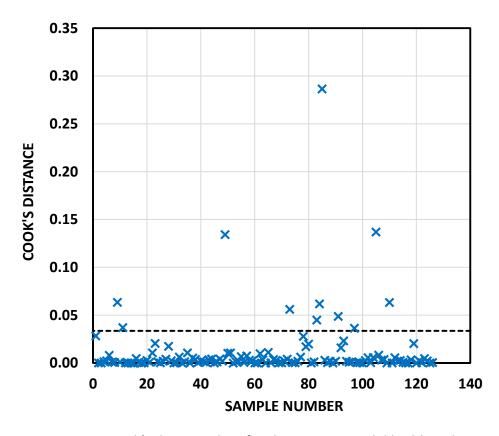


Figure 7.2: Cook's distance data for the cement model building data

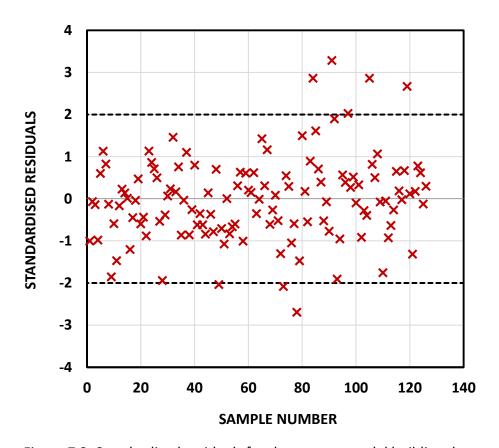


Figure 7.3: Standardised residuals for the cement model building data

These respective leverage, Cook's distance and standardised residual limits are shown as dotted lines in Figures 7.1, 7.2 and 7.3. These guidelines were very useful for identifying weaknesses and aspects of the strength model that had not been accounted for during the development process. However, given that this model is breaking new ground in predicting concrete strength performance with incinerated ashes, a cautious approach was adopted regarding the removal of outliers from the final model. Although the strength of this work is that it avails of global data on incinerated ashes, this inevitably leads to some deviations. It is expected that this model can continue to evolve as the work with incinerated ashes continues, and as such, to get the most out of the current data and provide the best opportunity for further growth, the decision was made to remove data points only when there was a strong reason to, and not to simply discard points that did not fit.

The data points that fell outside the aforementioned Cook's distance or standardised residual limits, which could be considered as potential outliers, are presented in Table 7.6. On re-

examining the source of these concrete mixes, no obvious reasons were evident to suggest invalidity that would prompt the removal of these data points. It is evident that out of the 12 potential outliers, five were considered as high leverage points, and of those five, two points exceeded the three respective Cook's distance, leverage and standardised residual limits. Nevertheless, despite the definite improvements in the R² that would be gained by removing these points, the conservative decision of including this information in the model was made in this foundational work on incinerated ash concrete strength modelling.

Table 7.6: Potential outlier data points in the ash-concrete cement model

SAMPL NO.	E ASH	r, %	w/c	f _c , MPa (ACTUAL)	f _c , MPa (ESTIMATED)	COOK'S DISTANCE	LEVERAGE	STANDARDISED RESIDUAL
9	SSA	26	0.5	25.9	34.9	0.064	0.129	-1.86
11	MIBA	10	0.36	42.2	49.4	0.037	0.121	-1.47
49	MIBA	30	0.7	42.0	51.4	0.134	0.206	-2.04
73	SSA	50	0.5	12.0	22.3	0.056	0.094	-2.09
78	SSA	15	0.6	13.9	27.7	0.028	0.030	-2.70
83	MIBA	30	0.5	66.0	62.2	0.045	0.312	0.89
84	SSA	20	0.5	55.0	40.6	0.062	0.057	2.86
85	SSA	50	0.5	50.7	44.6	0.287	0.469	1.61
91	MIBA	20	0.48	53.9	37.2	0.049	0.035	3.29
97	SSA	30	0.5	46.4	36.2	0.036	0.066	2.03
105	MIBA	20	0.51	51.0	37.1	0.137	0.118	2.86
119	SSA	20	0.5	60.0	46.3	0.022	0.022	2.67

Red – marks the values that exceed the Cook's distance limit of 0.034, standardised residual limits of < -2 or > +2, or the leverage limit of 0.127.

There was, however, one additional data point that was identified as an outlier in the model building process. This consisted of a mortar mix containing SSA as a replacement of 60% of the cement, which exhibited a huge strength decrease, to 5 MPa from the initial control strength of 62 MPa (Alcocel et al., 2006). The use of pozzolanic materials at this high replacement level is not recommended and it is clear that the hydration process was completely compromised in this case. As such, this result was justifiably removed from the model building data set. This point, if included, would have also yielded Leverage, Cook's

distance and standardised residual values of 0.146, 0.114 and -2.31, respectively, all of which exceed the identified outlier limits.

Additional plots of the raw residuals vs the fitted values were used throughout the model building process to check that the errors were randomly distributed. The last version, based on the final cement model data, is presented in Figure 7.4. No definitive patterns stand out in the data. It is apparent that the bulk of the data lies in the central region, with strengths from 20 to 50 MPa, and accordingly the greatest spread in the results is evident there. The distribution of the positive and negative errors appears relatively even. Based off this analysis of the residuals, the model was judged to be valid in this regard.

