

**Fat reduction in chocolate: a multidisciplinary approach considering
emulsion science and consumer expectations**

by

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A thesis submitted to

The University of Birmingham

for the degree of

DOCTOR OF PHILOSOPHY

School of Chemical Engineering

The University of Birmingham

September 2011

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BIRMINGHAM

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Abstract

Chocolate is consumed in large quantities, but is high in fat and calories, and has limited nutritional benefits. Producing reduced-fat chocolate offers a way of reducing energy consumed, if the consumer will accept the product. The aims of the research presented in this thesis were: i) to investigate consumer response to reduced-fat chocolate; ii) to investigate formulation routes for producing a reduced fat chocolate.

Focus group data indicated ambivalence towards chocolate, with some negativity towards the concept of a reduced-fat indulgent product. By manipulating label information it was shown that whilst consumers expected to like chocolate labelled 'reduced-fat' less than the standard chocolate, ratings of actual liking and of sensory attributes were similar. Thus, personal experience plays a greater role than expectations. This suggests that if the sensory characteristics of a reduced-fat chocolate can be matched to a standard chocolate, actual liking should not be affected. Packaging concepts indicated how different components of the package affect liking.

Water in oil cocoa butter emulsions were produced using a high shear mixer and a margarine line. Formulation changes (emulsifier type and concentration, and gelatin concentration) and processing parameters (shaft speeds and temperatures, and flow rate) were considered. All emulsions had small droplets (typically 1-5 μ m) and little 'free water'. When produced on the margarine line fat crystals in polymorphic form V were produced. Crystalline shells were also observed at the droplet interface. Pilot plant experiments resulted in comparable emulsions, with small droplets and fat crystals in form V. Although a full chocolate was not produced, the potential for

margarine technology to produce a reduced-fat chocolate was demonstrated.

For Mum and Dad

Acknowledgement

I would like to thank the EPSRC for funding this PhD project, and my supervisors Professor Peter Fryer, Dr Phil Cox (both of University of Birmingham) and Dr John Parkinson (Bangor University) for their help, guidance and support during my PhD. I would also like to thank Dr Joanne Hort for her help and assistance during the completion of the work presented in Chapter 5. I would like to thank the company that I visited to conduct scale up work, for allowing me to use their facilities, and for their hospitality during my visit. I would like to thank Lynn Draper and the other staff in the Chemical Engineering Office for all their help during my PhD. I would also like to thank my fellow students and colleagues for making my PhD a lot of fun. Finally, I would like to thank my family and my friends for all their support, particularly my parents for their encouragement and motivation during the writing of this thesis.

Nomenclature

Acronyms

ANOVA – Analysis of Variance

BMI – Body Mass Index

BMR – Basal Metabolic Rate

DEBQ – The Dutch Eating Behaviour Questionnaire

DSC – Differential Scanning Calorimetry

EE – Energy Expenditure

EI – Energy Intake

ES – Energy Stores

H - Hypothesis

HLB – Hydrophile-Lipophile Balance

JAR – Just About Right

NMR – Nuclear Magnetic Resonance

O/W – oil in water emulsion

PGPR – Polyglycerol polyricinoleate

SEM – Scanning Electron Microscopy

TAGs - triacylglycerols / triglycerides

VAS – Visual Analogue Scale

W/O – water in oil emulsion

WTP – Willingness to pay

XRD – X-ray Diffraction

Symbols

γ , α , β' , β - Triglyceride polymorphs

I, II, III, IV, V, VI – Chocolate industry method for classifying chocolate polymorphs

λ - Wavelength of an x-ray (Å)

θ - Diffraction angle of an x-ray (°)

W, M, S, V – used to characterise strength of x-ray peaks

d – distance (m)

$d_{3.2}$ – surface-weighted mean droplet diameter (μm)

$d_{3.3}$ – volume weighted mean droplet diameter (μm)

T – Temperature ($^{\circ}\text{C}$)

ϕ - dispersed phase volume fraction

p – probability value

F – F-ratio (ANOVA)

t – t value (t-test)

σ – Standard Deviation

\bar{x} - Mean

Contents

1. Introduction and Literature Review	1
Context of study.....	1
Objectives	3
Layout of the Study	3
Literature Review	4
1.4.1 Obesity.....	5
1.4.2 Expectations	9
1.4.3 Labelling.....	14
1.4.4 Individual Differences	21
1.4.5 Chocolate.....	23
1.4.5.1 Cocoa Butter.....	26
1.4.5.2 The Importance of Fat in Chocolate.....	29
1.4.5.3 Differential Scanning Calorimetry (DSC).....	30
1.4.5.4 X-ray Diffraction (XRD)	32
1.4.6 Methods for Calorie Reduction in Chocolate	34
1.4.7 Emulsions	39
1.4.7.1 Emulsification.....	40
1.4.7.2 Emulsion Stability.....	41
1.4.7.3 Emulsifier use.....	44
1.4.7.3.1 Lecithin	46
1.4.7.3.2 Polyglycerol polyricinoleate.....	47
1.4.7.5 Hydrocolloids / gelatin.....	50
1.4.7.6. Analytical Methods.....	51
1.4.7.6.1 Nuclear Magnetic Resonance (NMR)	51
1.4.7.6.2 Cryogenic Scanning Electron Microscopy (cryo-SEM).....	53
1.4.8 Summary of Literature Review	55
2. Exploring consumers beliefs and expectations: Materials and Methods	57
2.1 Introduction.....	57
2.2 General Methods.....	57
2.3 Chocolate Consumption Focus Group.....	58
2.3.1 Participants	58
2.3.2 Apparatus.....	58
2.3.3 Procedure	59
2.3.4 Data Analysis.....	61
2.4 Health and Indulgence Focus Group	61
2.4.1 Participants.....	61

2.4.2 Apparatus.....	62
2.4.3 Procedure	62
2.4.4 Data Analysis.....	63
2.5 Labelling Study.....	63
2.5.1 Design.....	63
2.5.2 Participants.....	63
2.5.3 Apparatus.....	64
2.5.4 Procedure	65
2.5.5 Data Analysis.....	70
2.6 Packaging Study	72
2.6.1 Part A: Focus Groups.....	72
2.6.1.1 Participants	72
2.6.1.2 Procedure.....	73
2.6.1.3 Data Analysis	74
2.6.2. Part B: Rank – Rating.....	74
2.6.2.1 Participants	74
2.6.2.2 Materials and Methods.....	74
2.6.2.3 Data Analysis	75
3. Understanding consumers’ beliefs about chocolate, and the concepts of health and indulgence.....	76
3.1 Introduction.....	76
3.2 Experimental Design	78
3.3 Results.....	79
3.3.1 Chocolate Consumption.....	79
3.3.2 Health and Indulgence	86
3.4 Overall Conclusions.....	92
4. Using labelling to explore consumer expectations of reduced-fat chocolate.....	95
4.1 Introduction.....	95
4.2 Hypotheses.....	97
4.3 Results.....	98
4.3.1 Carryover Effects.....	98
4.3.2 Expected and Actual Liking.....	99
4.3.3 Sensory Attributes	103
4.3.4 Individual Differences	104
4.3.5 Expected consumption amount and individual differences	108
4.3.6 Willingness to pay and individual differences.....	113
4.4 Discussion.....	117
4.4.1 Liking.....	118

4.4.2 Sensory attributes.....	120
4.4.3 Consumption Amount	120
4.4.4 Willingness to pay.....	122
4.4.5 Additional Individual Differences	123
4.5 Conclusions and Future Work	123
5. Exploring packaging of reduced-fat chocolate.....	125
5.1 Introduction.....	125
5.2 Focus Groups	127
5.2.1 Results.....	127
5.2.1.1 Female Focus Group	127
5.2.1.2 Male Focus Group	130
5.2.2 Overall Conclusions.....	132
5.3 Rank – Rating Task	132
5.3.1 Packaging Concepts.....	132
5.3.1.1 Colour.....	133
5.3.1.2 Size / shape	133
5.3.1.3 Slogan.....	133
5.3.1.4 Logo	134
5.3.2 Hypotheses.....	135
5.3.3 Results.....	136
5.3.4 Conclusions.....	140
5.4 Discussion.....	141
5.5 Conclusions.....	144
6. Creation of cocoa butter emulsions and reduced fat chocolates: Materials and Methods	146
6.1 Introduction.....	146
6.2 Materials	146
6.3 Preparation of emulsions	147
6.3.1 High Shear Mixer	147
6.3.2 Margarine line.....	148
6.3.3 Schröder Kombinator.....	152
6.4 Analytical Techniques	153
6.4.1 Nuclear Magnetic Resonance (NMR)	153
6.4.2 Differential scanning calorimetry (DSC)	153
6.4.3 Conductivity.....	156
6.4.4 Microscopy	156
6.4.5 Gravimetric Method	156
6.4.6 X-Ray Diffraction (XRD).....	157

7. Development of cocoa butter emulsions using two processing methods: a high shear mixer and a bench-scale margarine line	158
7.1 Introduction.....	158
7.2 High Shear Mixer	159
7.2.1 Emulsifier Type.....	160
7.2.3 Processing Parameters	167
7.2.4 Storage Time.....	169
7.2.5 Conclusions	174
7.3 Margarine Line	175
7.3.1 Conclusions	181
7.4 Overall Conclusions.....	181
8. Effect of changes in formulation and processing parameters on melting profile and water droplet size.....	184
8.1 Introduction.....	184
8.2 Formulation Changes.....	186
8.2.1 Percentage of PGPR.....	186
8.2.2 Gelatin Concentration.....	188
8.3 Processing Parameters	193
8.3.1 A & C Unit Rotation Speeds	193
8.3.2 A & C Unit Temperatures	196
8.3.3 Throughput.....	200
8.4 Conclusions.....	201
9. Scaling up the emulsification process to pilot plant scale, and investigating the addition of dry chocolate ingredients	205
9.1 Introduction.....	205
9.2 Emulsions	208
9.3 Chocolate	225
9.4 Discussion.....	226
10. Conclusions and Future Work.....	229

List of Figures

Fig. 1.1 Schematic model of the effects of expectations on product selection and evaluation.....	12
Fig. 1.2 Chocolate Processing	27
Fig. 1.3 A diffracted beam from a crystal lattice, indicating the elements of Braggs Law.....	33
Fig. 1.4 An illustration of a polydispersed water in oil emulsion	44
Fig. 1.5 An illustration of water in oil emulsion stabilised by solid particles.....	48
Fig. 1.6 The set up of the Bruker minispec NMR	52
Fig. 1.7 Schematic representation of the pulse sequence used in stimulated echo sequence.....	53
Fig. 1.8 Construction of cryo-SEM.....	54
Fig. 4.1. Rated scores for expected and actual liking of chocolate samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’	100
Fig. 4.2. Distribution of JAR scores for each attribute	104
Fig. 4.3. Ratings of expected liking of chocolate samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’ for those participants who said they were likely or unlikely to try low-fat foods.....	105
Fig. 4.4. Rated scores for expected liking of chocolate samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’, for participants with below median, and above median levels of external eating	107
Fig. 4.5. Ratings of expected normal consumption amount (in squares of chocolate) of samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’ for participants below, and above, the median ambivalence.....	109
Fig. 4.6. Ratings of expected unrestricted consumption amount of samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’ for participants below, and above, the median BMI.....	110
Fig. 4.7. Ratings of expected unrestricted consumption amount of samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’ for participants who are likely, and unlikely to try low-fat foods.....	112
Fig. 4.8. Ratings of price of samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’ for participants below, and above, the median BMI	114

Fig. 4.9. Ratings of price for chocolate samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’, for frequent and occasional consumers of chocolate	115
Fig. 4.10. Ratings of price of samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’ for participants below, and above, the median restriction.....	117
Fig. 5.1 Two of the bars used for the rank-rating task	135
Fig. 5.2 Average liking scores for each of the packaging concepts (for all participants)	136
Fig. 5.3 Calculated part worths for each of the components.....	139
Fig. 6.1 A schematic of a. the Silverson high shear mixer, and b. the emulsion screen used.....	148
Fig. 6.2 A schematic of a. the scraped surface heat exchanger (A unit), and b. the pin stirrer (C unit).....	149
Fig. 6.3 A schematic of the margarine line	150
Fig. 6.4 DSC data indicating melting profile of the same emulsion (containing 30% water and 1% PGPR), heated from 5°C to 50°C at a rate of 0.5, 1, or 2°C/min	155
Fig. 7.1. Percentage of water in small droplets (<100µm), determined by NMR analysis, for samples containing soya lecithin and PGPR with different percentages of water	163
Fig. 7.2. Scanning electron micrographs of cocoa butter emulsions made with soya bean lecithin emulsified using a Silverson high shear mixer.....	164
Fig. 7.3 SEM images of cocoa butter emulsions containing 20% water, 1% sugar and 1% PGPR that were processed using a Silverson high shear mixer	166
Fig. 7.4 DSC curves indicating melting temperatures of cocoa butter emulsions emulsified using a high shear mixer. The fat phase of the emulsions was heated to different temperatures (40, 55 and 65°C) prior to emulsification	168
Fig. 7.5 DSC curves indicating the melting temperatures emulsions containing a. 10% aqueous phase, b. 30% aqueous phase, and c. 50% aqueous phase.....	171
Fig. 7.6 Average temperature peak of cocoa butter emulsions (containing 10, 20 and 30% water) emulsified using a high shear mixer, at varying time intervals (from 1 day to 24 weeks).....	174
Fig. 8.1 DSC curve indicating the melting profile of solid gelatin solutions, containing 6, 8 and 10% gelatin	185
Fig. 8.2 Micrographs of cocoa butter emulsions.....	192

Fig. 8.3 Mean droplet size (μm) as determined by NMR analysis, as a function of varying both the A and the C Unit rotations speeds (rpm).....	194
Fig. 8.4 DSC curves for emulsions processed under different conditions	195
Fig. 8.5 T_{peak} ($^{\circ}\text{C}$) as a function of A unit outlet temperature and C unit outlet temperature	197
Fig. 8.6 T_{peak} ($^{\circ}\text{C}$) as a function of the difference in temperature ($^{\circ}\text{C}$) between the process inlet temperature and the A unit outlet temperature	198
Fig. 8.7 Droplet size ($d_{3,2}$) as a function of A unit outlet temperature and C unit outlet temperature ..	199
Fig. 8.8 Droplet size as a function of pump speed	201
Fig. 9.1 Mean droplet size ($d_{3,2}$) as a function of storage time for emulsions containing 20% aqueous phase, 40% aqueous phase, and 20% aqueous phase with milk fat in addition to cocoa butter, stored in both ambient and chilled conditions.....	210
Fig. 9.2 Mean percentage of free water as a function of storage time for emulsions containing 20% aqueous phase, 40% aqueous phase, and 20% aqueous phase with milk fat in addition to cocoa butter, stored in both ambient and chilled conditions.....	211
Fig. 9.3 DSC curves indicating the melting profile of emulsions a. containing 20% aqueous phase, b. containing 40% aqueous phase, and c. containing 20% aqueous, with milk fat in addition to cocoa butter.....	216
Fig. 9.5 Mean T_{peak} ($^{\circ}\text{C}$) as a function of storage time for emulsions containing 20% aqueous phase, 40% aqueous phase, and 20% aqueous phase with milk fat in addition to cocoa butter, stored in both ambient and chilled conditions.....	218
Fig. 9.6 Mean enthalpies (J/g) as a function of storage time for emulsions containing 20% aqueous phase, 40% aqueous phase, and 20% aqueous phase with milk fat in addition to cocoa butter, stored in both ambient and chilled conditions.....	219
Fig. 9.7 X-Ray Diffraction: a). an emulsion containing 20% aqueous phase, b) an emulsion containing 40% aqueous phase, c) an emulsion containing 20% aqueous phase, with milk fat in addition to cocoa butter.....	220
Fig. 9.8 Cryo-SEM images of cocoa butter emulsions produced during scale-up experiments	224
Fig. 9.9 A cryo-SEM image of an emulsion that has been mixed with the dry chocolate ingredients ..	226

List of Tables

Table 1.1 The World Health Organisation BMI classification system for adults.....	6
Table 1.2 The predicted effects of disconfirmed consumer expectations on the direction of increase/decrease in perceived product performance for the assimilation, contrast, generalised negativity and assimilation-contrast models	14
Table 1.3 Melting point (°C) of cocoa butter polymorphs from the literature	31
Table 1.4 Heat of fusion, and calculated melting enthalpy for cocoa butter polymorphs.....	31
Table 1.5 Short spacings X-ray data for cocoa butter polymorphisms.....	33
Table 4.1 Mean ratings of expected and actual liking of the chocolates labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’ tasted during session 1 and session 2	98
Table 5.1 Mean rating scores for each of the packages for female consumers	137
Table 5.2 Mean rating scores for each of the packages for male consumers.....	137
Table 5.3 Mean rating scores for each of the factors, for all consumers, females and males.....	138
Table 6.1 Melting enthalpies of replicates of the same cocoa butter emulsion heated at a rate of 0.5, 1, or 2°C/min	155
Table 7.1 NMR analysis for cocoa butter emulsions containing soya lecithin produced using a high shear mixer	161
Table 7.2 NMR analysis for cocoa butter emulsions containing PGPR produced using a high shear mixer.....	161
Table 7.3 Melting properties of three cocoa butter emulsions in which the fat phase was heated to 45, 55 or 65°C.....	169
Table 7.4 The temperature onset, peak and end and enthalpy (J/g) of cocoa butter emulsions over time	172
Table 7.5 Details of emulsion formulation and margarine line set up, along with measured exit temperatures	176
Table 7.6 NMR analysis for cocoa butter emulsions produced using the margarine line	177
Table 7.7 DSC analysis of cocoa butter emulsions made using the margarine line.....	178