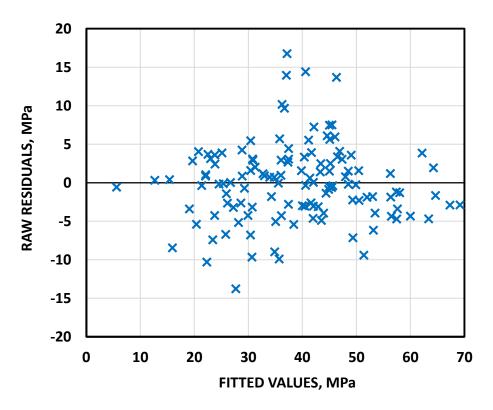


Figure 7.4: Assessment of the raw residuals for the cement model

The final piece of information on this cement model consisted of plots of the actual measured concrete strength vs the predicted strength, for both the model building data (85% of the total) and the validation data (15% of the total), as shown in Figure 7.5 (a) and (b), respectively. The line of zero error, for which the predicted strength is equal to the actual strength, is also shown in these Figures.

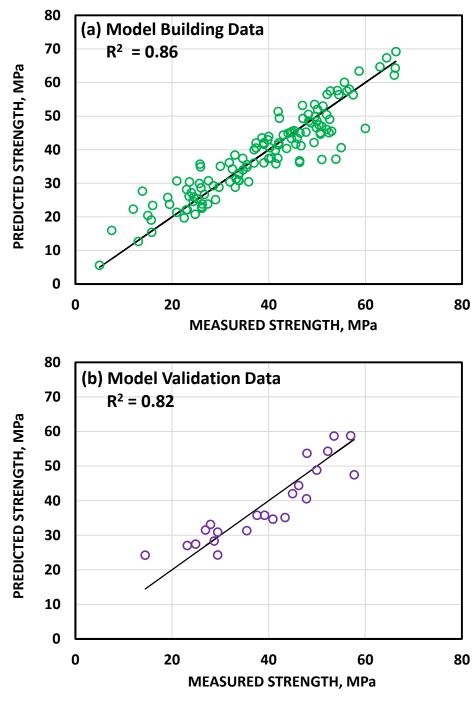


Figure 7.5: Predicted strength vs measured strength for cement model (a) building data and (b) validation data

The estimated strength results show good correlation with the actual measured values. For both the model building data and the validation data, the results, on the whole, lie close to the line of zero error. Some deviations are apparent around the 50 MPa region, though this

stems mainly from the deliberate decision to avoid discarding a small number of the rarer results in this opening work on the prediction of concrete strength performance with incinerated ashes.

As one of the aims of this work, the model can serve as a useful tool for engineers in formulating initial trial concrete mix designs using MIBA and SSA as cement components. It is envisioned that as the work with incinerated ashes in concrete continues to gain momentum, driven by increasing emphasis on sustainability in construction, the model can evolve and, indeed, potentially be extended to be applicable for incinerated ashes in general.

7.6 Aggregate Model

The overall approach for the aggregate model was kept consistent with the previous cement model. As such, to avoid repetition, less emphasis was placed on the method in this section. Following the same Abrams' law format, the aggregate model developed is presented in Equations 7.17.1 and 7.17.2.

$$f_c = \frac{A}{B^{w/c}}$$
 (Eq. 7.17.1)

where,

$$A = A_1 \left(f_c'^{0.8} \right) \times A_2^{\frac{w/c'}{w/c}} \times (1 - A_3. \ln(r)) \times (1 + [A_4. P_{Fur}. r] + [A_5. W_{Control}. r]$$

$$+ [A_6. G_{Well}. r] + [A_7. I_{DAL}. r])$$
(Eq. 7.17.2)

A₁, A₂, A₃, A₄, A₅, A₆, A₇, B, are regression coefficients

 f_c = Ash mix strength (28 days) in MPa,

 $f_c' = Control mix strength (28 days) in MPa,$

w/c = Ash mix water cement ratio,

w/c '= Control water cement ratio,

r = Aggregate replacement level (as a fraction),

 $P_{Fur} = Ash$ subjected to further processing (washing, chemical or thermal treatment),

 $W_{Control}$ = Ash mixes, where water content is controlled.

 $G_{\text{well}} = Ash$ categorised as well graded,

 $I_{DAL} = Ash$ with an inhibiting factor, low density/high absorption/high LOI.

Note: P_{Fur} , $W_{Control}$, G_{Well} , I_{DAL} are dummy variables which = 1 if true, or otherwise = 0.

For ash to be classified as well-graded, the following criteria are required to be met:

- For fine aggregate: $\frac{D_{60}}{D_{10}} > 6$ and $\frac{D_{30}^2}{D_{10} \times D_{60}}$ 1-3.
- For coarse aggregate: $\frac{D_{60}}{D_{10}} > 4$ and $\frac{D_{30}^2}{D_{10} \times D_{60}}$ 1-3.

Inhibiting factor (I_{DAL}) is incurred if the ash exhibits any of the following properties:

- Density $< 2100 \text{ kg/m}^3$
- Absorption > 15%
- LOI > 6%

The aggregate model has similarities with the cement model, particularly regarding the first five terms of the top line, accounting for the control mix strength, control w/c ratio, ash replacement level, ash processing and water control of the mix. Additional parameters on the ash characteristics, focusing mainly on the physical properties, are also included.

Aggregate grading plays a key role in determining the particle packing, and consequentially the concrete density and strength, as well as its workability and stability. The challenge with this model was to determine the most appropriate way to account for this aspect. The past British standard, BS 882 (1992), specified full grading envelopes for fine and coarse aggregate, and indeed the former fine limits included three subdivisions (coarse, medium and fine). Aggregate grading in the current standard, EN 12620 (2002), is more open, specifying percentage passing limits for fine aggregate for just the top 15% of the particle size distribution. Although these limits regulate sand grading, they have been found to be less suited to evaluating ash grading for strength prediction. Given that all ash samples input into the model are likely to be earmarked for use in concrete in accordance with a particular standard, there is a concern that a grading parameter in the model that followed the exact requirements of a standard would end up redundant.

Exploring further options, the BRE (1988) concrete mix design method does not adopt the BS 882 grading curves, stating that these limits were too wide for mix design purposes. Instead the grading is taken into account based on the percentage of the fine aggregate passing the 0.6 mm sieve, though maximum particle size and aggregate type (crushed/uncrushed) are also considered. This measure is used to determine the fine aggregate proportion based on the workability and maximum particle size, though there is an obvious limitation in representing the grading as a single value. As such, considering the pros and cons of grading measures in the standards and the BRE mix design method, along with the physical properties of the incinerated ashes, an amalgamation of the two was adopted in this model. As outlined after Equation 7.17, the grading of the MIBA/SSA aggregate was classified based on the distribution of their D_{10} , D_{30} and D_{60} sizes. The sensitivity of this method was more suitable for evaluating ashes assigned for use as aggregate. Approximately half of the obtained incinerated ashes met the well graded criteria, and indeed, superior strength performance was reflected in these well graded samples.

The inhibiting factor (I_{DAL}) takes into account the strength reduction associated with ash samples with low densities, high water absorption and high LOI values. Efforts were made to directly include these parameters during the model building process; however, incompleteness found in the data suggested that in many cases the full set of values may not be available to the engineer. As a more flexible alternative, samples that exhibited unfavourably extreme values for any of these three related properties were grouped together. Very high water absorption and low density values are a strong sign of high porosity, whilst high organics also reduce the density, encourage higher water retention and cause weak pockets in the material, all of which translate into lower strength.

The aggregate/cement ratio was also considered during the model building process, though this parameter is generally considered to be a minor factor and was found not to show a strong correlation with strength. In addition, it was interesting to find that there was no significantly different relative effect on strength when using the ash as a coarse or fine aggregate component.

Using the same approach as the cement model, a random selection of 85% of the data was used to build the model and the remaining 15% was set aside for validation. Coefficient estimates, standard errors and p-values determined for six iterations of this process are shown in Table 7.7 and again, for neatness, the additional t-statistic results are not included. Accompanying RMSE, R² and adjusted R² values are presented in Table 7.8.

Table 7.7: Coefficient estimates, standard error and p-values for six iterations with the aggregate model

DADAMETED	COEE	FACTOR	RESULTS						
PARAIVIETER	COEF.		Iteration 1	Iteration 2	Iteration 3	Iteration 4	Iteration 5	Iteration 6	
Coefficient	В	w/c	0.938	1.110	1.074	1.003	0.914	1.035	
Estimate	A1	f _c '	0.423	0.454	0.414	0.420	0.407	0.451	
	A2	w/c'	1.482	1.425	1.543	1.521	1.529	1.432	
	А3	r	0.814	0.893	0.878	0.832	0.807	0.860	
	A4	P_{Fur}	1.055	1.048	1.063	1.044	0.994	1.038	
	A5	W_{Control}	0.816	1.022	1.005	0.920	0.898	0.891	
	A6	G_{Well}	0.673	0.821	0.871	0.695	0.833	0.751	
	A7	I_{DAL}	-0.381	-0.435	-0.445	-0.345	-0.415	-0.435	
Standard	В	w/c	0.159	0.195	0.173	0.176	0.151	0.185	
Error	A1	f_c	0.079	0.087	0.076	0.082	0.075	0.089	
	A2	w/c'	0.108	0.111	0.115	0.116	0.113	0.118	
	А3	r	0.109	0.118	0.111	0.116	0.114	0.121	
	A4	P_{Fur}	0.179	0.178	0.178	0.190	0.185	0.188	
	A5	W_{Control}	0.170	0.187	0.195	0.182	0.174	0.190	
	A6	G_{Well}	0.128	0.143	0.140	0.136	0.141	0.146	
	A7	I _{DAL}	0.132	0.141	0.140	0.139	0.139	0.153	
P-Values	В	w/c	5.90E-08	1.37E-07	1.41E-08	1.28E-07	2.69E-08	2.08E-07	
	A1	f _c '	5.88E-07	9.45E-07	3.62E-07	1.38E-06	4.80E-07	1.99E-06	
	A2	w/c'	3.84E-24	1.99E-22	1.57E-23	5.71E-23	1.01E-23	6.62E-21	
	А3	r	4.08E-11	2.48E-11	5.61E-12	1.46E-10	2.76E-10	2.35E-10	
	A4	P_{Fur}	5.78E-08	6.32E-08	3.98E-08	3.29E-07	5.95E-07	3.02E-07	
	A5	W_{Control}	6.12E-06	3.91E-07	1.36E-06	2.01E-06	1.41E-06	9.15E-06	
	A6	G_{Well}	9.61E-07	1.15E-07	1.27E-08	1.79E-06	5.62E-08	1.45E-06	
	Α7	I_{DAL}	0.0048	0.0026	0.0020	0.0151	0.0036	0.0054	

COEF - Coefficient

Table 7.8: RMSE and R² values for the aggregate model and validation data RMSE

DADAMETED	RESULTS						
PARAMETER	Iteration 1	Iteration 2	Iteration 3	Iteration 4	Iteration 5	Iteration 6	
Model Building (random 85% of the data, 102 observations)							
RMSE	4.18	4.30	4.11	4.37	4.27	4.39	
R^2	0.835	0.847	0.854	0.831	0.835	0.829	
R ² adjusted	0.823	0.836	0.844	0.818	0.822	0.816	
Model Validation (remaining 15% of the data, 18 observations)							
RMSE	4.64	3.96	4.92	3.47	4.27	3.30	

Coefficient estimates close to 1 were attained for B. As such, it can be deduced that the w/c term on the bottom line of Equation 7.17.1 will have a lesser effect on the overall result. The coefficients attached to the control strength, control w/c and ash replacement level terms are slightly lower than the equivalent values for the cement model, though all three factors still retain a key effect on the strength estimate. In order from the largest to the smallest effect, the remaining four categorical variables ranked as follows: P_{Fur}, W_{Control}, G_{Well}, I_{DAL}.