Table 8.1 NMR results for cocoa butter emulsions containing different percentages of PGPR (from 0.1 to 5% within the lipid phase).....	187
Table 8.2 Results acquired from both NMR and DSC for cocoa butter emulsions with different percentages of water, and different percentages of gelatin	190
Table 8.5 <i>T_{peak}</i> and melting enthalpy of emulsions produced using different pump speeds	200
Table 9.1 Recipes used for each of the formulations used during the scale up experiments.....	206
Table 9.2 The ratio of dry mix to emulsion, and the percentage of different ingredients within the dry mix	207
Table 9.3 Processing parameters used for each of the emulsions produced during scale-up experiments	208
Table 9.4 Mean droplet size, sigma and free water content for samples heated to 40°C and held at this temperature for 1, 2, 3 and 5.5 hours.....	213
Table 9.5 Results of the gravimetric method, in which the samples were weighed, before being placed in a dry oven at 110°C for 8, 12 and 30 hours.....	214
Table 9.6 X-ray diffraction short spacings typical of cocoa butter polymorphisms IV, V and VI observed for emulsions, with the strength of peak	221

1. Introduction and Literature Review

Context of study

Chocolate manufacture is a big business for the UK food industry: Britons, on average, eat 10kg of chocolate a year, costing £72 per person (Barnett, 2006). Chocolate is notoriously high in fat (30 – 40%wt) and calories (typically around 500kcal/100g), but is consumed by a large proportion of the western population. For many it may be detrimental to maintaining a healthy weight. Furthermore, it is not eaten at meal times or as part of a staple diet, but is eaten as a snack or a treat. In a modern diet, where calories are cheap and plentiful, a food product that is high in fat and calories and provides only limited nutritional benefits is no longer beneficial. As such, the advent of a reduced-fat chocolate may provide a means of reducing unnecessary and unhealthy energy intake.

Obesity is increasing throughout the world, with rises in prevalence of the disease being seen in the UK. According to National Health Service reports, in 2006 23.1% of men and 24.8% of women were classified as obese, as compared with 13.2% and 16.4% respectively in 1993. The health risks of obesity include the development of non-insulin dependent diabetes mellitus, hypertension, hyperlipidaemia, cardiovascular disease, coronary heart disease, stroke, gallbladder disease, arthritis and certain types of cancer (NHS, 2006). The British Government are becoming increasingly concerned with the effects of obesity on the health and longevity of the nation (McPherson, Marsh & Brown, 2007); obesity presents a major public health problem, with increased healthcare costs.

The consumption of reduced fat products offers a way of reducing the fat and energy ingested, with beneficial effects for body weight and health. Increasing global obesity has fuelled an increase in the production of foods that are lower in fat, sugar and calories. Reducing dietary fat is particularly important as it is energy dense (carbohydrate = 3.75kcal/g, protein = 4kcal/g, alcohol = 7kcal/g, and fat = 9kcal/g; Food Standards Agency, 2002), but has only a limited effect on suppressing appetite, compared with carbohydrate (Egger & Swinburn, 1997). This can result in ‘passive consumption’, in which excess energy is ingested without a large quantity of food being consumed (Prentice & Jebb, 1995). Furthermore, having a high daily caloric intake from fat is related to an increased risk of cardiovascular disease, cancer, stroke and diabetes. Consequently, reducing dietary fat may reduce energy intake and help prevent obesity and related health problems.

Currently, although some diabetic chocolates (that are reduced in sugar) are available on the British market, low or reduced fat chocolates are currently not readily available. Producing a chocolate that is reduced in fat presents an engineering challenge: it is difficult to produce a product that is easy to manufacture, yet still performs when consumed.

Conflict between taste and health may be significant for some categories of foods. Consumers may believe that sensory quality must be sacrificed in order to eat healthily, trading enjoyment for health benefits (Tuorila & Cardello, 2002); it has been reported that consumers feel that there is a reduction in taste quality associated with reduced-fat diets (Lloyd, Paisley & Mela, 1995). It seems unwise to assume that consumers demand a chocolate that is lower in fat or calories. Not only may

consumers be unwilling to make trade-offs in terms of sensory characteristics, but they may not favour the concept of a reduced-fat chocolate, even if it is still highly palatable. Therefore, it is important to identify what consumers expect from reduced-fat chocolate (both in sensory and hedonic terms), whether they would consider it as equally indulgent, whether they would consume more (and thus ‘undo’ any benefits gained from it being reduced in fat), and whether different cohorts of consumers would respond differently to the product. Such questions are important for food manufacturers; if the consumer is not willing to accept a product, however healthy it may be, the product will be a failure.

Objectives

The aims of this work were twofold:

- To investigate chocolate consumption, beliefs about health and indulgence and consumer response to reduced-fat chocolate;
- To investigate formulation engineering routes for producing a reduced fat chocolate that has a similar flavour, texture and mouthfeel to a full fat equivalent.

Layout of the Study

This thesis is multidisciplinary, spanning both chemical engineering and consumer psychology. It aims to discover the possible routes for producing a reduced-fat chocolate, and the psychological issues surrounding the consumption of reduced-fat products. The thesis is presented in two halves for ease of reading, but both sections

complement one another. Thus, in the current chapter a review of the available literature is presented, covering all the areas studied during the project. Chapter 2 and 6 present the materials and methods used for both the consumer psychology studies, and the engineering experiments. Chapters 3, 4 and 5 present the results of the three psychology studies conducted. Chapter 3 presents the findings of focus groups conducted to explore chocolate consumption, and beliefs about health and indulgence. Chapter 4 details the results of a labelling experiment, in which the expectations of reduced-fat chocolate are studied. Chapter 5 explores current packaging of chocolate, and considers approaches to packaging a reduced-fat chocolate. The work presented in Chapter 5 has also been submitted as part of the research project for the Post Graduate Certificate in Sensory Science, at the University of Nottingham. Chapters 7, 8 and 9 describe the results of experiments on the engineering of a reduced-fat chocolate. Chapter 7 introduces the production of a cocoa butter emulsion for fat reduction in chocolate, using two processing methods. Chapter 8 considers the addition of gelatin to the aqueous phase of the emulsion, in addition to further formulation and processing changes. Chapter 9 presents the results of a scale-up trial, in which cocoa butter emulsions were produced at pilot-plant scale. Finally, Chapter 10 contains the major conclusions obtained from the results chapters, with ideas for possible future work.

Literature Review

The following literature review covers the important research in the areas relevant to this study i.e. both in the psychology underpinning the choice of low-fat foods, and in the manufacture and characterisation of chocolate. It begins with a discussion of

research in obesity, to give the reader a brief understanding of the disease, and the motivation for producing food products that are reduced in fat, or calories. The causes of obesity are complex, but it is clear that food plays a key role; thus, by modifying the food that is consumed it may be possible to reduce the prevalence of the disease. This is followed by a section addressing the psychological studies that have been conducted to consider the effect that expectations about food products, specifically with altered fat contents, health claims or novelty, have on hedonic and sensory perceptions, and on self regulated consumption.

Next, chocolate will be introduced, with a brief summary of its history, and the modern day production methods used. This is followed by a description of the fat (cocoa butter) within chocolate, and methods used to analyse the polymorphic form of the fat. Finally, possible methods for reducing the fat in chocolate will be presented, with emphasis on emulsion science, with a review of emulsification techniques, emulsion instability and stability (i.e. the use of emulsifiers, Pickering stabilisation, fat crystals at the interface, and the use of hydrocolloids) and analysis techniques.

1.4.1 Obesity

Ravussin and Swinburn (1992) state that obesity is a condition of excess body fat. Body Mass Index (BMI, Quetelet's Index) is the currently accepted, and most widely used, measure of obesity among adults, which is calculated using weight (kg) and height (m), in the following formula:

$$BMI = \frac{BodyWeight(kg)}{Height(m)^2} \quad (1.1)$$

Table 1.1 details the World Health Organisation (WHO) classifications of BMI, providing a scaling system from ‘underweight’ to ‘obese’.

Table 1.1 The World Health Organisation BMI classification system for adults (WHO, 2004).

Classification	BMI
Underweight	<18.5
Severe thinness	<16
Moderate thinness	16 – 16.99
Mild thinness	17 – 18.49
Normal	18.5 – 24.99
Overweight	≥25
Pre-obese	25 – 29.99
Obese	≥30
Obese class I	30-34.99
Obese class II	35-39.99
Obese class III	≥40

Obesity is thought to be a result of a chronic energy imbalance. Ravussin and Swinburn (1992) describe the classic energy balance equation:

$$ES = EI - EE \quad (1.2)$$

where, ES is change in energy stores, EI is energy intake, and EE is energy expenditure. During weight maintenance:

$$EI = EE \quad (1.3)$$

After a small increase in stored energy, there is a corresponding increase in energy expenditure that brings the body back into energy balance, i.e. heavier people expend more than lighter people in order to do the same task (Ravussin & Swinburn, 1992). Physiological adjustment, in terms of behavioural changes, also minimise fluctuations in body weight (Egger & Swinburn, 1997). However, if energy intake far exceeds

expenditure (i.e. a positive energy balance), the excess energy is stored as adipose tissue, which will eventually result in obesity. Only one factor, calorie consumption, contributes to energy intake, whilst a number of factors contribute to energy expenditure:

1. Basal metabolic rate (BMR): the energy that is expended to keep a resting, awake body alive. In sedentary adults, BMR accounts for 50-70% of energy expenditure.
2. Thermogenesis: the increase in metabolic rate in response to food intake, cold or psychological influences (fear or stress). The thermic effect of food accounts for approximately 10% of daily energy expenditure.
3. Voluntary activity: this is the most variable component of daily energy expenditure.

Obesity exhibits both genetic and familial associations. Ravussin and Swinburn (1992) suggest that approximately 70% of the variance in BMI is due to genetic influences, and approximately 30% to the non-shared environment that is unique to the individual. Research concerning the identification of specific genes or allelic variations that are associated with weight gain or obesity is advancing (Blundell *et al.*, 2005) although as yet a gene responsible for human obesity has not been identified. It is important to recognise that the rate at which obesity is increasing far exceeds the rate at which genetic changes occur, suggesting that environmental or behavioural factors must have more of a significant role in the development of obesity than genetic factors.

Neel (1962) was the first to suggest that natural selection resulted in “thrifty genes” that provided the evolutionary advantage of promoting metabolic efficiency and fat storage during times of plenty so there would be adipose reserves for times of food scarcity. According to the theory, during our evolutionary history people with greater adipose reserves would have had a selective advantage by their greater survival. However, in affluent societies where food is plentiful this adaptation would predispose people to gain weight and become obese. Modern environments have been described as “obesogenic” (Egger & Swinburn, 1997) i.e. they promote obesity through the availability of palatable foods that are cheap, persuasively advertised, and increasingly more convenient. The increased reward value and palatability of foods overrides satiety signals and leads to overeating (Rolls, 2007). Furthermore, the number of calories derived from dietary fat has dramatically increased, and physical exercise has decreased (Lichtenstein, 1999).

Blundell and Finlayson (2004) suggest that an exaggerated hedonic response (feelings of pleasure, reward, pleasantness, desire, preference, liking, wanting, or palatability) to the sensory attributes of a food could promote overconsumption. Blundell and Finlayson (2004) also show that the palatability of food has a positive effect on intake and meal size, suggesting that palatability is involved in satiation processes. It has been shown that by manipulating the flavour of a food, and thus increasing its palatability, intake increases (for a review see Yeomans, 2007). Liking is thought to drive ingestion, so that reward can override metabolic regulatory systems: people will eat more because of elevated sensations of pleasure during eating. Sensory cues

(sights, sounds and smells for example) may motivate a person to seek a certain food item.

Drewnowski and Greenwood (1983) investigated the perception and hedonics of sweet and fatty tastes using different combinations of milk/cream and sucrose. The results indicated a sweetness breakpoint, after which scores of pleasantness decline. However, hedonic preference ratings for fat continued to rise, and showed no clear breakpoint. Furthermore, the acceptance of the high fat dairy products was greatly enhanced by the addition of sucrose, and the hedonic responses to sweetness was modulated by the fat content.

It is clear that food consumption plays a major role in obesity. Not only are calories and fat plentiful in modern societies, but fat often increases a food's palatability, and humans are unable to regulate its intake (i.e. there is not an autoregulatory system to maintain a fat balance). Fat reduction in foods should effectively decrease the prevalence of obesity, providing that the hedonic value of the food is not compromised, and the consumers' expectations of the food remain high.

1.4.2 Expectations

Producing reduced fat chocolate may lead to altered expectations in consumers. In psychology, the construct of expectations was introduced by Tolman (1938), who used the concept of "expected consequences of behavior" as an explanatory variable to account for animal learning i.e. a behaviour occurs in response to expected pleasure. Meehl and MacCorquodale (1951) and MacCorquodale and Meehl (1953)

expanded the concept of expectancy theory to human learning. Helson (1948) introduced his 'adaption-level theory' which viewed all perceptual events as resulting from a comparison of the external stimulus to an internal 'adaption level' (a direct consequence of the individual's past experience with the stimuli). The view that stimuli in the environment are interpreted through comparison with internal cognitive states was central to Festinger's theory of cognitive dissonance. Festinger (1962) proposed that cognitions (beliefs, attitudes, values and perceptions) and behaviours must be psychologically consistent in order to avoid psychological dissonance (a state of psychological discomfort). He proposed that when cognitions or behaviours are in conflict, cognition dissonance arises, producing a negative drive state, which motivates the individual to change the cognition or the behaviour to make them more consistent. This led to the conclusion that pre-existing experiences, beliefs, and expectations about stimuli strongly determine perceptions, attitudes and behaviours towards it. If there is a mismatch between sensory information and expectations the perception of the stimuli may change, the cognition may change, or both may change.

Expectancy effects have been observed for tastants (i.e. substances that stimulate the sense of taste). Carlsmith and Aronson (1963) administered a series of iso-intense (of the same intensity) solutions of sucrose and quinine sulphate to participants who rated them for perceived intensity of sweetness or bitterness. Prior to presentation, the investigators created an expectation by stating whether the solution would be either sweet or bitter. The cue and the stimulus were either congruent or incongruent. Results showed that i) sucrose solutions that disconfirmed an expectancy were rated less sweet than sucrose solutions that confirmed an expectancy, and ii) quinine

solutions that disconfirmed an expectancy were rated as more bitter than quinine solutions that confirmed an expectancy. The authors suggested that this finding is consistent with cognitive dissonance theory i.e. disconfirmed expectations about both solutions resulted in negative affect towards them, lowering perceived sweetness and increasing perceived bitterness (both reflecting reduced pleasantness).

Visual cues have also been found to produce an expectancy effect. DuBose, Cardello and Maller (1980) found that not only did the colour of a cherry-flavoured beverage affect participants' ability to recognise its flavour, but the colour also altered perception, with participants believing the drink was lime-flavoured when it was coloured green. Zampini, Wantling, Phillips and Spence (2008) reported that accuracy in identifying a beverage's flavour (blackcurrant or orange) improved significantly when the beverage was coloured appropriately (grey or orange respectively). When Blackwell (1995) asked participants to describe the odour of six fruit solutions, four of which were inappropriately coloured, it was found that the identification of fruit odour was significantly more difficult when the colour of the solution was inappropriate. Morrot, Brochet and Dubourdieu (2001) investigated this phenomenon using white wine samples that had been coloured red, finding that participants generally used red wine descriptors to characterise the coloured white wines. These studies highlight the effect that expectations can have on the perception of taste, both in its identity and perceived intensity.

Expectations play an important role in food consumption as they may improve or degrade the perception of a product, even before it is tasted, with consumer satisfaction being strongly related to the degree of disparity between expectations and

actual product performance (Deliza & MacFie, 1996). A number of extrinsic cues are used by consumers to judge product quality, including brand name, brand familiarity, advertisements, product name, label, packaging, nutritional information and price (Deliza & MacFie, 1996). If the consumer has no direct prior experience of the product then information has more of an impact on expectations.