The standard errors of the coefficients were lower than those of the equivalent cement model value. All values were below 0.2 and remained stable across the six iterations. Additional p-values in Table 7.7 verify that each parameter in the equation had a significant effect, as all values were within the 5% significance level.

The adjusted R² values from 0.82 to 0.84 in Table 7.8 show that good correlation between the actual and the predicted strength has been achieved. Associated RMSEs varied from 4.1 to 4.4, which is lower than the equivalent cement results. Combined with the fact that the R² correlation was marginally higher for the cement model, it can be deduced that the overall variability in the aggregate data is lower. This is somewhat surprising as it was initially expected that the higher replacement levels when using the ash as an aggregate component, compared with as a cement replacement, would create greater changeability.

For the validation stage, RMSE values calculated using the remaining 15% of the data were deemed to be at a level similar to the main model results. The RMSE range for the validation data was marginally wider, varying from 3.3 to 4.9, though the lower sample size was an obvious contributing factor to this. The stability present in the model building RMSE and R² values, across the six iterations, also supports the validity of the model.

Using the same reasoning as for the cement model, the coefficients for the final aggregate model were selected to balance the RMSE for the model building and validation data. As such, coefficients from iteration 5 were chosen, which yielded the following final model:

$$f_c = \frac{A}{0.91^{w/c}}$$
 (Eq. 7.18.1) where,
$$A = 0.41(f_c'^{\ 0.8}) \times 1.53^{\frac{w/c'}{w/c}} \times (1 - 0.81.\ln(r)) \times (1 + [0.99.P_{Fur}.r] + [0.90.W_{Control}.r]$$
 (Eq. 7.18.2)
$$+ [0.83.G_{Well}.r] - [0.42.I_{DAL}.r])$$

Diagnostics

Leverage, Cook's distances and standardised residual measures were again used throughout the model building and fine-tuning process. The results for these parameters, for the final aggregate model, are given in Figures 7.6, 7.7 and 7.8, respectively.

The three figures were collectively used to assess the validity of the model and identify potential outliers. The same guideline limits used in the cement model diagnostics were adopted to identify high leverage points ($>2 \times L_{sum}/n$), high Cook's distances ($>3 \times$ mean Cook's distance) and outlying standardised residuals (less than -2, greater than +2). It is apparent that there are fewer high variance points in the aggregate model data, with just three points falling outside the -2 to +2 range for standard residuals and all Cook's distance values were below 0.11. Nonetheless, these points with high Cook's distance and high standardised residual values were brought together in Table 7.9 for further analysis.

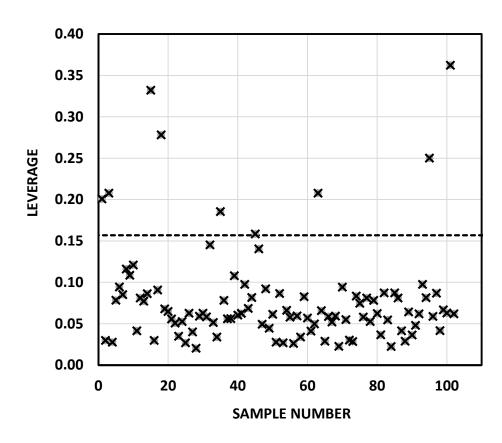


Figure 7.6: Leverage values for the aggregate model building data

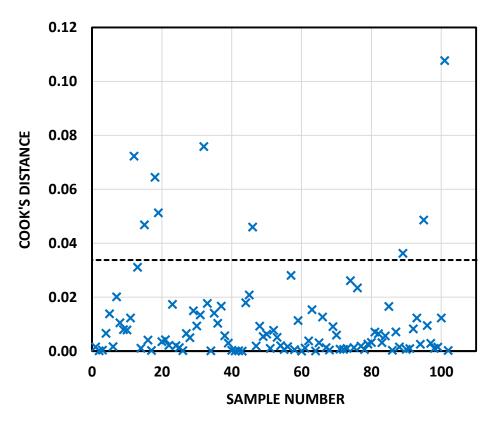


Figure 7.7: Cook's distance values for the aggregate model building data

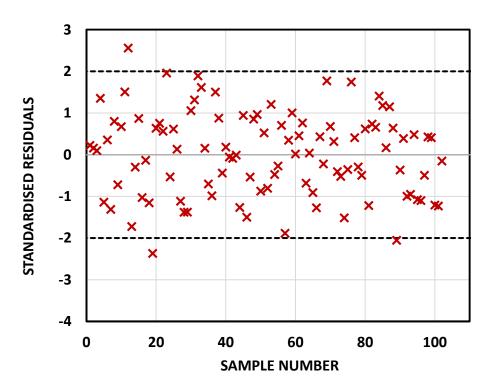


Figure 7.8: Standardised residuals for the aggregate model building data

Table 7.9: Potential outlier data points in the ash-concrete aggregate model

SAMPLE NO.	ASH	r, %	w/c	f _c , MPa (ACTUAL)	f _c , MPa (ESTIMATED)	COOK'S DISTANCE	LEVERAGE	STANDARDISED RESIDUAL
12	SSA	25	0.5	33.8	23.4	0.072	0.081	2.56
15	MIBA	100	0.26	40.0	37.0	0.047	0.332	0.87
18	MIBA	50	0.22	48.0	52.2	0.064	0.278	-1.16
19	MIBA	30	0.5	18.6	28.4	0.051	0.068	-2.37
32	SSA	20	0.45	56.1	48.6	0.076	0.145	1.89
46	SSA	50	0.5	9.3	15.2	0.046	0.140	-1.50
89	MIBA	30	0.5	19.1	27.6	0.036	0.064	-2.05
95	MIBA	100	0.55	41.0	45.0	0.049	0.250	-1.08
101	MIBA	50	0.21	50.0	54.2	0.108	0.362	-1.23

Red – exceeds Cook's distance, leverage or standardised residual guideline thresholds.

It is found that none of the three data points that fell outside the -2 to +2 standardised residual range were classed as high leverage, and as such, their overall influence on the final model should be limited. Absolute values of the Cook's distance were also quite low, despite exceeding the 3×2 mean guideline limit. In any case, from examining the original mix series for these samples, there were no noticeable signs that indicated that these strength results were

invalid. As such, the aforementioned data samples were retained in the aggregate model dataset. The one exception was a mix series using MIBA as 10% and 40% replacements for sand (Pavlik et al., 2011, 2012). The strengths for this series were substantially different than the rest, yielding standardised residuals of almost 4. On closer inspection, the measured strength increases appeared inconsistent with simultaneous increases in the porosity and decreases in the density and elastic modulus of the mixes, and as such, these results were excluded during the model fine-tuning stage.

Additional plots of the raw residuals (Figure 7.9) and the measured vs estimated strengths for both the model building data [Figure 7.10 (a)] and validation data [Figure 7.10 (b)] were developed as the final part of the aggregate model diagnostics. The random distribution of the residuals suggests that the model is an appropriate fit for the data. Using the lines of zero error shown in Figure 7.10 (a) and (b) as references, it is evident that estimated strength fit well with the measured values, for both the model building and the validation data. It appears that there are fewer estimated points with high deviations from the measured value in the aggregate data compared to the cement data, though the former has perhaps a lower amount of samples that lie exactly on the line of zero error.

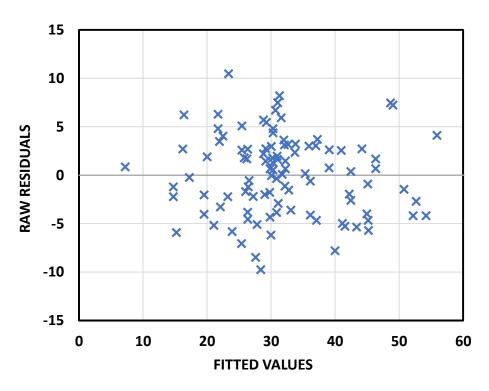


Figure 7.9: Raw residuals for the aggregate model building data

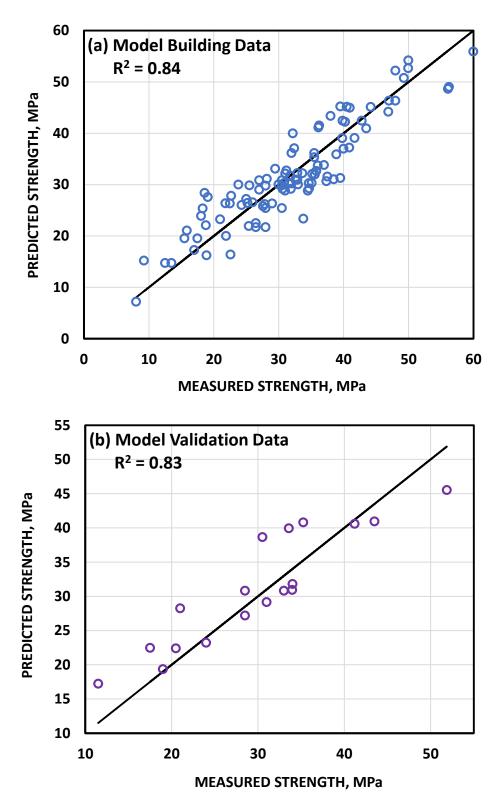


Figure 7.10: Predicted strength vs measured strength for aggregate model (a) building data and (b) validation data

7.7 Model Application

The fundamental concept of the power relationship between the w/c and the compressive strength, stemming from Abrams' law, is commonly used as part of the concrete mix design process. The models developed in this chapter offer the facility to account for incinerated ashes in the strength prediction, as both aggregate and cement (in ground form) components.

There has been substantial work undertaken using MIBA and SSA in concrete, taking samples from around the world and adopting various processing techniques and mix designs. However, up until this point there had been no real system available for estimating the strength performance in general with these ashes. Reductions in strength were probably expected, yet outside of the individual findings, the rate of this strength change and the full list of factors involved have not be established. The analysis and evaluation of the global data and the subsequent modelling work undertaken in this project have produced a method to quantify the effects of the key factors on strength, and how this varies with a diverse range of ash characteristics. This information can be used by the engineer to inform the selection of the parameters for the trial mix, based on the target strength.

Although there is an extensive list of models available to predict the strength of normal concrete mixes, the newness of this work is that the proposed models can account for the effects of incinerated ashes. The form of these models requires the control mix strength to be used as an input, from which the effects of the ash are positioned. When incorporating new materials, it usual for engineers to relate the results back to familiar benchmarks, such as the control Portland cement or natural aggregate performance. Indeed with coal fly ash, K_{eff} factors have been used to consider the material in terms equivalent to cement (Smith, 1967).