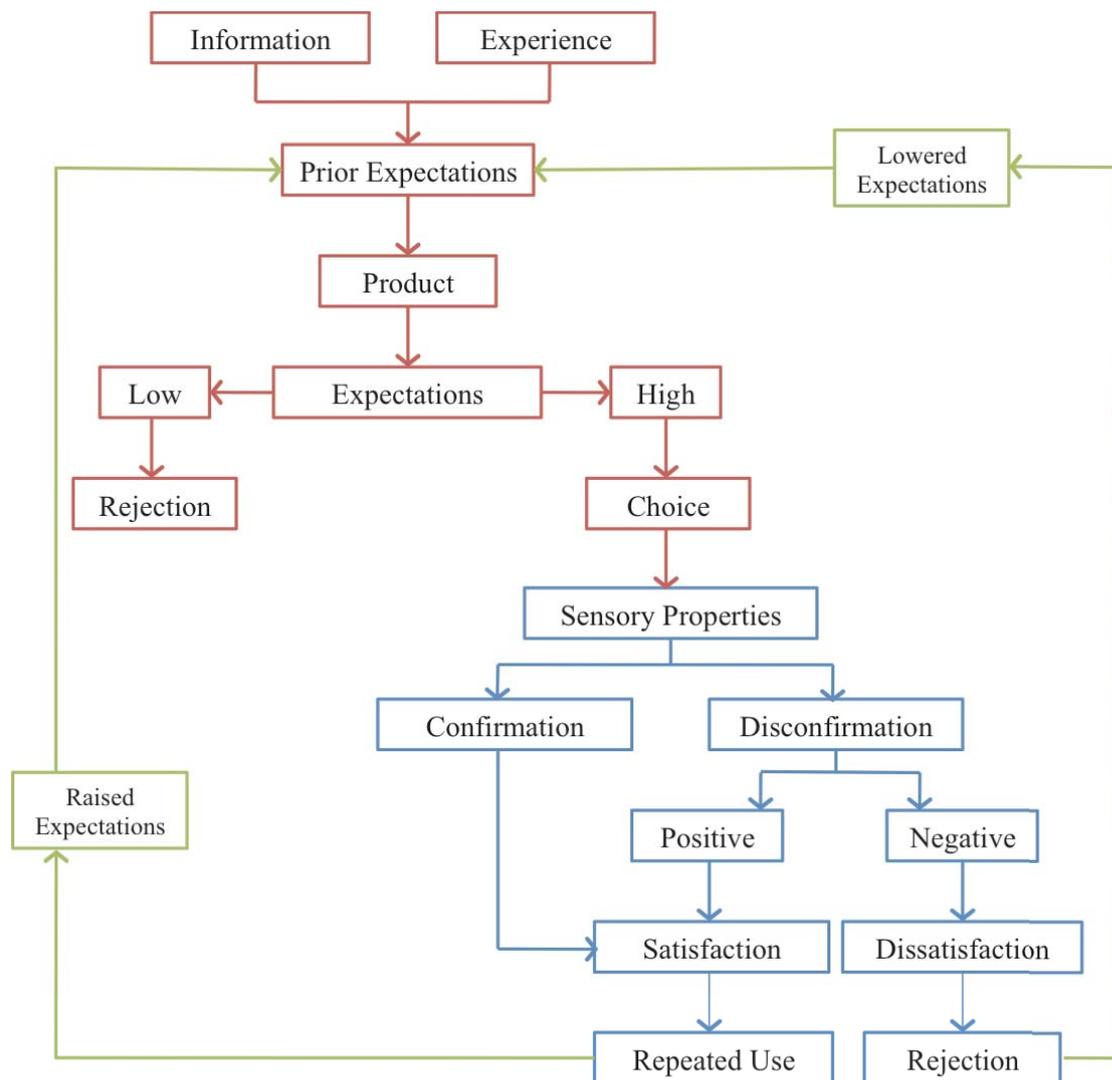


Fig. 1.1 Schematic model of the effects of expectations on product selection and evaluation. Modified from Deliza *et al.* (1996). For explanations of colour refer to text below.

Figure 1.1 is taken from Deliza *et al.* (1996), and depicts the chain of events that are hypothesised to occur during consumer product choice. The colours are defined as:

- In *red* the role of expectations on product choice: information, experience with the same or similar products in the category, prior expectations, and the product itself lead to expectations that are either low, and result in rejection, or high and result in choice.
- In *blue* the consequences of use on product evaluation: after choosing the product its sensory properties will be compared with those expected, so the consumer may confirm or disconfirm their expectation; both confirmation (matches expectation), and positive disconfirmation (better than expected) lead to satisfaction and repeated use, whilst negative disconfirmation (worse than expected) leads to dissatisfaction and rejection.
- In *green* how the experience of the product will result in raised or lowered expectations.

There are four psychological theories to describe how disconfirmation of expectations may influence perception: assimilation, contrast, generalised negativity and assimilation-contrast (Deliza & MacFie, 1996). The effect of these models are shown in Table 1.2. Assimilation theory suggests that any discrepancy between expectations and product performance will be minimised, so the consumer will alter the perception of the product to bring it in line with expectations. So, if expectations are low the product will suffer, but if expectations are high the product will be perceived as better than its actual attributes would suggest. Thus, creative marketing that establishes a

positive product and brand image, and therefore positive expectations, will improve liking of the product (Cardello, 2007). Contrast theory proposes that the consumer will magnify the disparity, and evaluate the product less favourably. Generalised negativity suggests that any discrepancy will result in a generalised hedonic state, resulting in unfavourable rating. Assimilation-contrast assumes that if the disparity is sufficiently small to be within the limit of acceptance the product will be put in line with expectation (assimilation), but if the discrepancy is large, and falls into the zone of rejection, the consumer will exaggerate the disparity (contrast). Large discrepancies between what the consumer expects and what the product can deliver should be avoided, as awareness of the discrepancy will create negative effect. The largest body of research supports assimilation theory (for example: Wansink, van Ittersum & Painter, 2005; Yeomans, Lartamo, Procter, Lee & Gray, 2001).

Table 1.2 The predicted effects of disconfirmed consumer expectations on the direction of increase/decrease in perceived product performance for the assimilation, contrast, generalised negativity and assimilation-contrast models. Adapted from Cardello and Sawyer (1992).

Model	Product Performance vs. Expectation	
	Positive Disconfirmation	Negative Disconfirmation
Assimilation	Decrease	Increase
Contrast	Increase	Decrease
Generalised Negativity	Decrease	Decrease
Assimilation-Contrast	Decrease	Increase
	(under low disconfirmation)	(under low disconfirmation)
	or	or
	Increase	Decrease
	(under high disconfirmation)	(under high disconfirmation)

1.4.3 Labelling

Many studies have investigated how label information influences expectations or sensory and hedonic ratings of foods, in addition to intake and willingness to pay for

the product. The studies documented here refer to the manipulation of label, the majority concerning fat content, or novel foods. Whilst Aaron, Mela and Evans (1994) found no significant affect of label ('reduced-fat spread 40% fat' or 'full-fat margarine 80% fat') on sensory or hedonic ratings, many researchers have found that labelling significantly affects hedonic ratings. Kähkönen and Tuorila (1998) found that when given information about fat content, participants expected the light version to be less pleasant, and Stubenitsky, Aaron, Catt and Mela (1999) showed that reduced-fat information had a small negative effect on the acceptance ratings of chocolate snack bars. Kähkönen and Tuorila (1999) found that reduced-fat information decreased pleasantness ratings and buying probability of chocolate bars compared with regular products.

Levin and Gaeth (1988) found that when information about fat was framed in a positive way the product was rated higher than when framed in a negative way. However, this framing effect is more pronounced in studies where the product is not consumed, implying that personal experience plays a prominent role in judgments.

Light, Heymann and Holt (1992) investigated the influence of label information on hedonic responses to normal and low fat versions of ice cream and cheese. Ice cream with information was liked significantly more than when presented without information. When participants knew the fat content of the cheese they tended to prefer the low-fat product over the higher version, possibly due to 'impression management' or social desirability: participants were trying to appear healthier than they really were. Furthermore, Tuorila, Kramer and Engell (2001) found that participants often chose a fat-free fudge against their hedonic preference, which the

authors suggests is evidence that motivated people can accept a less preferred product when they are informed of its reduced fat content.

Wansink, van Ittersum and Painter (2005) found that descriptive food names (geographic, nostalgic or sensory-related in nature, for example ‘Traditional Cajun Red Beans with Rice’) resulted in the belief that the meals were more appealing, tastier and more caloric than food presented with basic descriptive names (for example ‘Red Beans and Rice’).

Labelling has also been found to influence sensory ratings, with Kähkönen, Tuorila and Rita (1996) finding that a high-salt spread was rated as saltier when information about the salt content was available, suggesting that expected qualities and the sensory experience combine and result in higher ratings. Kähkönen, Hakanpää and Tuorila (1999) found that different types of information raised different expectations, with reduced-fat information leading to lower expected melting rate in chocolate. However, information did not affect the pleasantness ratings of the chocolate bars after eating, suggesting that consumers expect sensory differences between reduced-fat and regular fat products, but these expectations do not seem to affect hedonic ratings. However, Kähkönen (2000) found that receiving reduced-fat information during tasting decreased pleasantness ratings, and decreased fattiness, flavour intensity and melting-rate ratings for chocolate. Yeomans *et al.* (2001) found that when participants tasted both low-fat and high-fat soups presented with fictitious brand names that implied that the soup was high or low in fat, soups labelled as high in fat were rated as significantly more creamy, and more pleasant, regardless of the actual fat content.

Wansink, van Ittersum and Painter (2004) suggest that health or diet labels are likely to influence the subjective taste of unhealthy foods, but not foods that are already viewed as healthy, where the label has less of an impact. Wansink and Park (2002) found that a label indicating the presence of a "phantom ingredient" led to health claims becoming more believable, but negatively influenced taste perceptions, whilst Tuorila and Cardello (2002) found that liking of a fruit juice with an off-flavour (due to the addition of potassium chloride) was higher when accompanied by a health message. Furthermore, if authors informed participants that its special ingredient would improve either exercise endurance and energy or mental alertness and memory, the likelihood of consumption in the future was higher than if participants were told it would improve their mood and emotional well being.

Labelling also has an effect on hedonic and sensory ratings of novel food products. Yeomans, Chambers, Blumenthal and Blake (2008) found that a novel smoked salmon ice-cream was rated as more pleasant when labelled as a 'frozen savoury mousse' than when labelled 'ice-cream'. Furthermore, the 'ice-cream' was rated higher than the 'frozen savoury mousse' for saltiness, and overall strength of flavour. It was found that the 'ice-cream' label generated an expectation of a sweet, fruity flavour, and not salty or fishy flavour; this large disconfirmation led to an extreme dislike, when ratings during the control (unlabelled) condition were neutral.

Westcombe and Wardle (1997) looked at the effects of novelty and fat content, giving participants three versions of three types of food to taste: cheese (considered to be high in fat), yogurt (considered low in fat), and a novel, tofu based food. Each samples was labelled as either 'Higher', 'Normal' or 'Lower' fat, whilst the actual fat

content remained constant. An effect of labelling on ratings of several sensory attributes (creamy texture, oily and light texture), was observed, with the 'lower' fat samples being less creamy in texture and those labelled normal, or higher in fat. Furthermore, the foods labelled as lower in fat were thought to be less pleasant. However, those participants more concerned with health issues tended to perceive the higher fat labelled foods as less pleasant than those who were less concerned did. The novel food was affected by labelling type in the same way as the other foods; the authors suggest that knowledge and experience of a food probably facilitates greater responding to labels, but the effect still exists in novel foods. Tuorila, Meiselman, Cardello and Lesher (1998) investigated whether information could increase positive responses to novel products. Participants tasted novel products accompanied by different types of information: no information; positive information emphasising the product's uniqueness; positive information with details of the product category and other familiar foods. It was found that product category information led to decreased hedonic responses, due to negative disconfirmation, whilst hedonic ratings increased without this information. Furthermore, degree of liking, or frequency of use of a reference product predicted novelty acceptance, regardless of the information given.

Labelling may also affect energy intake. Provencher, Polivy and Herman (2009) investigated how describing an oatmeal-raisin cookie as either a healthy snack or an unhealthy snack affected *ad libitum* (self-regulated) intake, and how restrained eating (i.e. monitoring and attempting to limit food intake) and having one's weight made salient (i.e. being told one's current weight) affected intake. The authors found that all participants (regardless of restraint status, or weight salience) ate 35% more when the

cookies were described as a healthy snack than when they were described as unhealthy. However, the weight salience manipulation affected restrained and unrestrained eaters differently, with restrained eaters having a more negative evaluation of the snack foods (both in the healthy and unhealthy conditions) when they received weight feedback before eating, whilst unrestrained eaters gave more positive evaluations in the same condition. The authors suggest that this is due to the fact that the restrained eaters had a higher BMI, and possibly greater weight dissatisfaction.

Aaron, Evans and Mela (1995) investigated the effect that nutritional information had on nutrient intake during lunch at a cafeteria. Surprisingly, nutritional information resulted in an increased intake of total energy, fat and carbohydrate, and decreased intake of protein, although this finding was mostly due to changes in eating behaviour of both men and less restrained eaters. Authors suggest that this may be a consequence of participants anticipating that the foods that were higher in fat and calories would be superior in sensory quality, and would be more satiating. Furthermore, they highlight the issue of peer pressure that may affect behaviour in a public setting.

Wansink and Chandon (2006) investigated the effect that low-fat labels and serving size information had on consumption in both normal-weight and over-weight consumers. The authors suggest that when serving size is ambiguous consumers infer this from other cues, such as prior experience, or information found on the package or nutrition label, such as low-fat information. As such, nutrition labels could create misleading 'health halos', in which consumers believe that it is acceptable or

appropriate to consume more of a food labelled as being lower in fat. Furthermore, this may be related to food-related guilt: low-fat claims may lead consumers to eat more because it reduces the conflict between the hedonic goal of pleasure gratification, and the long-term goal of health preservation. The authors found that low-fat labels increased the consumption of foods by up to 50% across both hedonic (chocolate) and utilitarian (granola) snacks, with greater consumption of both foods by overweight consumers. Furthermore, serving size information prevented normal-weight participants from overeating foods labelled as low-fat, but did not influence overweight people. The authors conclude that consumers may trade-off taste reductions for increased consumption, so truthful labels and claims may not be sufficient to improve eating behaviour. The authors also point out that is important to consider when a low-fat claim would lead someone to eat so much more that it offsets lower-calorie density of low-fat foods, and how much low-fat labels affect consumption in subsequent meals (overconsumption and the choice of more indulgent extras).

Estimates of willingness to pay (WTP; the maximum price a buyer is willing to pay for a given quality of a good) can be a sensitive and valuable method for assessing the intrinsic value of a product (Lange, Martin, Chabanet, Combris & Issanchou, 2002), and has been used in labelling experiments. There are a number of methods of assessing WTP, including directly asking consumers how much they would be willing to pay, conducting experimental auctions where participants bid for a product (and often ultimately pay for it if they win) or choice-based conjoint analysis. Bower, Saadat and Whitten (2003) investigated the interplay of taste, price and information

(about a health benefit) on the intention to buy and willingness to pay for a fat spread. Authors found that label information had a significant effect on intention to buy, especially when combined with higher liking. Consumers were willing to pay more for the spread than the control spread, with willingness to pay being higher for females, older participants, those with high health concern and those with higher nutritional knowledge.

The effect that labelling has upon liking, perception of sensory attributes, intake and willingness to pay is clearly evident. Furthermore, the majority of researchers find that reduced-fat information decreases liking, whilst foods thought to be high in fat are rated as more pleasant.

This has a crucial impact on a reduced fat chocolate. Low expectations may prevent the initial purchase of the product, or may affect the hedonic or sensory ratings of the chocolate following consumption. The effect that fat content has on consumption is clearly important when aiming to reduce the prevalence of obesity. Furthermore, many researchers find that individual differences (e.g. health concern, restraint status, weight, age or gender) have an effect on hedonic ratings, *ad libitum* intake and WTP.

1.4.4 Individual Differences

A consumer's eating style, health concerns or individual characteristics such as gender, age or BMI may affect the response towards a reduced-fat chocolate. Gender differences may be important, as not only do women report liking and craving chocolate more than men (Rozin, Levine & Stoess, 1991), but they are more positive

towards low-fat foods (Solheim & Lawless, 1996), consume more energy-reduced and fat-reduced products (Fagerli & Wandel, 1999), and are more likely to be calorie conscious (Kiefer, Rathmanner & Kunze, 2005). Age may also affect liking, as it has also been found that reported cravings for sweet foods decrease with age (Pelchat, 1997), and that age influences one's preference for comfort foods (Wansink, Cheney & Chan, 2003). Wansink, Cheney and Chan (2003) found that as people aged, snack-related comfort foods (such as chocolate) made them feel less unhealthy than the same foods made younger people feel. BMI may also have an impact on attitudes towards reduced-fat chocolate, with overweight and obese individuals showing a tendency towards greater liking and selection of energy-dense foods (Mela, 2001). Furthermore, Drewnowski, Kurth, Holden-Wiltse and Saari (1992) suggest that “preferences for major nutrient sources of fat as opposed to carbohydrate may be a primary characteristic of human obesity syndromes”, with obese women naming chocolate, among other sweet, high fat foods, as one of their most preferred foods.