When applying the incinerated ash models, it is important to be aware of the boundaries of the data used in the development process. This information on the numerical input parameters is provided in Table 7.10. It is evident that the strength data range is broad and should encompass the vast majority of mixes for which the use of ashes could be considered. The w/c ratio range also covers most values adopted in practice. The ratio of the control mix

w/c to the ash mix w/c shows that the model can allow for reasonable adjustments in the w/c ratio, compared with the initial mix, though extreme variations from one end of the range to the other would be cautioned against. Only mixes using incinerated ashes were included in the model source data (though the control strength is incorporated as a separate input); as such, these models are not meant to be adopted for mixes with 0% cement/aggregate replacement levels. The 5%-60% and 5%-100% replacement levels for the cement and aggregate models, respectively, cover the full range of ash contents that are practically feasible. The wide SiO_2/CaO range confirms that a chemically diverse collection of global ash samples has been accounted for.

Table 7.10: Input parameter ranges for the incinerated ash models source data

INPUT PARAMETER	CEMENT MODEL DATA	AGGREGATE MODEL DATA
f _c , MPa	5-66	8-60
f _c ', MPa	26-65	20-63
w/c	0.36-0.77	0.21-0.86
w/c'	0.35-0.75	0.42-0.86
w/c' to w/c ratio	0.73-1.28	0.84-2.27
r, %	5-60	5-100
SiO₂/CaO, %	0.36-13.1	-

To demonstrate the use of the models for strength estimation, a trial run is presented using typical concrete mix parameters. The relationship between strength and mix design is generally well established by the concrete producers for normal mixes, though for this work, a w/c of 0.5 and control strength of 40 MPa were assumed. Ash properties shown in Table 7.11 were selected for this exercise. These properties were very favourable, though still considered as realistic targets in most cases. Further processing was not selected as this option may not be available at times. Replacement levels of 10%, 20% and 30% of cement, and 10%, 20%, 30% and 50% of aggregate, were chosen.

Table 7.11: Typical ash properties used for the strength design demonstration

PARAMETER	AS A CEMENT	AS AN AGGREGATE	
PANAIVIETEN	COMPONENT (ground)	COMPONENT	
d ₅₀ , mm	0.02	-	
d _{max} , mm	0.06	-	
Fineness Classification	High	-	
d ₆₀ , mm	-	2.6	
d ₃₀ , mm	-	1.65	
d ₁₀ , mm	-	0.4	
C_{u}	-	6.5	
C_c	-	2.6	
Grading Classification	-	Well-graded	
SiO ₂	35.0	-	
CaO	17.5	-	
SiO ₂ /CaO	2	-	
Further Processing	No	No	
Water Controlled	Yes using SP	Yes using SP	
Density, kg/m³	-	2400	
Water Absorption, %	-	10	
LOI, %	-	4	
Inhibiting Factor	-	No	
CDalaatisiaa.	•		

SP = superplasticiser

Compressive strength results determined for the ash mix w/c ratios are shown in Figure 7.11 (a) for the cement model and 7.11 (b) for the aggregate model. Power trend lines were fit to the data for each replacement level.

The estimated ash mix results exhibited strong performance relative to the control, with these favourable parameters selected. Higher strengths were estimated for the lower replacement levels, as the positive effects of the high fineness/well graded classification and the water control outweighed the reduction associated with increasing replacement level, though this behaviour soon reversed as the replacement level increased further. The w/c curves for the aggregate model are flatter than the counterpart cement model, which is attributed principally to the lower B coefficient in Equation 7.18.1 compared to Equation 7.16.1.

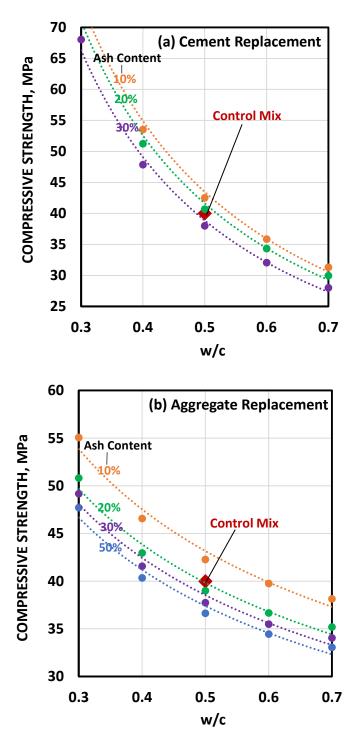


Figure 7.11: Compressive strength vs w/c for concrete using incinerated ash as (a) cement and (b) aggregate replacements.

The strength curves in Figure 7.11 represent just a sample use of the ash models. Researchers can use the developed equations to produce their own curves to fit their mix conditions and

ash properties. It is interesting to consider the estimated ash strength performance under the most unfavourable conditions (low fineness/not well graded, no further processing, no water control and inhibiting factor incurred). For example, with the same 40 MPa, 0.5 w/c control mix, strengths of 32 and 27 MPa were estimated for 20% cement and aggregate replacements, respectively, at the same 0.5 w/c. Large reductions close to these levels have been have been seen in past ash-concrete mixes, though with a clearer understanding on evaluating the ash properties, this behaviour can be knowingly prevented.

These strength estimates can be useful heading into the design process and determining the right territory for the ash mixes, though the results are not meant to be considered a final solution. With concrete mix design in general, a degree of trial and error is involved in obtaining the target characteristic strength, and subsequent adjustments of the strength-w/c curves may be required. Nevertheless, the models can result in more focused initial trial testing. In addition, as the incinerated ash model relates back to the control concrete strength, these models can be readily used as part of the strength estimation in existing procedures, such as the BRE or Faury mix design methods.

This work can also serve to provide an informed view of important factors involved, particularly regarding the MIBA/SSA characteristics and potentially incinerated ashes in general, which can be useful to researchers in proactively evaluating and potentially modifying the quality of the local samples. However, it should be noted that this is the first work of its kind undertaken on concrete strength prediction with incinerated ashes and it is expected that some further maturation of these models may take place as the development continues with a wider range of incinerated ash types.