A consumer's eating style, and levels of restraint, emotional or external eating may also have an effect on their attitude towards a fat reduced chocolate, as may their levels of ambivalence towards chocolate. Cognitive dietary restraint is “the perception that one is constantly monitoring and attempting to limit food intake in an effort to achieve or maintain a desired body weight”, and has been related to a higher probability of choosing reduced-calorie or reduced-fat foods (Rideout, McLean & Barr, 2004). Emotional eating describes eating, or overeating, in response to emotional arousal states such as fear, anger, anxiety or loneliness, and has also been related to a higher consumption of sweet foods in both men and women (Konttinen,

Männistö, Sarlio-Lähteenkorva, Silventoinen & Haukkala, 2010). External eating describes eating more in response to external cues such as the sight, smell or taste of food, or the anticipation of food. It is thought that external eaters are insensitive to internal, physiological hunger and satiety signals, so the external environment determines eating behaviour (Tatjana van Strien, Herman & Verheijden, 2009). It has been found that the external environment is a good predictor of food craving, and that the craving of high fat foods has been linked to the development of obesity (Burton, Smit & Lightowler, 2007). Finally, ambivalence has been described as the “simultaneous existence of positive and negative evaluations of an attitude object” (Sparks, Conner, James, Shepherd & Povey, 2001), and can be associated with the conflicts between the sensory appeal of sweet and fatty foods and their perceived implication for health and body image (Cartwright & Stritzke, 2008; Rogers & Smit, 2000). Although chocolate is frequently liked and craved (Rogers & Smit, 2000), guilt often follows its consumption (Macht & Dettmer, 2006). Information about individual characteristics may indicate differences between the consumers that could be used to identify target markets, and result in successful marketing of a reduced-fat milk chocolate.

1.4.5 Chocolate

In the following section, a brief history of chocolate is given, the modern day production method is described, and methods of analysis are presented. Chocolate is a suspension of non-fat particles (sugar, cocoa solids and milk solids) in a continuous fat phase (cocoa butter). The main attractions of chocolate are its distinctive flavour, and melting properties: chocolate is solid at room temperature, but melts at body

temperature. However, the methods of producing, and the consumption of, chocolate has changed dramatically since its discovery. The following history of chocolate has been compiled from Beckett (1999, 2000), Coe and Coe (2000) and Minifie (1989).

The cocoa tree, *Theobroma Cacao* (meaning ‘Food of the Gods’), was discovered in the Yucatan Peninsula in southern Mexico in 600 AD by Mayans, who used the beans to produce the bitter chocolate drink “chocolatl”. This drink was made by roasting and grinding cocoa nibs, which were then mixed with cold water, spices, chilli pepper, vanilla or honey. It was used in everyday life and for ceremonial purposes. When the Aztec civilisation conquered the Mayans during the 1300s they discovered the drink, and used the beans as currency.

Christopher Columbus was the first European to discover cocoa beans, but it was Don Hernán Fernando Cortés who realised the commercial value of the beans, and following the invasion of the Aztecs in 1528, took cocoa beans and recipes for making chocolate back to Spain. The Spaniards omitted chilli pepper, but added vanilla, cinnamon, nutmeg and sugar to the drink, and served it hot. The popularity of the drink spread to Italy, Holland and France in the 1600s. The first chocolate drinking house opened in London in 1657, and in 1727 milk was added to the drink (generally attributed to Nicholas Sanders).

In 1828 van Houten of Holland invented the cocoa press, which removed part of the cocoa fat from the bean, resulting in a powder that made the drink easier to prepare and digest. This method also released cocoa butter making it possible to produce a fluid chocolate that could be moulded. In 1847 Joseph Fry’s factory in Bristol was the

first British factory to produce a plain eating chocolate, and in 1876 Daniel Peter in Switzerland invented milk chocolate. His invention was facilitated by the development of condensed milk by Henri Nestlé. In 1880 Rodolphe Lindt in Switzerland invented the conche to produce a smoother, better tasting chocolate, resulting in many manufacturers producing chocolate products during the early 1900s.

The modern day method for producing chocolate has been explained extensively by Beckett (2000), from which the following description has been adapted. Cocoa beans (cotyledons) are fermented, dried and roasted to produce cocoa nibs. The nibs are then milled, which melts the fat (cocoa butter), and releases it from the cells of the beans, so that it can be separated from the cocoa solids. Chocolate also contains sugar, which can be produced from both sugar cane and sugar beet, and milk chocolate contains milk powder from cows milk. The sugar, cocoa and milk powder are often dried and mixed together to make chocolate crumb. The crumb is then refined, reducing the particle size from 2000 micrometers (μm) to 18-38 μm (depending on formulation). In the UK chocolates are refined to particle sizes between 20 and 30 μm (Beckett, 1999). Chocolate refined to over 35 μm can be detected as gritty and sandy and is generally not accepted. The ingredients are then conched for several hours, which turns the mixture from a paste into a liquid. Conching evenly distributes the cocoa butter, refines the particles, dramatically decreases the viscosity of the chocolate, reduces moisture content, evaporates volatile acids and is essential for the development of the chocolate flavour (Cook, 1963). During conching the temperature is controlled to between 55 and 65°C (Cook, 1963), although this differs depending on the type of chocolate (white, milk or dark) and the desired taste (e.g. caramel flavour can be

formed due to Maillard Reaction). Following conching the chocolate is tempered: it is cooled and heated according to a particular temperature profile to ensure that the cocoa butter crystallises in the right polymorphic form (discussed in more detail in 1.4.5.1). The chocolate can then be moulded, and packaged. The schematic presented in Figure 1.2 describes the chocolate process, from cocoa bean to chocolate bar.

1.4.5.1 Cocoa Butter

Cocoa butter is important in chocolate for its physical properties, especially melting characteristics. It is solid at room temperature, but can melt rapidly within the mouth: between 30 and 34°C. This melting temperature affects both the textural properties of the chocolate, and subsequent flavour release. Cocoa butter is mainly composed of triacylglycerols (TAGs), although it also includes di- and monoacylglycerols, free fatty acids, phospholipids, and other complex lipids (Smith, 2001). TAGs are composed of fatty acids that are attached to a glycerol backbone. Cocoa butter contains palmitic (20-26%), stearic (29-38%) and oleic (29-38%) acids in almost equal amounts, although this varies depending on the subspecies of the cocoa plant, and climate conditions (Smith, 2001). TAGs have polymorphic behaviour, i.e. they can exist in more than one crystal form with different molecular packing: α (alpha), β' (beta-prime) and β (beta), in order of increasing melting point and thermodynamic stability, due to increasingly more dense crystal packing. Cocoa butter is thought to have six polymorphic forms, which the chocolate industry numbers from I to VI (Wille & Lutton, 1966). The polymorphic form of the cocoa butter has a large impact on product quality as it is related to physical characteristics (snap, moulding, contraction, gloss and blooming) (Loisel, Keller, Lecq, Bourgaux & Ollivon, 1998).

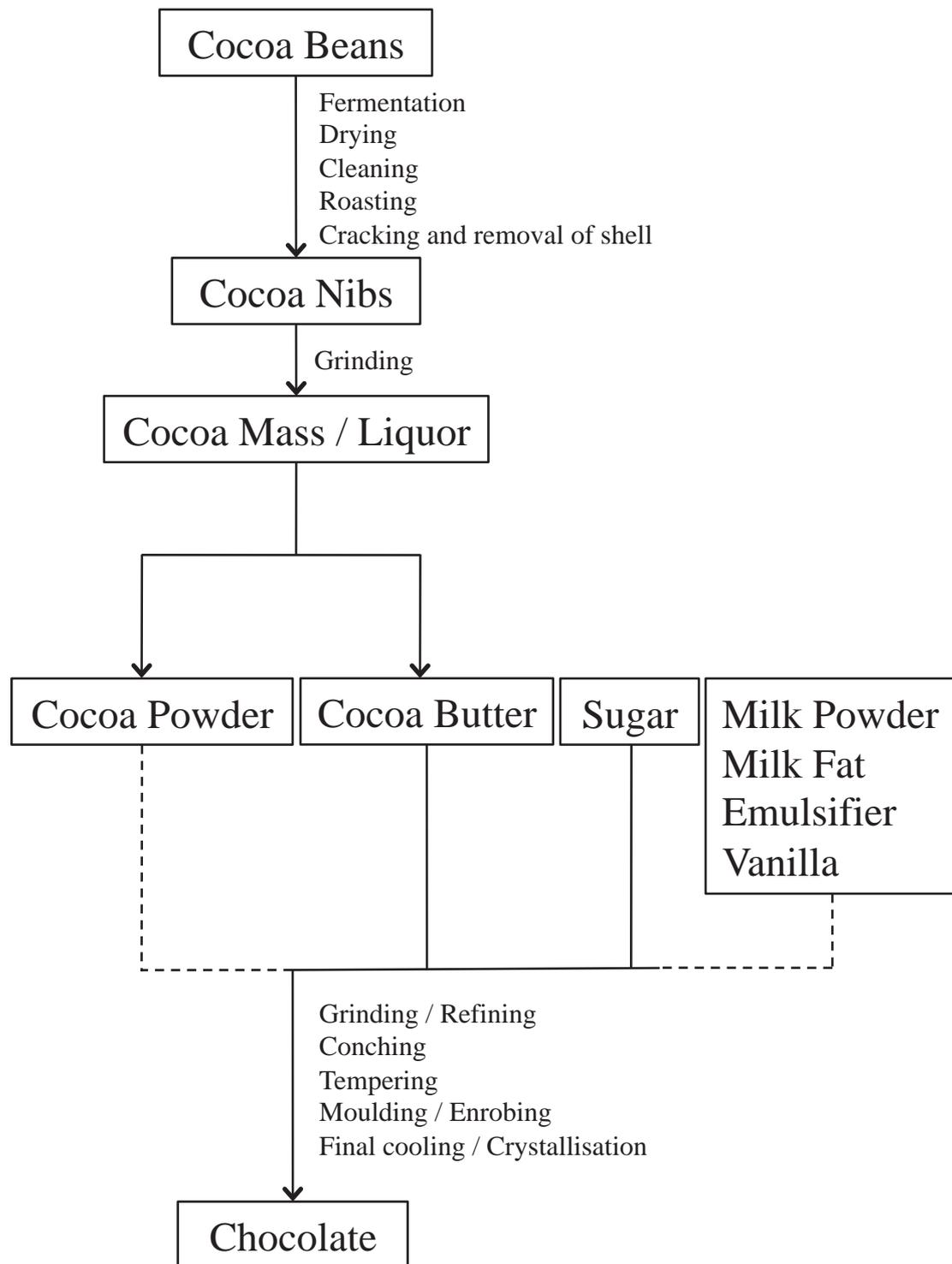


Fig. 1.2 Chocolate Processing adapted from Beckett (1999). The dotted lines represent optional additions.

Although the thermodynamically stable polymorph is Form VI (β_1), consumers find V (β_2) the most attractive, as it melts just below mouth temperature and is glossy; furthermore, this form demoulds efficiently, making manufacture easier. This form does not occur directly from melting, so crystallisation is usually into a less stable form (α or β'). In order to produce Form V, chocolate has to undergo a tempering (controlled crystallisation) process: a thermal regime carried out under shear. It involves heating the chocolate to between 50 and 60°C (to melt all crystalline material), cooling to a temperature above the α melting point (22-24°C, depending on formulation) to initiate crystallisation, before re-heating to a temperature above the β' melting point (26-28°C), and finally cooling to allow complete solidification of the stable Form V. This method relies on the development of stable Form V crystals to 'seed' subsequent crystallisation. 'Bloomed' chocolate has an inferior visual and textural quality. It results in a dull, rather than glossy, appearance, and white coating or spots on the surface. Several situations can lead to the development of bloom, as described by Bricknell and Hartel (1998):

- Insufficient tempering, so that stable V (β_2) crystals are not present;
- Cooling too quickly during tempering, so that the formation of unstable polymorphs will be promoted;
- High storage temperatures, or fluctuations in temperature, resulting in melting and recrystallisation;
- The presence of liquid fat components (fats with a lower solid fat content than cocoa butter), found in the fillings of some chocolates (e.g. ganache or nuts),

which can interfere with the formation of stable V (β_2), causing bloom on storage.

The addition of milk fat and milk fat fractions can also be used to inhibit bloom, although this can also result in softening (Bricknell & Hartel, 1998).

The two main techniques described in the literature for the analysis of chocolate and cocoa butter are Differential Scanning Calorimetry (DSC) and X-ray Diffraction (XRD). Both techniques are described here, in Sections 1.4.5.3 and 1.4.5.4, respectively.

1.4.5.2 The Importance of Fat in Chocolate

Fat gives chocolate its desirable physical characteristics, such as creamy texture, rich taste and melt-in-the-mouth ability. Fat content is particularly important as it has a dramatic effect on the material properties of chocolate, for example viscosity, snap and melting properties. Fat coats solid non-fat particles (such as sugars and cocoa powder) so they can flow past one another when chocolate melts (Beckett, 2000); the more fat there is in chocolate the easier it will flow both in manufacture and in the mouth. Most chocolates contain between 25% and 35% fat, although high quality chocolate may have a higher fat content (Beckett, 2000). Above 32% fat content there is very little change in viscosity with any further additions, but a 1% increase to a 28% fat content has a dramatic effect, and a chocolate with a 23% fat content is a paste (Beckett, 2000). Fat content may also affect flavour release. Although Knapp (1937) found that the chocolate precursors formed during fermentation were not in the fat phase, fat does contribute certain flavour characteristics to chocolate. Flavour

compounds tend to equilibrate between the lipid and aqueous phases: hydrophobic compounds will partition into the lipid phase, and hydrophilic compounds will partition into the aqueous phase (Decker, 2006). Reducing fat from a system exposes flavour volatiles to a more-hydrophilic environment, causing hydrophobic compounds to move from the hydrophilic environment into the gaseous phase (Decker, 2006). On consumption the nose will detect these compounds rapidly, causing flavour to be perceived as too strong. Fat within a food coats the mouth and may block some of the receptors, delaying and / or prolonging flavour release; similarly, removing fat from a food may result in an undesirably heightened experience of flavour at the early stages of consumption, and even a reduction in aftertaste. Thus, fat reduction in chocolate is challenging: the product should still flow during manufacture, be stable at room temperature, and melt in the mouth, tasting creamy and rich.

1.4.5.3 Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) is a thermoanalytical technique that measures the heat flow associated with thermal transitions in materials as a function of temperature (Höhne, Hemminger & Flammersheim, 2003). It can be used to determine the polymorphic form of cocoa butter because the polymorphs have different melting points and melting enthalpies. Many researchers have used DSC to study the polymorphs of cocoa butter, and Loisel *et al.* (1998) collated the data, which is presented in Table 1.3. Similarly, Chapman, Akehurst and Wright (1971) calculated the heat of fusion (cal/g) for each of the crystal forms, which can be converted to melting enthalpy, and is presented in Table 1.4. During a DSC experiment, a sample and a reference are subjected to identical temperature regimes, being heated or cooled

at a controlled rate. The amount of energy required to change the temperature of the sample in comparison to the reference (typically containing only air) is then measured. Two separate heaters (one for the sample and one for the reference) and a cooling system are used to keep the pans at the same temperature throughout the experiment. The energy required is a measure of that required to change the temperature, i.e. the heat capacity and latent heat changes in the sample relative to the reference.

Table 1.3 Melting point (°C) of cocoa butter polymorphs from the literature (Loisel *et al.*, 1998).

Polymorph	Duck (1964)	Wille & Lutton (1966)	Chapman, Akehurst & Wright (1971)	Huyghebaert & Hendrickx (1971)	Lovegren, Gray & Feuge (1976)	Merken & Vaeck (1980)	Davis & Dimick (1986)
I (γ)	18	17.3		14.9-16.1	13	16-18	13.1
II (α)	23.5	23.3		17-23.2	20	20.7-24.2	17.7
III (β'_2)		25.5	20.7	22.8-27.1	23		22.4
IV (β'_1)	28	27.5	25.6	25.1-27.4	25	26-28	26.4
V (β_2)	33	33.9	30.8	31.3-33.2	30	33.7	34.9
VI (β_1)	34	36.3	32.3	33.8-36	33.5		

Table 1.4 Heat of fusion, and calculated melting enthalpy for cocoa butter polymorphs (Chapman *et al.*, 1971).

Polymorph	Heat of fusion (cal/g)	Melting Enthalpy (J/g)
I (γ)	-	-
II (α)	19.5	81.5
III (β'_2)	21.5	89.9
IV (β'_1)	24.8	103.7
V (β_2)	28.1	117.5
VI (β_1)	32.9	136.3

1.4.5.4 X-ray Diffraction (XRD)

X-rays are high-energy electromagnetic radiation, with wavelengths between 0.1 Å and 100Å. The Ångstrom (Å) is a unit of length equal to 10^{-10} m (or 0.1nm). X-ray diffraction (XRD) is a technique used to characterise crystal structures. A beam of x-rays diffracts from the crystal structure differently depending on the arrangement of atoms, giving it a fingerprint. X-rays are used to produce the diffraction pattern because their wavelength (λ) is typically the same order of magnitude as the spacing (d) between planes in the crystal. From the angles and intensities of the diffracted beams it is possible to determine the density of electrons and the positions of atoms within the crystal. When the scattered waves cancel one another out there is no resultant energy leaving the sample; this is destructive interference. However, within a crystal, where atoms are arranged in a regular pattern, the waves will be in phase and add to produce a wave with a larger amplitude; this is constructive interference. A schematic of a diffracted beam from a crystal lattice is given in Figure 1.3. The conditions for constructive interference may be mathematically represented as Bragg's Law:

$$n\lambda = 2d \sin \theta \quad 1.4$$

where, n is an integer, λ is the wavelength, d is the distance between the planes in the atomic lattice (i.e. the distance between atomic layers in a crystal), and θ is the incident angle (see Fig. 1.3).

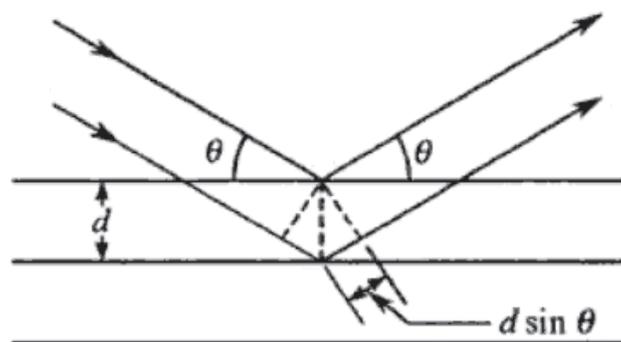


Fig. 1.3 A diffracted beam from a crystal lattice, indicating the elements of Bragg's Law, adapted from (Naik, 2010)

XRD can be used to identify cocoa butter polymorphs, as each has a different layer spacing, resulting in different diffraction patterns, typically between 3 and 5 Å (van Malssen, van Langevelde, Peschar & Schenk, 1999). It is used to determine the packing of the chains within the crystal, and therefore the polymorphic form, i.e. chains pack more densely in the β form than either the β' or α forms (thus, giving different scattering angles). Wille and Lutton (1966) first proposed the polymorphisms of cocoa butter, and presented x-ray diffraction patterns, and d spacings (as presented in Table 1.5).

Table 1.5 Short spacings X-ray data for cocoa butter polymorphisms, where W=weak, M=medium, S=strong and V=very (Wille & Lutton, 1966)

Polymorph	I (γ)	II (α)	III (β'_2)	IV (β'_1)	V (β_2)	VI (β_1)
d spacing (Å)	4.19 VS	4.24 VS	4.92 VW	4.35 VS	5.40 M	5.43 M
	3.70 S		4.62 W	4.15 S	5.15 W	5.15 W
			4.25 VS	3.97 M	4.58 VS	4.59 VS
			3.86 S	3.81 M	4.23 VVW	4.27 VW
					3.98 S	4.04 W
					3.87 M	3.86 M
					3.75 M	
					3.67 W	3.70 S
					3.39 VW	3.36 VW

Very strong peaks at different d spacings can be used to identify the different polymorphs, for example a very strong peak at 4.58Å, and a peak at 3.75Å are characteristic of form V (β_2). XRD is often used in conjunction with DSC to clearly identify the different polymorphs, for example Chapman *et al.* (1971), Keller *et al.* (1996) and Loisel *et al.* (1998).

1.4.6 Methods for Calorie Reduction in Chocolate

There are a number of methods for reformulating chocolate so that it has a lower energy density, or a lower fat content. These include replacing the sugar, reducing the quantity of fat used, using lower calorie fats, introducing air or creating a cocoa butter emulsion. There are many patents in the area of calorie reduction in chocolate, or chocolate confectionary components.

Both polyols and bulking agents, although providing some energy, have greatly reduced energy densities compared with sucrose. Polyols (sugar alcohols such as sorbitol, isomalt, maltitol, lactitol, erythritol and xylitol) are used to replace sucrose in chocolate, as they are lower in calories and suitable for diabetics (Beckett, 2000). Bulking agents (such as polydextrose, oligofructose and inulin) are polymers of sugars such as glucose or fructose linked by bonds that cannot be split by enzymes in the digestive system. However, the energy density of the chocolate can still be high as the fat content remains unchanged, or is even increased. Polyols and bulking agents change the rheological properties of chocolate (Sokmen & Gunes, 2006), so that fat levels need to be kept high, thus preventing overall calorie reduction.

An alternative approach is to add micron sized air cells, or ‘microbubbles’, which maintain the physical properties of the chocolate, but reduce the calories on a volume basis. Robert (2006) patented a method for making aerated chocolate, in which gas bubbles of approximately 25 microns in diameter were dispersed homogeneously throughout the chocolate.

Alternative methods have included reducing the quantity of fat within the chocolate, such as Zumbo (1999), who created a reduced fat chocolate by manufacturing the chocolate as normal, then removing some of the fat using a cocoa liquor press. However, the fat content cannot be dramatically reduced without impacting on viscosity, causing processing problems (e.g. when tempering, moulding or enrobing), and changing the texture and mouthfeel of the product (see Section 1.4.8). Emulsifiers, for example lecithin, can be used to coat the particles, reducing the viscosity, but not if the fat content is below 30%. Alternatively, calorie content may be reduced by using lower calorie fats. Fat replacement should effectively mimic the characteristics of cocoa butter, in order to maintain the textural components of standard chocolate products. Beckett (2000) describes both Procter and Gamble’s Caprenin™ (from caprylic, capric and behenic acids) and Nabisco’s Benefat™ (a mixture of long and very short chain fatty acid triglycerides) as fat replacements. Furthermore, Kong-Chan (1989) patented a low calorie, low fat chocolate product containing an artificial sweetener, a non-digestible bulking agent and a cocoa butter substitute (a nondigestible polyol fatty acid polyester); McCoy, Madison, Self and Weisgerber (1989) patented a method of using sucrose polyesters as a cocoa butter substitute, and Cooper (1995) patented a reduced calorie cocoa butter substitute that

mimics melting properties of natural cocoa butter. However, cocoa butter substitutes are often incompatible with cocoa butter, so have to be used with cocoa powder, they are calorie reduced and not calorie free, and their use is restricted as they have side effects, including being nondigestible (Beckett, 2000).

The calorie content of chocolate (according to weight) could be greatly reduced by introducing water, which is calorie free. Molten chocolate typically has a moisture content of 0.5-1.5%, which is present mainly in the cocoa solids, and does not affect chocolate flow (Afoakwa, Paterson & Fowler, 2007). However, conventional chocolate production and processing methods avoid contact with water since even small amounts of water cause sugar particles to aggregate and form lumps, increasing friction and viscosity (Afoakwa *et al.*, 2007; Minifie, 1989). Thus, water can greatly affect chocolate quality, often resulting in softening, bloom and grittiness. However, by carefully controlling water introduction by creating a fat continuous water in oil emulsion (i.e. by making stable droplets and preventing water migration) the energy density of the chocolate could be greatly reduced.

Beckett, Hugelshofer, Wang and Windhab (2010) patented a process for manufacturing milk chocolate containing a higher than normal water content (up to 30% by weight water). The method involves mixing a dark chocolate material with a water in oil emulsion and mixing sufficiently to disperse the water throughout, without creating a continuous phase. Hugelshofer (2000) investigated this method in depth during his PhD study.

Ducret, Holz, Wang and Wille (1999) patented a method for producing chocolate that is reduced in calories, has a similar taste to chocolate and has increased heat resistance. The chocolate was produced by adding a composition containing water to tempered dark, milk or white chocolate in an extruder. The extruder used could be a single or twin screw extruder, with a double jacket for temperature control. The composition containing water could be an emulsion (e.g. cream or evaporated / condensed milk) or a gel (made with a gelling agent, such as carrageenan, gellan, a gum, gelatin or microcrystalline cellulose). Furthermore, the inventors suggest that water-soluble substances, such as flavourings, preservatives or minerals and vitamins (to produce a functional chocolate) could be added to the aqueous phase. The inventors also state that it is preferable to use the water containing chocolate as a centre, covered with standard chocolate. Both methods (Beckett *et al.*, 2010; Ducret *et al.*, 1999) add water to standard chocolate as opposed to starting from the raw ingredients.

Morikawa and Kurooka (2000) patented a method for manufacturing chocolate containing a water in oil emulsion, in which the chocolate was added to an aqueous component (e.g. strawberry puree, banana puree, fresh cream or evaporated milk). The inventors state that this water containing chocolate could be used in icings for cakes or coatings for ice-cream bars.

Padley and Talbot (1992) patented a method for producing a chocolate filling, comprising a water in oil emulsion, with an average droplet size of approximately 10µm. The emulsion was produced using a scraped surface heat exchanger and a scraped surface crystalliser, in which a dispersion of liquid fat in an aqueous phase is

phase inverted (see Section 1.4.7), with the aid of emulsifiers that promote phase inversion, to produce a fat continuous emulsion. The emulsion could also be stabilised using thickening agents, such as an edible gum or gelatin. Sweetening agents or flavourings could also be added to the aqueous phase. This confectionary filling can then be enrobed with chocolate.

Rey *et al.* (2007) patented a method for producing a low-fat confectionary product containing 0-20% oil phase (cocoa butter or equivalents), and 60-90% aqueous phase. A structuring agent (e.g. carrageenans, pectins, gellan, gelatins, guar, alginates, gums or proteins) is added to gel the water, in addition to any flavourings, colourants, sugars, preservatives or aromas. The product is a suspension of particles (cocoa, milk or starch) within the aqueous phase, which can then be added to the fat phase, which structures the product through a continuous crystal network. The cocoa powder is thought to temper the cocoa butter because the crystal seeds within the fat of the cocoa solids are sufficient to ensure crystallization in to form V. The product produced has a calorie content of less than 300kcal/100g. This product can then be used as a filling, or as a chocolate component.

Finally, Rousset, Rey and Sandoz (2008) patented a method for producing a low-fat frozen confectionary coating by producing a water in oil emulsion with at least 45% aqueous phase (water, sugar, milk powders and gelling agents) and less than 35% oil phase (cocoa butter or equivalent, emulsifiers and cocoa solids). This product can be used on ice cream or other frozen desserts, and has a caloric reduction of at least 50% compared with standard confectionary coatings.

As can be seen, there are many patents in the area of fat reduction in chocolate, specifically in the addition of water. However, the products mentioned in these patents are described as similar to a ganache: a soft mixture made when adding cream to chocolate that does not snap when broken. This type of product can only be used as a centre or filling, covered or enrobed in chocolate, or as a coating for a frozen dessert. None of the patents detailed are suitable for producing a solid chocolate bar. Thus, the production of a solid chocolate containing a fat continuous emulsion is an interesting challenge. In the next few sections the principles of emulsion science will be detailed, with the aim of producing a cocoa butter emulsion that can be used to reduce the fat and calorie content of chocolate.

1.4.7 Emulsions

Emulsions can be used to reduce the overall fat content of many foods through the addition of water, without dramatically changing their sensory attributes. An emulsion consists of two immiscible liquids (typically water and oil or fat), where one liquid (the dispersed phase) is distributed within the other (the continuous phase) as small droplets (Bancroft, 1912), normally in the size range of 0.1 - 100 μ m in foods (Dickinson, 1992). An oil in water (O/W) emulsion (e.g. cream or dressings) consists of oil droplets dispersed within the aqueous phase, whereas a water in oil (W/O) emulsion (e.g. butter or margarine) consists of water droplets dispersed within the lipid phase. Emulsions are created by homogenisation or emulsification, whereby supplying energy and mechanical agitation, it is possible to break up the dispersed phase and produce droplets. In the absence of emulsifiers, the type of emulsion formed is dependent on the fractions of the two phases, so that the phase with the

lowest volume will become the dispersed phase. Emulsions are thermodynamically unstable, so the two phases will separate over a period of time, attempting to minimise contact area (Rousseau, 2000). Emulsifiers are surface-active molecules that are used to stabilise emulsions by adsorbing to the surface of the droplet (discussed in more detail in 1.4.7.3). The type of emulsifier used then determines whether the emulsion will be O/W or W/O. Thickening and gelling agents can also be used to stabilise emulsions through increased viscosity or gelation of the aqueous phase, thus retarding movement of the droplets (discussed in more detail in 1.4.7.4). Particles, or crystals can also be used to stabilise an emulsion (discussed in more detail in 1.4.7.4).

1.4.7.1 Emulsification

Emulsions are created by supplying energy to the two immiscible liquids, normally as mechanical agitation. Emulsion formation, and the size of the droplets produced, is dependent upon droplet break up, droplet coalescence, and emulsifier absorption. Droplet break-up is determined by both interfacial forces and disruptive forces. The interfacial force responsible for keeping a droplet in a spherical shape is characterised by the Laplace pressure, which acts across the oil-water interface towards the centre of the droplet. Thus, to break up a droplet it is necessary to apply an external force that is larger than the interfacial force. For a droplet to be broken during emulsification, the disruptive forces must exceed the interfacial forces.

High-shear (high-speed) mixers are a commonly used method of emulsification. The two phases are placed in a vessel, and are agitated by a mixing head that rotates at a high speed, which disrupts the interfaces between the oil and aqueous phases,

breaking larger droplets in to smaller ones. The mixing head can be altered to gain smaller droplets, decrease emulsification time, and gain uniform mixing. This method typically has no temperature control, resulting in increases in temperature (the mechanical energy is converted in to heat).

Margarines are produced in a continuous process, which comprises a sequence of cooling and crystallisation steps. Thus, emulsification and fat crystallisation occur simultaneously. A ‘margarine line’ comprises a scraped surface heat exchanger (‘A unit’), and a pin stirrer (‘C unit’) (de Bruijne & Bot, 1999). The A unit consists of rows of scraper blades mounted on a rotating axis, which scrape the inside of the shaft. The droplet size of the emulsion is primarily determined by the intensity of flow around the scraper blades, typically producing a droplet size of 5µm in margarine (Bot, Flöter, Lammers & Pelan, 2007). Water, or a cooling fluid, is flowed through the cavity of the jacket during emulsification. The combination of cooling and shearing in the A unit is necessary to promote the formation of fat crystals around the water droplets, required for Pickering stabilisation (see 1.4.7.4). The C unit consists of a shaft with pins on the inside, and a rotor with pins mounted on it. In margarine manufacture the C unit is used to provide residence time for crystallisation of the fat that was cooled in the A unit (Bot *et al.*, 2007). The process can be used to manipulate crystal nucleation and growth.

1.4.7.2 Emulsion Stability

There are a number of ways in which an emulsion may become unstable (Dorota Johansson, Bergenståhl & Lundgren, 1995; Rousseau, 2000):

1. *Creaming*: a form of gravitational separation in which droplets move upward because they have a lower density than the continuous phase. The rate of creaming is proportional to the difference in density;
2. *Sedimentation*: a form of gravitational separation in which droplets move downward because they have a higher density than the continuous phase. Again, the rate of sedimentation is proportional to the difference in density;
3. *Flocculation*: a type of droplet aggregation, where individual droplets remain, but aggregate together, due to collisions between droplets;
4. *Coalescence*: a type of droplet aggregation, where flocculated droplets merge to form larger droplets when the thin film between the droplets ruptures. It is a mechanism by which an emulsion moves toward its most thermodynamically stable state, because of a decrease in the contact area between the oil and water phases. It causes droplets to cream or sediment more quickly as they increase in size, and will eventually result in a layer of oil at the top of the sample, or water at the bottom. Coalescence can be controlled by the type of emulsifier used, and the environment (for example, temperature), although these factors depend upon individual emulsion properties;
5. *Ostwald ripening*: the diffusive transfer of the dispersed phase from smaller to larger droplets, observed in inhomogeneous emulsions. It is driven by the difference in Laplace pressure (the pressure difference between the inside and the outside of the droplet, caused by surface tension) between droplets. The rate of droplet growth is determined by molecule diffusion across the continuous phase, due to solubility. It occurs because smaller droplets have a

higher surface energy than larger droplets and so have higher solubility. It can occur in both W/O and O/W emulsions;

6. *Phase Inversion*: observed when a W/O emulsion is converted into an O/W emulsion, or vice versa. Partial coalescence can lead to phase inversion. It may be induced by a change in the relative phase volume of the oil and water, or as a result of changes in temperature, or surfactant composition.

The rate at which an emulsion breaks down is influenced by formulation, environmental conditions (e.g. temperature or pH), and processing conditions (Rousseau, 2000). The droplet size distribution can affect the shelf life (e.g. microbiological stability), appearance (e.g. emulsion colour), texture and flavour release in emulsions. It is therefore important to control, and measure, droplets within emulsions. Droplet size distribution is affected by the type and amount of emulsifier used (whether there is sufficient emulsifier to completely cover the surface of the droplets, and the speed at which the emulsifier can adsorb to the surface of the droplet), the energy input (the intensity and duration of homogenisation), and temperature (which affects the viscosity of the lipid phase, and can alter the effectiveness of emulsifiers). Emulsions can either be monodispersed (containing droplets of the same size), or polydispersed (containing a range of droplet sizes).