7.8 Summary

This chapter presents the modelling work undertaken for estimating the strength of concrete and mortar mixes using MIBA/SSA as cement and aggregate components. Extensive work has been undertaken on strength estimation with conventional Portland cement and natural

aggregate mixes; however, this project contributes the first comprehensive framework to account for the effects of incinerated ashes on this key aspect of performance.

With the goal of creating models that are practically usable, are straightforward to operate, rely only on widely available parameters and retain familiarity with established practices, a multiple regression analysis approach using the structure of Abrams' law as the foundation was adopted. The diverse collection of global compressive strength data (specifically at 28-days) for concrete and mortar mixes using MIBA/SSA, including associated data on the mix proportions and ash characteristics, formed the basis of these cement and aggregate models.

As the factors influencing strength emerged through the model building and fine-tuning process, the cement and aggregate models were found to share commonalities regarding the key parameters on control strength, w/c and ash replacement level. Ash processing, specifically further washing, chemical or thermal treatments, also surfaced as a key parameter in both models, whilst water control, potentially involving the use of a water-reducing admixture, also delivered greater strength by negating the interference of the ash with moisture movement and the hydration process. Specifically in the cement model, the fineness (classified by D₅₀, D_{MAX} or Blaine fineness) and chemical composition (represented by SiO₂/CaO) of the ash, both of which are connected to its reactivity, were accounted for. In the aggregate model, the ash grading was categorised based on its D₆₀, D₃₀ and D₁₀ particle sizes, and inhibiting effects associated with high absorption, low density and high LOI values were also taken into account.

With the approach of using 85% of the data for model building and 15% for validation, repeated for six iterations, the cement and aggregate models yielded adjusted R² values from 0.84 to 0.86 and 0.82 to 0.84, respectively. Root mean square errors of approximately 5 and 4 MPa were also determined for the two respective models. These values for the model building data were comparable to corresponding results for the validation data, which, combined with the stability evident in the RMSE and R² values across the six iterations, supported the validity of the models. For the final models, regression coefficients were selected from one iteration

to best match the RMSE for the model building and validation data, whilst concurrently maximising the adjusted R² value.

Measures of leverage, Cook's distance, standardised residuals and raw residuals were used throughout the fine-tuning of the model to evaluate its validity and identify outliers. Regarding the outliers, data points were removed only when there was good reason to believe that the results were invalid after assessment of the source mixes and not simply because they exceeded a particular guideline outlier limit. Further plots showing good correlation between predicted and measured strength values for the model building and validation data suggested that the cement and aggregate models were both appropriate fits for their respective data.

In applying the models, it was demonstrated that when assuming favourable, though still realistic, target ash properties, good performance was predicted relative to the control mix, when using the incinerated ashes at lower replacement levels. Strength estimates obtained from the models can be utilised to determine the right territory for the ash mixes, heading into the mix design process, which allows for much more focused initial trial testing to be undertaken. The proposed models offer an informed view of how the incinerated ash properties influence the concrete strength, which can be used to proactively evaluate the suitability of local ashes and avoid unfavourable conditions that lead to large strength losses. The format of the models is also such that they can be readily used alongside the strength estimation part of existing mix design procedures, such as the BRE method.

CHAPTER 8

CONCLUSIONS AND RECOMMENDATIONS

8.1 Concluding Remarks

In addition to specific summary given in each chapter, this section presents the overall findings emerging from this research on the characteristics of incinerated ashes and their use in concrete-related applications.

It has been found that MIBA and SSA offer value as aggregate and cementitious components, though certain conditions, as set out in the previous chapters, are required for their effective utilisation. Results comparable to those of coal fly ash can be achieved with MIBA/SSA for many aspects of concrete performance, though the incinerated ashes present additional challenges, most notably regarding their effects on the water demand. Nevertheless, coal fly ash is a fitting example of a secondary material that overcame initial obstacles before becoming an acceptable cementitious material for use in concrete.

Much of the work with MIBA and SSA in concrete-related applications has been exploratory, using them as direct replacements of natural aggregate or of Portland cement, when ground. This has been useful in establishing a comparison between MIBA/SSA and recognised benchmarks, though without certain conditions on their use and an awareness of specific inhibiting traits that can arise with the ashes, their effectiveness as concrete materials can drop off. Expansion caused by the reaction of the metallic aluminium in MIBA, under the alkaline conditions of cement, was the most damaging behaviour that arose. This expansion appeared in less than half of the products that used MIBA, though when necessary, implementing special thermal treatment plus slow-speed grinding, or wet grinding techniques on the ash beforehand removed this issue.

Control of the water demand of both MIBA and SSA is another factor to account for in the mix design, which may require the use of a water-reducing admixture. Attributed to the ashes' porosity and particle morphology, increased water demand can potentially compromise the workability and interfere with the hydration reactions of the concrete. The extra demand is

greatest when using the ashes as an aggregate, though it lessens as a result of grinding the ash so that it can be used as a cement component. Conversely, as a lightweight aggregate, the smoothing effect on the ash particles caused by the pelletisation and sintering treatments results in improved workability when used as a natural aggregate replacement in concrete.

Given the worldwide variability, a proportion of the MIBA/SSA samples exhibited a particularly low density, high water absorption or high organics content. By avoiding these unfavourable traits, whilst optimising the grading of the ashes as an aggregate, or their fineness as a cement component, a strong strength performance close to that of the control can be achieved with MIBA/SSA in concrete. Products with MIBA/SSA were also comparable to the control, at an equivalent strength, with regard to shrinkage, permeation properties and durability. As a cement component, despite overall higher porosity at times with the ash mixes, the pore refinement associated with the pozzolanic activity can result in improvements of these parameters relative to natural aggregate mixes.