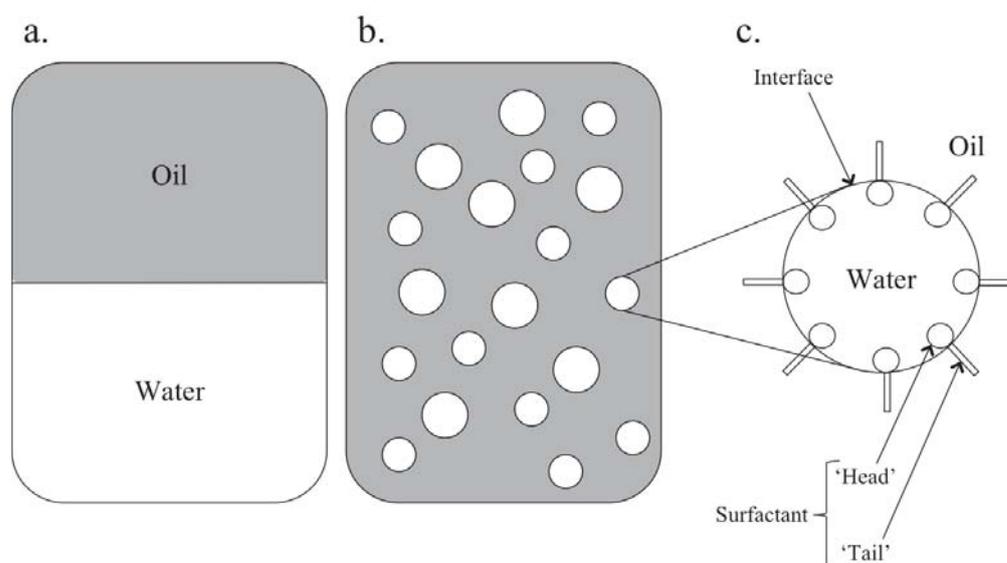


Fig. 1.4 An illustration of a polydispersed water in oil emulsion. a. showing the two phases at their most thermodynamically stable, separated according to density. b. an emulsion created by homogenisation with water droplets within a continuous oil phase. c. a single water droplet within the oil phase, with the surfactant adsorbed to the oil-water interface, showing the hydrophilic 'head' group, and the lipophilic 'tail' group.

1.4.7.3 Emulsifier use

An emulsifier is a surface-active agent (surfactant) that can adsorb to an oil-water interface during homogenisation and prevent droplets aggregating and coalescing during formation, and on storage. Emulsifiers work by lowering the interfacial tension between the oil and water phases, and forming a mechanically cohesive film around the droplets (Rousseau, 2000). The interface will bend so that the side with the higher surface tension will become concave, producing droplets. Most emulsifiers are amphiphilic molecules, having a hydrophilic ('water loving') 'head' group, with a high affinity for water, which is attached to a lipophilic ('lipid loving') 'tail' group, which has a high affinity for oil. Bancroft's rule states that the phase in which an emulsifier is more soluble constitutes the continuous phase; a hydrophilic emulsifier

stabilises O/W emulsions, whilst a hydrophobic emulsifier stabilises W/O emulsions (Bot *et al.*, 2007).

The hydrophile-lipophile balance (HLB) system is used to classify surfactants according to their chemical structure, giving an indication of its relative affinity for oil and water, and thus the likelihood of forming an O/W or W/O emulsion (Griffin, 1949, 1954). The HLB system is related to both interfacial packing and film curvature. A HLB number between 0 and 40 is assigned to different surfactants. The more hydrophilic the surfactant the higher the solubility in water; these surfactants have high HLB numbers, and stabilise O/W emulsions. They are made up of single saturated chains, with larger, more hydrated head groups with less oil penetration. More lipophilic surfactants that are soluble in oil have low HLB numbers, and stabilise W/O emulsions. They are made up of unsaturated double chains, with smaller, less hydrated head group and greater oil penetration. A surfactant with a HLB number of 3-6 is predominantly hydrophobic and stabilises W/O emulsions, whereas a surfactant with a HLB of 10-18 is predominantly hydrophilic and stabilises O/W emulsions. Blends of emulsifiers can be used to gain required properties.

The emulsifier must adsorb quickly to the droplet interface (the higher the surfactant concentration, the quicker the absorption rate), and the concentration of emulsifier should be high enough to cover the droplet surface. The surface excess, or surface load, (mg/m^2) refers to the saturation of emulsifier at the interface, and provides a measure of the minimum amount of emulsifier required to produce an emulsion with a particular droplet size (McClements, 2005). This requires knowledge of the droplet surface area, and the volume of the dispersed phase. This can allow for an

approximation of the required emulsifier to be calculated, although droplet size and stability is still affected by the processing parameters. If there is insufficient emulsifier to completely cover the interface, coalescence could occur if the droplets come in to close proximity, resulting in a larger mean droplets size. In a system with excess emulsifier (i.e. more than is needed to completely cover the interface), the droplet size is more dependent upon the processing parameters. When there is excess emulsifier present, it may begin to form multiple layers around the interface.

In this study two food emulsifiers commonly used in chocolate manufacture, and which are also used to stabilise W/O emulsions, were selected: lecithin and polyglycerol polyricinoleate.

1.4.7.3.1 Lecithin

Lecithins (E322) are naturally occurring surface-active molecules that can be extracted from soya beans, rapeseed and egg, and are a mixture of phosphoglycerides (Minifie, 1989). Lecithin typically has a HLB number of 8, meaning it is limited when stabilising W/O or O/W emulsions when used in isolation, but is often used in combination with other surfactants (McClements, 2005). Lecithin is also the most widely used emulsifier in chocolate manufacture, and is used to reduce the viscosity and flow properties of the bulk (Beckett, 2000) by lowering the interfacial tension between the dispersed phase (sugar, cocoa solids and milk powder particles) and the continuous phase (cocoa butter) (Schantz & Rohm, 2005). Addition of lecithin reduces the quantity of cocoa butter needed to gain the required viscosity, and thus reduces production costs. Current EU regulations state that lecithins may be used

according to *quantum satis* (i.e. no maximum level is specified, Personal Communication, 2011).

1.4.7.3.2 Polyglycerol polyricinoleate

Polyglycerol polyricinoleate (PGPR) is a surface-active emulsifier made from castor oil (Wilson, van Schie & Howes, 1998). It consists of polyglycerol (dominated by di, tri and tetraglycerols (Afoakwa *et al.*, 2007)) as the hydrophilic group and interesterified ricinoleic fatty acids as the hydrophobic group. It typically has a HLB of 1.5 (+/-0.5), and so is a powerful W/O emulsifier used to stabilise bakery fats and low fat margarines. It is also used to reduce the viscosity and rheological properties of chocolate by lowering the friction between the particles in the liquid fat phase, with the best effects being obtained when it is used in conjunction with lecithin (Wilson *et al.*, 1998). As the friction between the particles is reduced by PGPR, the amount of cocoa butter used can be reduced, thus reducing manufacturing costs (Schantz & Rohm, 2005). There are strict regulations governing the use of PGPR, with current EU regulations allowing a maximum of 0.5% in chocolate (Personal Communication, 2011), but it is often used in combination with other emulsifiers, such as lecithin.

1.4.7.4 Pickering emulsions and crystals at the interface

A Pickering emulsion is an emulsion stabilised by solid particles that adsorb at the interface between two phases (Pickering, 1907), stabilising O/W emulsions when wetted (as measured by contact angle) more by the water than the oil, whereas particles wetted more by oil will stabilise W/O emulsions (Binks, 2002). Fat crystals can also stabilise an emulsion. Individual crystals can behave like a) a Pickering

emulsion by stabilising the interface, and b) a fat crystal network that physically ‘locks’ droplets, preventing droplet movement and coalescence (Hodge & Rousseau, 2005; Norton, Fryer & Moore, 2006; Rousseau, Zilnik, Khan & Hodge, 2003). These fat crystals either crystallise directly at the droplet interface, or diffuse towards the droplet interface (Hodge & Rousseau, 2005).

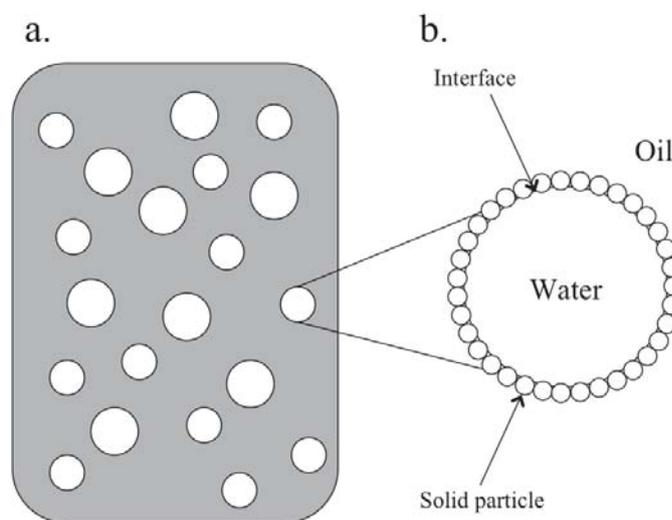


Fig. 1.5 An illustration of water in oil emulsion stabilised by solid particles i.e. Pickering stabilisation Hodge and Rousseau (2005) prepared emulsions in which the solid fat was crystallised either before or after homogenisation. The authors found that crystallising after homogenisation resulted in the formation of fine solid fat crystals, evenly distributed throughout the continuous phase, to restrict movement of the water droplets. However, emulsions made with precrystallised fats do not have a refined crystal structure, but consist of larger crystals, so water droplets, and the crystals themselves, are freer to migrate. Rousseau *et al*, (2003) show that the stability of the dispersed phase within spreads relies on the presence of a fat crystal network, and surface active crystals. In margarine and reduced-fat margarine, interfacial crystals

assist in the stabilisation of dispersed phase, especially at higher temperatures. Frasc-Melnik, Norton and Spyropoulos (2010) found that a combination of mono- and triglyceride crystals acted like Pickering particles to stabilise a W/O emulsion without the addition of emulsifiers, and observed crystals sintering to form ‘shells’ around the water droplets. Frasc-Melnik *et al.* (2010) and Johansson and Bergenstahl (1995) suggest that during crystallisation three processes occur: a) nucleation of new crystals, b) crystal growth, and c) the formation of bridges between crystals (sintering). These bridges form in the narrow gaps of the fat crystal network. Johansson *et al.* (1995) found that a small amount of fat crystals within emulsions increased flocculation and/or coalescence, whereas the addition of fat crystals above a critical concentration increased stabilisation. Johansson *et al.* (1995) suggest that fat crystals have a density close to water, which decreases gravity-induced flocculation and coalescence. Additionally, when crystals adsorb to the oil/water interface, the interfacial viscosity and elasticity increase, and thus slow down the coalescence process. Crystals slow down molecular movement at the interface requiring more time for the interfacial layer to be disrupted to allow coalescence. They suggest that stability is dependent upon the rate of fat crystallisation, the amount and form of the crystals produced, the temperature and shear, as well as the amount of emulsifier used.

Garti, Binyamin and Aserin (1998) used α -form crystals of hydrogenated fat (obtained by flash-cooling the oil phase) in combination with PGPR to stabilise liquid W/O emulsions. They suggest that whilst the fat particles play a role in Pickering stabilisation, the emulsifiers are molecular bridges and wetting agents that facilitate adsorption to the interface. They conclude that it is essential to have a sufficient

amount of emulsifier to ensure adsorption onto the fat particles to reduce aggregation of the crystals in the oil phase and crystal growth. They also state that α -form crystals are significantly more hydrophilic than β' or β crystals, and so will wet and anchor the water interface more successfully.

1.4.7.5 Hydrocolloids / gelatin

The word 'hydrocolloid' is commonly used to describe a group of polysaccharides and proteins (for example gelatin) that are commonly used in food products to control rheology and texture, stabilise emulsions (prevent coalescence, flocculation, sedimentation or creaming), and control organoleptic properties (for example mouthfeel or flavour release) (Williams, 2006). Hydrocolloids can be used in low-fat products to mimic fat, and may improve the spreadability of low-fat spreads and reduce moisture loss (Bot & Vervoort, 2006). Gelatin is a water-soluble protein derived from animal collagen (found in bone, tendon, skin and connective tissue), prepared by hydrolysing collagen by boiling it in the presence of acid (Type A) or alkaline (Type B) (Karim & Bhat, 2008). Gelatin is a versatile, multifunctional hydrocolloid, which has been used commercially in beverages, desserts, ice creams, yogurts, margarines and low-fat spreads, confectionary, bakery products, sauces, soups and cheese, as a stabiliser, thickener, or texturiser (Karim & Bhat, 2008). Gelatin is available in different gel strengths and particle sizes. Gel strength is determined by 'Bloom value', a standard method of measuring the force required to depress the surface of the gel (Karim & Bhat, 2008). Gelatin is often added to fat-reduced spreads to hinder coalescence by increasing the viscosity of the dispersed phase (Rousseau *et al.*, 2003). Furthermore, improvements in sensory characteristics

have been achieved by adding hydrocolloids to the aqueous phase (Clegg, Moore & Jones, 1996). The addition of hydrocolloids can give good flavour release and breakdown in the mouth. Gelatin is particularly favourable as it has a similar consistency and ‘melt-in-mouth’ behaviour to the fats typically used in foods (Clegg, Moore & Jones, 1996). Other gelling agents such as starch, alginate, pectin, agar and carrageenan lack the melting characteristics of gelatin (Karim & Bhat, 2008). Additionally, gelatin has a clean flavour profile, and transparent appearance, which has not been imitated by any other polysaccharide (Karim & Bhat, 2008). Finally, gelatin is easy to use, not requiring a change in pH, or addition of salts or sugars to set. Disadvantages of using gelatin include that it is an animal derived ingredient (Karim & Bhat, 2008), and it has variable physical properties and chemical heterogeneity due to differences in collagen sources and preparation techniques (Djagny, Wang & Xu, 2001).

1.4.7.6. Analytical Methods

1.4.7.6.1 Nuclear Magnetic Resonance (NMR)

It is important to measure the droplet size of the dispersed phase within an emulsion as it can be used to determine stability (i.e. as an indication of coalescence). Nuclear magnetic resonance (NMR) can be used to measure droplet size within both W/O and O/W emulsions (Goudappel, van Duynhoven & Mooren, 2001) when used in conjunction with droplet size analysis software. Droplet size distribution can be measured with precision similar to other techniques, with the advantage that sample preparation is non-perturbing and non-invasive, and opaque samples can be measured

(Johns, 2009; van Duynhoven *et al.*, 2002). It has been used to measure water droplet size in margarines and low-calorie spreads (Balinov, Söderman & Wärnheim, 1994). NMR works on the basis of restricted diffusion of the molecules (typically hydrogen atoms) within the droplet, and by applying pulsed field gradient sequences the parameters of a Gaussian distribution can be determined (Johns, 2009). When placed in a magnetic field the hydrogen nuclei are excited, resulting in a detectable signal.

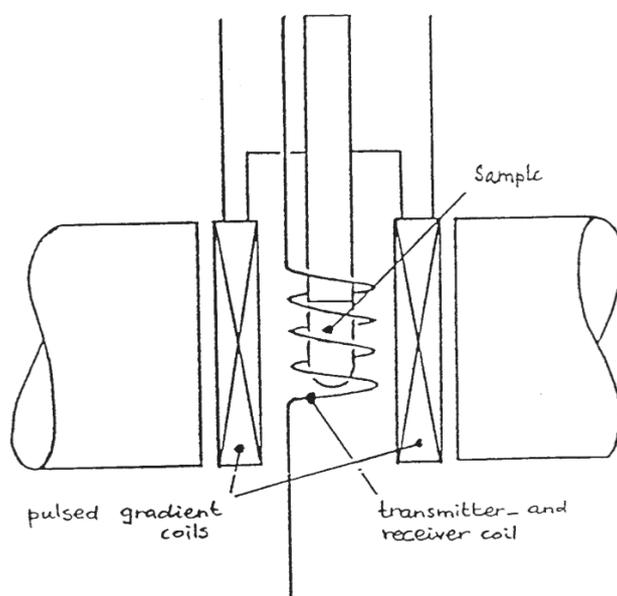


Fig. 1.6 The set up of the Bruker minispec NMR. Taken from the minispec manual.

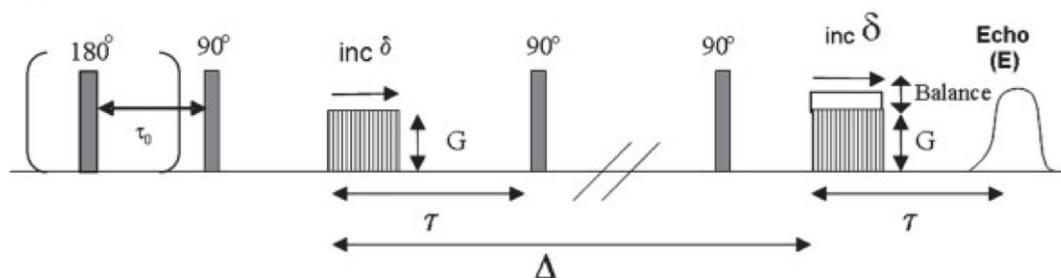


Fig. 1.7 Schematic representation of the pulse sequence used in stimulated echo sequence. Δ is diffusion time, δ is field gradient duration and G is strength of field gradient. In brackets the 180° pulse- τ_0 sequence which acts as a T_1 filter used for measuring W/O emulsions. Arrows correspond to increments and fixed parameters. Taken from van Duynhoven *et al.* (2007)

The technique gives a value of $d_{3,3}$, which is the volume weighted mean droplet diameter. This means that 50% of the total volume of water is present in droplets with a diameter smaller than $d_{3,3}$, 50% is present in droplets with a diameter larger than $d_{3,3}$, and σ is the standard deviation of the logarithm of the droplet diameter (van Duynhoven *et al.*, 2002). The surface-weighted value ($d_{3,2}$) can be calculated from the volume-weighted average, to gain a value that can be compared with that found from other droplet measurement techniques (van Duynhoven *et al.*, 2002). The methods does present some limitations, such as its reliance on sufficient restricted molecular diffusion due to the droplet wall (Johns, 2009), it cannot be applied to flowing systems (Johns, 2009), and assumes spherical droplets (van Duynhoven *et al.*, 2002).

1.4.7.6.2 Cryogenic Scanning Electron Microscopy (cryo-SEM)

Cryogenic scanning electron microscopy (cryo-SEM) is a standard technique for visualising the microstructure of foods, and is a suitable method for visualising emulsions. It allows observation at a nanometre (nm) to micrometre (μm) scale, to produce three-dimensional images. The path of the electron rays do not form a real

image, but construct a virtual image by creating line scans of the surface. As the beam moves through the defined area of the specimen, the signals generated vary in strength, which reflects the difference in the surface morphology. In cryo-SEM the water is physically fixed i.e. it is frozen. This is achieved using liquid nitrogen. The sample can also be fractured (using a knife) to reveal the internal structure of the sample, and etched to produce a stronger image. A schematic of the construction of the cryo-SEM can be found in Figure 1.8.

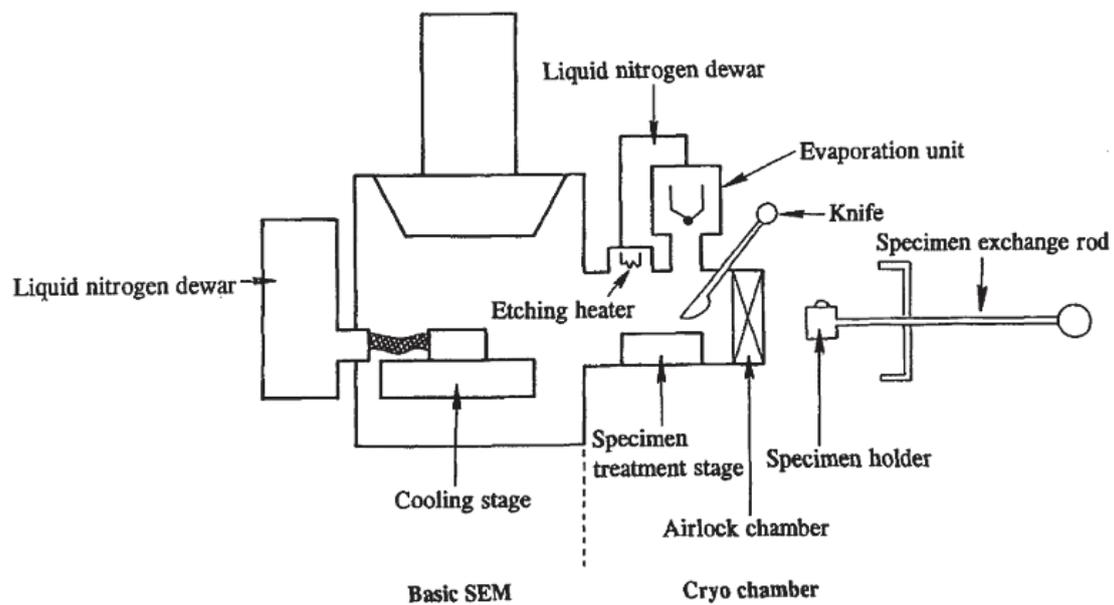


Fig. 1.8 Construction of cryo-SEM (Lee & Netto, 2006)

1.4.8 Summary of Literature Review

The aim of this chapter was to review the available literature with direct relevance to this study. A number of areas of literature have been covered, including:

- Research in obesity, and the reasons for reducing fat in food in order to reduce the prevalence of the disease;
- Psychological studies that have been conducted to consider the effect that expectations about food products, specifically with altered fat contents, health claims or novelty, have on hedonic and sensory perceptions, and on self-regulated consumption;
- The history and production of chocolate, the polymorphic behaviour of cocoa butter, and methods of analysis;
- Methods for reducing the fat content of chocolate, with emphasis on emulsion science, with a review of emulsification techniques, emulsion instability and stability (the use of emulsifiers, Pickering stabilisation, fat crystals at the interface, and hydrocolloids) and analysis techniques used to characterise emulsions.

The aim of the literature review was to identify the current understanding in the above areas, and to highlight areas of work to be carried out in this thesis. If a reduced-fat chocolate is to be adopted by the consumer, then combined understanding of the engineering needed to produce the chocolate, and the psychology of the consumer, is needed. If the chocolate is not adopted, or is consumed in excess by the consumer, in such a way to increase calorie consumption, then there will be no effect on obesity.

The literature review is in context to the two main areas of research that will be covered in this thesis:

- The investigation of chocolate consumption, beliefs about health and indulgence and consumer response to reduced-fat chocolate. This will be addressed using focus groups, a labelling study, and a packaging study, and is discussed in Chapters 3, 4 and 5;
- The investigation of formulation engineering routes for a reduced fat chocolate that has a similar flavour, texture and mouthfeel to a full fat equivalent. This will be addressed using a fat continuous W/O cocoa butter emulsion, and is discussed in Chapters 7, 8 and 9.

2. Exploring consumers beliefs and expectations: Materials and Methods

2.1 Introduction

A series of studies considering consumer beliefs about chocolate, health and indulgence, consumer expectations of reduced-fat chocolate and preferences for packaging are described in Chapters 3, 4 and 5. In this chapter a description of the materials and methods used for each of the psychology studies will be presented. General information, and information specific to each of the studies will be given.

2.2 General Methods

All studies were conducted at the University of Birmingham. Participants were recruited from the university, through a poster advertising campaign, or a combination of posters and flyers. The posters and flyers specified that the study was only open to those who consume solid milk chocolate. Undergraduate and postgraduate students and staff from The University of Birmingham, and local residents were recruited. The University of Birmingham ethics committee approved each study design for a term of three years (the time period in which all studies were completed; see Appendix 1). At the beginning of each study participants were asked to read a consent form (an example consent form can be found in Appendix 2), and at the end of each study participants received a debriefing form (an example can be found in Appendix 3). For all studies described in Chapters 3 and 4, except the packaging study (described in Chapter 5), participants received a £10 book voucher for completing the study. For

the focus group section of the packaging study, participants were given a £10 book voucher and a chocolate goodie bag for attending. For the rank-rating section of the packaging study participants did not receive any compensation. All statistical analysis was performed using SPSS version 16.0 for Mac and Microsoft Excel 2008 for Mac version 12.2.4.

2.3 Chocolate Consumption Focus Group

The results of this study are described in Chapter 3.

2.3.1 Participants

Nine participants (6 female, 3 male) were recruited into focus group 1, and six into focus group 2 (6 female). All participants were part or full time students, and varied in age (between 16 and 40). All participants in each group were British. Measurements of body mass index (BMI) were calculated from self-reported heights and weights in order to gain average data about the group. The groups had mean BMIs of 22.5 (ranging from 18.9 to 26.3), and 25.3 (ranging from 18.9 to 36.4). Therefore, on average both the groups were in the ideal, or healthy, weight to height ratio.

2.3.2 Apparatus

An Apple Mac OS X Version 10.6.4. laptop was used for video and audio recording. Soft drinks, plastic cups and napkins were provided for the participants. Sticky labels were used for participants to display their names. A flip chart, flip chart paper and

pens were required for recording important points, and plain paper and pens were given to participants to make notes if they wished.

2.3.3 Procedure

The discussion sessions took part in a quiet meeting room. Participants arrived and were seated at a large table, facing a flip chart board. The purpose of the session was explained to the group, and participants were asked to read and sign a consent form if they agreed to take part. Participants were informed that their input was optional and they could withdraw at any point. It was emphasised that there were no right or wrong answers, that it was the individual's opinions that were important, so nobody would be judged by their answers. Furthermore, they were informed that the aim of the session was to generate discussion by involving everyone in the group. Important points were written on the flip chart by the experimenter. The discussion session was also audio and video recorded using iMovie HD 6.0.3 (267.2) for Mac. Following the session this recording was transcribed, then deleted for confidentiality reasons. The following questions were addressed, in turn, during the session:

1. Which brands of chocolate do you eat regularly?
2. What do you look for in a chocolate?
3. Where does chocolate fit into your normal diet?
4. When do you eat chocolate? e.g. time of day
5. Would you eat chocolate as an evening meal?
6. When would you choose solid chocolate over a chocolate bar with a filling?

7. When would you choose to eat chocolate over other snacks?
8. How often do you eat solid milk chocolate?
9. How much do you eat in one session (portion size)?
10. Why do you eat chocolate (motivations for eating it)?
11. Do you think there any downsides to eating chocolate?
12. What do you feel like after eating chocolate?

Following the discussion participants were given a questionnaire (see Appendix 4), which included questions about gender, age, height and weight, occupation or area of study, nationality and questions taken from the The Dutch Eating Behaviour Questionnaire (DEBQ). The DEBQ was developed by van Strien, Frijters, Bergers and Defares (1986), and is used to indicate levels of emotional eating (eating in response to emotional arousal, such as fear, anger or anxiety), external eating (eating in response to external food cues, such as the sight and smell of food), and restraint (restriction of food intake). The questionnaire contains 33 questions measuring restrained eating (10 questions e.g., ‘Do you try and eat less at mealtimes than you would like to eat?’), external eating (10 questions e.g. ‘If you have something delicious to eat, do you eat it straight away?’) and emotional eating (13 questions e.g., ‘Do you have a desire to eat when you are emotionally upset?’). Response categories are never, seldom, sometimes, often, very often and not relevant (the questionnaire can be found in Section 2 of Appendix 4). Participants were then thanked for their participation, debriefed, and given their reward.

2.3.4 Data Analysis

The focus group audio recording was listened to and transcribed. The transcripts were then read in detail, as were the flip chart notes taken during the groups. This allowed for major themes and ideas to be identified. General themes, and contrasting opinions of individuals, were noted, and important quotes were highlighted. The themes and quotes were then interpreted to gain an understanding of the outcomes of the focus groups. Rabiee's (2004) paper was used as a guide for analysing focus group data. All the focus groups conducted during this work were analysed in the same way.

2.4 Health and Indulgence Focus Group

The results of this study are described in Chapter 3.

2.4.1 Participants

Three discussion groups were conducted. The size of groups varied (6, 8 and 10 participants). All participants were between 16 and 20 years old, and all but one were students (the remaining person worked locally). British and international students were recruited. Measurements of BMI were calculated from self-reported heights and weights to gain average data about the group. The groups had mean BMIs of 21.8 (ranging from 18 to 28), 23.6 (ranging from 17 to 32) and 22.5 (ranging from 19 to 24). Therefore, on average, both groups were in the ideal, or healthy, weight to height range.

2.4.2 Apparatus

Apparatus was as described in section 2.3.2.

2.4.3 Procedure

The procedure for this focus group was the same as the Chocolate Consumption discussion session. The following questions were addressed, in turn, during the session:

1. The meaning of the word 'healthy'.
2. Foods considered to be healthy.
3. The meaning of the words 'indulgence' or 'indulgent'. Other words or phrases that come to mind.
4. Is 'indulgence' a positive or a negative thing?
5. What sort of foods are considered to be indulgent?
6. Imagine a product that is aimed as an indulgent product, but which is healthy. What are your initial thoughts considering its characteristics?
7. What would you expect it to be like?
8. Who do you think would buy this type of product?
9. Would you be interested in buying a healthy indulgent product? Why?
10. Are there any particular occasions when you would eat a healthy indulgent product?
11. What would deter you from buying a healthy indulgent product?

2.4.4 Data Analysis

Focus group data was analysed as specified in 2.3.4.

2.5 Labelling Study

The results of this study are described in Chapter 4.

2.5.1 Design

The study was a within subjects design: participants tasted both samples to rule out individual differences. The order in which the samples were tasted was counterbalanced for participants between the first and second session to allow for any order effects.

2.5.2 Participants

One hundred participants signed up, 91 attended the first session, and 87 completed both sessions (63 female, 24 male); all participants were untrained consumers. The mean participant age was 24.3 years ($\sigma = 9.6$), but participants ranged from 18 to 60 years. The mean BMI was 22.5 ($\sigma = 3.9$), which is classified as normal, but participants ranged from 16.6 (underweight) to 40.6 (obesity class III). Of the 87 participants, 63 were British, and 65 said that English was their native language. 64% of participants said that they eat chocolate three times a week or more. 78% of participants said that they were very likely, or quite likely to try a lower-fat version of a food that they have tasted before.

2.5.3 Apparatus

Plain white ceramic plates (19 cm diameter) were used to present the chocolate to the participant. Labels, printed in black ink on white laminated paper, were displayed on the plate next to the chocolate. The chocolates were labelled either 'Reduced-fat Milk Chocolate' or 'Milk Chocolate'. The labels also included a random 3-digit code to make it plausible that the label was for experimental use, rather than for the participant's benefit (for example 'Sample 276: Reduced-Fat Milk Chocolate' and 'Sample 530: Milk Chocolate'). As potentially there would be 100 participants, 20 different random numbers were used (10 for 'Reduced-fat Milk Chocolate' and 10 for 'Milk Chocolate'), each participant received a different combination of numbers, to rule out any effects caused by the codes. The combinations were randomised between participants. The labels were printed (CMU Bright Roman, font size 28, centred, black ink) on plain white paper, cut to size (18cm x 6cm), and laminated.

On each testing occasion three squares of Somerfield own brand milk chocolate were given to each participant for expected liking, hedonic and sensory rating, and a further square for consumption questions. The squares of chocolate had a mean weight of 6.59g ($\sigma = 0.57$; the mean was taken from weight of all the squares in one 200g bar of the chocolate). Only full squares of chocolate were given; no half squares or broken pieces were presented. The chocolate was chosen as it did not have any branding on the surface. The chocolate was broken into squares prior to the session, and was served at room temperature.

2.5.4 Procedure

Prior to taking part in the study, potential participants were required to fill out a screening questionnaire (see Appendix 5) to assess their suitability for the study. It included questions on diet and food allergies, which was used to screen out those people who could not take part (for example, it could not be guaranteed that the chocolate did not contain traces of nuts or other allergens). The questionnaire also included questions related to chocolate consumption ('How often do you eat chocolate?', with the options 'more than once a day', 'once a day', '3 times a week', 'once a week', 'once a month', 'once every few months' and 'never'), and willingness to try low-fat foods ('If given the opportunity, how likely is it that you would try a lower fat version of a food you have tasted before?' with the options 'very likely', 'quite likely', 'neither likely nor unlikely', 'not likely' and 'very unlikely'). These questions were presented amongst other consumption questions, in order to disguise the aims of the study. Participants were asked to attend two sessions four weeks apart (on the same day of the week and at the same time of day). The chocolates were tasted on two separate occasions, exactly four weeks apart to ensure that female participants were tested at the same point in their menstrual cycle on both occasions (assuming that the average cycle is 28 days long). Chocolate cravings occur more frequently perimenstrually, especially for women in the UK, Canada and America (Zellner, Garriga-Trillo, Centeno & Wadsworth, 2004), so it was important that female participants tasted the two chocolates at the same time in their menstrual cycle. Male participants were subjected to this too, to ensure that the method remained constant. It was also important that the participants tasted the two

chocolates on the same day of the week and at the same time of day in both sessions (all sessions took place between 2 and 5pm from Monday until Friday) (Kramer, Rock & Engell, 1992; Stroebele & De Castro, 2004). Participants were asked to refrain from eating and smoking for 30 minutes prior to each of the sessions. On both occasions participants took part in the experiment individually, in a small quiet room, with one experimenter present. Two locations were used for the experiment, although each participant attended the same location for their first and second sessions. The temperature of the room was taken at the beginning of each session; the mean temperature of the rooms during the first sessions was 23.8°C ($\sigma = 1.23$), and during the second session was 23.5°C ($\sigma = 1.82$). The temperature of the rooms was therefore well below the melting point of the chocolate, and similar on both occasions. There were slight differences between the two locations, such as the size and lighting of the room, but as participants attended sessions in the same location, effects are thought to be limited.