Concrete blocks offer an accessible outlet for the use of incinerated ashes. As an aggregate component, in particular, a series of successful case studies has been undertaken using MIBA and SSA in load-bearing, non-load-bearing, paving and interlocking blocks.

It has been established that MIBA and SSA are more suited to use as a lower content aggregate or cement component, though there are exceptions in lightweight applications. Elastic modulus results for ash mixes, at a given strength, remained similar to the Eurocode 2 natural aggregate curves at lower replacement levels, though these drop off at higher contents, owing to the lower ash stiffness. Similar to the pozzolanic coal fly ash, an upper content limit is needed with MIBA/SSA as a cement component to avoid low early-age strength, excessive setting times and interference with the hydration process.

As part of the raw feed for cement clinker production, the ashes contribute to the required quotas of SiO_2 , CaO, Fe_2O_3 and Al_2O_3 and deliver performance on par with the control cement clinker. It is recommended that the incinerated ash content be limited to 5% to avoid the build

up of minor components such as P₂O₅, SO₃, organic matter and metallics that can suppress C₃S formation and thus inhibit the cement setting behaviour and strength.

Lightweight aggregates produced from the pelletisation and sintering of MIBA/SSA achieved the target light weight and exhibited water absorption comparable to that of commercial lightweight aggregates. Strengths at the lower end of the range expected for Lytag were produced, whilst thermal conductivity requirements for insulating concrete were achievable with the ash lightweight aggregate. Concrete mixes incorporating these aggregates performed comparably to the corresponding mixes with Lytag.

Aerated concrete offers an opportunity for the use of MIBA/SSA as a bulk material. The ashes' porosity is favourable for the light weight and thermal conductivity requirements. MIBA, in particular, is ideally suited as its metallic aluminium expansion contributes to the desired low density, whilst its pozzolanic activity adds somewhat to the strength. As a replacement for cement or fly ash, the ash can also perform the role of the aerating agent, thus saving the need for expensive aluminium powder addition. Complete substitution of fly ash for incinerated ash results in a large discrepancy in the mix water demand, as was evident in commercial pilot-scale trials. Thus, it is recommended that up to approximately half of the fly ash be replaced, as MIBA-coal fly ash combination mixes were found to exhibit strength-to-density ratios superior to those of the control coal fly ash-aluminium powder mixes.

The related use of the ashes in foamed concrete has also shown promise. With SSA as a complete fine aggregate replacement, foamed concrete exhibited improved insulating properties, and strength requirements were met. A reduction in workability was evident, though at a significantly lower rate compared with the normal weight concrete and thus, the high flowability and self-compacting traits were retained.

Regarding the leaching of concrete-related products containing MIBA/SSA, heavy metals were effectively immobilised within the mix. The cement creates a solid form to encapsulate the trace elements and minimise moisture infiltration, whilst the chemical stabilisation effect also

reduces the mobility of these elements. The use of MIBA/SSA in concrete is more favourable, in terms of potential leaching, compared with an unbound road pavement layer; however, similar precautions should be adopted regarding beneficial ash treatments such as ageing and avoiding strongly unfavourable leaching conditions.

8.2 Recommendations for Further Work

Over the course of this study, several areas for potential further innovative work emerged. Gaps in the knowledge of the use of MIBA/SSA in concrete have also been identified. The following recommendations for further work are proposed:

(1) Metallic Aluminium Expansion with MIBA

The process by which the metallic aluminium in MIBA, under alkaline conditions of cement, forms aluminium hydroxide and hydrogen gas, leading to expansion, has been established. However, it is not fully understood why this behaviour arises with only some MIBA samples. Given that a wet grinding treatment is effective in dissipating the ash expansive reactions before its use in concrete, the nature of the post-incineration processing, specifically the quenching step, may have inadvertently led to the earlier dissipation of these reactions. Alternatively, the different behaviour may be attributable to variations in the composition of the MSW, influenced by economic status, cultural issues and waste management policies. The development of a procedure to distinguish beforehand between ash samples that will or will not cause expansion would be very useful.

(2) Aerated Concrete

Aerated concrete offers the potential for the quick uptake of MIBA in practice, because of its ideal suitability. It is recommended for the ash to be used as a partial coal fly ash replacement, which also reduces the required dosage of aluminium powder aerating agent.

(3) Expansion of the Modelling Work

As an addition to this study, it would be interesting to compare the strength of concrete mixes using MIBA/SSA to the model estimated results, when the ash characteristics are specifically

tailored to optimise performance according to the model parameters. The theory behind each of the parameters should remain valid; for example, high-fineness samples will produce higher strengths than equivalent low-fineness samples; however, minor adjustments to the classification limits may be desirable, for example, those distinguishing between high- and low-fineness ashes.

(4) Concrete with Composite Cement

The use of MIBA and SSA in a composite cement in concrete is a promising area for exploration. An approach could be adopted to maximise the content of SSA/MIBA, in combination with coal fly ash and GGBS, and develop optimal mix compositions for a range of non-structural and structural concrete applications. This combination would offset the increased water demands of the incinerated ashes. Early-age strength, setting behaviour and carbonation-induced corrosion, in particular, would also need to be addressed.

(5) <u>Life Cycle Assessment</u>

A life cycle assessment comparing the environmental and economic costs of using MIBA/SSA in various concrete applications with those arising from standard concrete materials and landfilling of the ashes would be very useful. This could also assess the viability of various ash processing options such as grinding, chemical and thermal treatments and the use of superplasticiser and nanomaterials.

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