During the first session, the nature of the experiment was explained to participants orally (without specifying the aim or labelling system); a script was used to ensure that instructions were given in the same manner to each participant (see Appendix 6). Written instructions were given, detailing what was expected of them, and how to fill out the questions that they would receive (see Appendix 7). Participants gave their written informed consent at the beginning of the first session. Participants were then required to eat one Matzo cracker (ingredients: wheat flour and water) and drink one cup of still, bottled spring water (Highland Spring), which was served at ambient room temperature. Both were given to cleanse the palate.

See Appendix 8 for the questionnaire used during the study. Expected liking ('How much do you think you'll like this chocolate?') was rated on a visual analogue scale (VAS) (147mm) with the anchors 'dislike extremely' and 'like extremely'. Participants were presented with the sample of chocolate, and asked to rate the expected liking of the chocolate in front of them. Ratings were made by drawing a vertical mark along the horizontal line at the point that best represented their opinion. Participants were asked to elaborate on their answer, and orally explain why they expected to like, or dislike, the product (answers were recorded by the experimenter). The participant was then asked to consume one square of chocolate completely. Participants were not instructed on how to eat the chocolate (i.e. to bite or not, suck or not), and if asked were told to eat it as they normally would. They were then asked to rate the chocolate for overall liking ('How much do you like the chocolate overall?'), again on a VAS, with the anchors 'Like not at all' and 'Like Extremely'. Again they were asked to orally explain their answer. The liking question was given before the sensory questions in order to prevent participants from becoming analytical in their rating of liking. The participant was then given a glossary of terms, which included a definition of each of the sensory terms they would be questioned on:

1. Melting in the mouth: the process of going from solid to liquid in the mouth;
2. Creamy / creaminess: having the consistency and texture of full fat cream;
3. Rich / richness: highly-flavoured; filling; may be overly sweet or heavy, possibly sickly or fatty;
4. Thick / thickness (mouthfeel): having a viscous consistency in the mouth when liquid; syrupy; the opposite of thin or watery;

5. Milky / milkiness: the taste / flavour of milk;
6. Smooth / smoothness: has a uniform, even texture; it isn't gritty or grainy;
7. Sweet / sweetness: one of the basic tastes; associated with the taste of sugar.

Participants were asked to read all the definitions, and refer back to them if they needed to. The definitions were given to standardise the questions, and to ensure that all participants (those of whom English was their native language, and those with English as their second or third language) understood all the questions given to them. The participants were then asked to work through the questionnaire at their own pace, and rate the chocolate according to a number of sensory attributes that are affected by the fat content of chocolate (melt-in-mouth characteristics and viscosity / thickness, (Guinard & Mazzucchelli, 1999) and those attributes described in focus groups as being important in chocolate (creaminess, milkiness, smoothness, sweetness and richness). Sensory attributes were accessed using both VAS questions (for example 'How fast do you think the chocolate melts in the mouth?' with the anchors "melts very slowly in the mouth" and "melts very quickly in the mouth") and Just About Right (JAR) questions. JAR scales are used to measure the desirability of a specific attribute (the deviation from ideal), and the optimum levels of an attribute in a product (Lawless & Heymann, 1999). Scales typically consist of five or seven points ranging from too little to too much for a given attribute, with a mid-point of 'just about right'. The participant was required to determine the intensity of the attributes, and rate them according to its distance from the ideal level of the attribute. If the intensity is ideal, the consumer should rate the attribute as 'just about right', but if it is not ideal the participant should choose the point on the scale that best represents the mismatch

between the intensity and the ideal point (Rothman, 2007). Prompts were given, indicating when the next two squares of chocolate should be consumed.

The participants were then given a second plate, with one square of chocolate, labelled in the same way, and were asked firstly to indicate how many squares of this particular chocolate they think they would eat during a normal sitting, and secondly, how much they would consume if they were not going to restrict their intake. Finally, the participants were asked whether they would purchase the chocolate, and at what price (for a 50g bar). Typical prices for chocolate were given (budget milk chocolate: 14p; supermarket own brand milk chocolate: 28p; branded milk chocolate: 49p; luxury milk chocolate: 89p), as a guide, although participants could give any price, and were not restricted to these categories. Participants were then asked to fill in a short questionnaire with details of time and date of last meal or snack time, and what they consumed, and if they are a smoker, when they last smoked (see Appendix 9). None of the participants ate, or smoked in the 30 minutes prior to the session, as requested. The participants were reminded of the date and time of the second session (participants were also reminded via email nearer the time).

The second session had the same format, although the chocolate was labelled in the alternative way (i.e. if in the first session they received 'Milk Chocolate', in the second session they received 'Reduced-fat Milk Chocolate', and vice versa). Following the second session, participants were given the same questionnaire containing questions about prior meals and snacks, and a questionnaire containing demographic questions (gender, current age, nationality, native language, current marital status, current occupation / area of study and educational status). Participants

were also given a chocolate consumption questionnaire (see Appendix 10). Sparks *et al.* (2001) developed a chocolate consumption questionnaire to assess both ambivalence and attitudes towards the consumption of chocolate, which includes measures of general attitude, semantic differential attitude, subjective norm, perceived behavioural control, health-conscious self-identity, intention, ambivalence and mixed feelings. Behavioural values were calculated using the methods described by Sparks *et al.* (2001). They were also asked to answer the DEBQ (van Strien *et al.*, 1986). Participants' height and weight was measured, which was used to calculate BMI. This was all conducted after the experimental sessions, to avoid priming participants' responses to foods. After the experiment participants were asked what they believed the purpose of the experiment was. Once the experiment was completed the participants were thanked for their participation, debriefed and compensated.

2.5.5 Data Analysis

A number of repeated measures ANOVAs (Analysis of Variance) were conducted to examine the main effects of labelling condition ('Milk Chocolate' or 'Reduced-fat Milk Chocolate') on expected liking, actual liking, sensory attribute ratings, consumption amount (normal and unrestricted consumption), purchase intent and price perception. Where appropriate, gender, age, BMI, normal chocolate consumption, willingness to try low-fat foods, restriction, external eating, emotional eating, and ambivalence were also used as between-subject variables in the analysis. T-tests were used to examine where differences in the data lie. All these variables, except gender, willingness to try low-fat foods and normal chocolate consumption, were separated according to a median split, to ensure groups were similar in size.

Groups varied slightly in size due to a large number of participants with the median value (for example participants with the same age as the median split value were placed in the same group, which meant that there were more participants in the below median age group than in the above median age group). Willingness to try low-fat foods was separated into two groups (likely and unlikely) from the original 5 groups. Those who gave the answer 'neither' were excluded from analysis. Normal chocolate consumption was separated into two groups according to frequency of chocolate consumption (three times a week or more, and once a week or less).

T-tests compare the values on a continuous variable for two groups. ANOVA compares the mean scores of more than two groups; it also compares the variance between the different groups (assumed to be due to the independent variable) with the variability within each group (assumed to be due to chance i.e. error). The F ratio represents the variance between the groups, divided by the variance within the groups; a large F ratio indicates more variability between the groups, than within groups. There are a number of assumptions underlying the use of both t-tests and ANOVA's:

- The dependent variable is measured using a continuous scales (interval or ratio data);
- The scores are obtained using a random sample of the population;
- Observations must be independent;
- The population are normally distributed (although large sample sizes can tolerate the violation of this assumption);

- Homogeneity of variance: samples are obtained from populations of equal variability.

Results were considered significant at $p < .05$, and trends towards significance were identified by $p < .1$. The results of the t-test are presented in the following way: $t(\text{degrees of freedom}) = t \text{ value}, p = \text{probability value}$. The results of the ANOVA are presented in the following way: $F(\text{hypothesis degrees of freedom, error degrees of freedom}) = F \text{ ratio}, p = \text{probability value}$. Means and standard deviations are also presented.

2.6 Packaging Study

The results of this study are described in Chapter 5.

2.6.1 Part A: Focus Groups

2.6.1.1 Participants

Eighteen chocolate consumers, 50% male and 50% female, were recruited and split into male and female groups, in order to encourage open, honest discussion, and to investigate possible differences in opinion between males and females. All participants were asked to complete a screening questionnaire prior to attending the focus group (see Appendix 11). It was required that participants typically consume chocolate at least once a month. Consumers were both British and non-British nationals, all were aged between 18 and 30, and were students.

2.6.1.2 Procedure

Participants gave their written informed consent at the beginning of the session. A discussion guide was used to ensure that the two groups followed the same procedure (see Appendix 12). A number of elicitation techniques were used to encourage discussion. A thought bubble completion task (see Appendix 13) was used at the beginning of the session to encourage the participants to think about chocolate consumption. The groups were then asked to work as a group to separate 24 photographs of chocolate bars (see Appendix 14) into those believed to be more healthy, and those believed to be less healthy. The images were printed on photographic paper using a commercial photographic printer. All were 5 x 7 inches in size, and were laminated. Prior to the study the photographs were selected out of a group of 41 chocolate bars, according to familiarity: thirteen males (Age: $\bar{\chi} = 29.7$, $\sigma = 10.4$) and nine females (Age: $\bar{\chi} = 22.9$, $\sigma = 0.9$) were asked to indicate whether they had seen the bars before. From this twelve familiar ($\geq 90\%$ of participants were familiar with the bars) and twelve unfamiliar ($\leq 90\%$ of participants were familiar with the bars) bars were selected. During the focus group, participants were encouraged to think about the packaging itself, and not on any previous knowledge of the chocolate enclosed. They were asked to explain their reasons for placing the bars in each of the piles. Finally, the participants were introduced to the idea of a reduced-fat chocolate bar. They were asked to work as a group on a sentence completion task. They were also asked to complete a similar thought bubble completion task, imagining that the man and women in the photographs were choosing the reduced-fat bar that was discussed during the session (see Appendix 15). Participants were then

thanked for their participation, debriefed, given a debriefing form, book vouchers, and a chocolate goodie bag as compensation.

2.6.1.3 Data Analysis

Focus group data was analysed as specified in 2.3.4.

2.6.2. Part B: Rank – Rating

2.6.2.1 Participants

Sixty female and sixty male participants were recruited. Participants were required to be 16 years old or above, and eat milk chocolate at least once a month. The mean participant age was 32.5 years ($\sigma = 11.9$), but participants ranged from 16 to 62, with similar mean ages for female ($\bar{\chi} = 32.3$, $\sigma = 12.2$) and male ($\bar{\chi} = 32.8$, $\sigma = 11.7$) participants.

2.6.2.2 Materials and Methods

Participants were given the sixteen packages (generated from the focus groups; details given in Chapter 5), which were displayed in a random order, and asked to fill out a questionnaire (see Appendix 16), which required consumers to rank the packages from least to most according to how much they expected to like the product. Participants were then asked to give the bar in position 1 (the least liked) a score of one, and the bar in position 16 (the most liked) a score of one hundred. Consumers were asked to score all the other bars in between. They were then asked to briefly explain their reasons for placing the bars in position 1 and position 16.

2.6.2.3 Data Analysis

An ANOVA was conducted to examine the main effect of packaging type on expected liking. Results were considered significant at $p < .05$. Multiple comparisons Tukey HSD was used in order to determine where the differences between samples lie. Conjoint analysis was used to determine the relative importance of each of the attributes, along with combined effects, in order to determine the optimum combination of attributes (Raz *et al.*, 2008). The overall evaluation of alternative products was then used to derive the partial contribution (or part worth utility) of each of the product features. Part worths for each of the packaging components were calculated using the following equation:

$$\text{Part worth of attribute A} = 0.5 * (\text{sum of mean scores for A} - \text{sum of mean scores for B}) \quad (2.1)$$

where A and B are manipulations of the same component (e.g. A = blue; B = caramel).

This method was applied to calculate part worths for all factors. A more positive part worth denotes greater expected liking for the reduced-fat chocolate bar with that particular factor.

3. Understanding consumers' beliefs about chocolate, and the concepts of health and indulgence

3.1 Introduction

As discussed in Chapter 1, chocolate is one of the most commonly craved foods ('chocoholism') and is associated with gift-giving, special occasions and reward (Hetherington & Macdiarmid, 1993). However, ambivalence (the simultaneous existence of positive and negative evaluations of an object, i.e. mixed feelings), towards chocolate is also often reported i.e. consumers may feel that the immediate benefits of sensory gratification (experienced when consuming sweet and fatty foods) conflict with longer term interests, such as preserving ones health and body image (Sparks *et al.*, 2001). Furthermore, Rogers and Smit (2000) suggest that chocolate is craved because it is highly palatable, resulting in positive hedonic and affective responses, and is often restricted, heightening its appeal, i.e. it is an indulgence. It is clear that the motivations for consuming chocolate are complex. Hetherington and Macdiarmid (1993) interviewed self-confessed 'chocolate addicts' (who state that they are unable to resist chocolate). Most indulged their cravings 75% of the time; of this group, 51% felt positive (happier, satisfied, calm, recharged or energetic) when consuming chocolate following a craving, whilst 49% felt negative (guilty, disappointed, unattractive, depressed, headachy, dissatisfied or sick). Furthermore, participants believed their excessive consumption of chocolate contributed to overweight status, and chocolate was considered high fat and of poor nutritional

value. The majority of participants stated that it was the sensory features of chocolate that made it addictive.

The aim of this section of work was to establish the motivations for consuming chocolate, including reasons for choosing one's favourite brand, where chocolate fits into the everyday diet, how much chocolate is typically consumed (i.e. portion size), occasions in which it would be avoided, and any downsides to consuming chocolate.

A number of hypotheses were identified:

H₁. Chocolate will be described as highly palatable, with pleasurable sensory characteristics;

H₂. Craving towards chocolate will be apparent in a proportion of chocolate eaters;

H₃. Ambivalence towards chocolate will be reported as it has high sensory appeal, but is highly calorific and is not consumed to sustain hunger. As such consumption is detrimental to health and body image;

H₄. Consumption of chocolate will often be restricted.

Secondly, the aim was to explore the concepts of health and indulgence, and discover whether there is a market for a product that is both 'healthy' and 'indulgent' (for example, a chocolate which is reduced in fat).

Participants were presented with the concept of a healthy indulgent product, and

asked to give their opinions. It is unclear whether consumers would favour it, as feelings of ambivalence are reduced (as energy density may be lower, so the impact on health and body image is decreased), or reject it as it has been shown that consumers often believe that sensory quality is often sacrificed in reduced-fat products (Lloyd *et al.*, 1995; Tuorila & Cardello, 2002). However, it was hypothesised that opinions of a healthy indulgence product would differ to a standard indulgent product, thus:

H₅. Opinions towards a healthy indulgent product will differ to opinions of standard indulgent products.

3.2 Experimental Design

During both the chocolate consumption focus group and the health and indulgence focus group a number of areas were discussed, as described in sections 2.3.3 and 2.4.3.

It should be noted that the answers given by the two groups for each of the discussion topics were very similar, with the same answers being given to many of the questions asked. It should be noted that the groups were only drawn from the University of Birmingham, and as a majority were students, so opinions may not be universal across all demographics.

3.3 Results

3.3.1 Chocolate Consumption

Participants were firstly asked to name the brands of solid milk chocolate that they eat regularly, and describe what it was that they particularly like about this brand. Brands given were those commonly seen in England, including Cadbury, Galaxy, Divine, Green & Blacks, Lindt, Milka, and Hershey, and participants in both groups also mentioned that they eat supermarket own-brand chocolate. Participants were then asked why this brand was their favourite. Reasons for liking this chocolate varied between individuals, but examples included those relating to taste such as creaminess, milky taste, smoothness and how well it melts, to the size of the bar, and the price. One participant said that being Fair Trade was particularly important, whereas another thought a high cocoa content was important.

Participants were next asked where chocolate fits into their normal diet. Participants said chocolate is eaten as a snack, in the afternoon, the evening or after a meal. Whilst some said that chocolate consumption is related to energy intake, and is eaten when feeling low in energy, or to boost one's energy before going to the gym, most gave emotional reasons for eating it. Reasons included being bored, such as on a journey, when feeling hormonal, in reaction to stress related to exams or studying, or after receiving bad news, when it is eaten for comfort:

“... if you are doing something you don't really want to do, if you have a bit of chocolate it makes it so much easier to do. It balances.”

Most said they eat chocolate at celebrations, such as Christmas, birthdays or Easter, and some said that chocolate consumption is related to relaxation, and is eaten when watching films at the cinema or at home:

“I feel like it’s a treat. It’s something you couldn’t eat a lot of all the time. You couldn’t have chocolate as your staple diet, unfortunately.”

Chocolate also seemed to be related to social interaction, being eaten with partners or friends:

“In our house it’s quite a social thing. I live with 5 other girls and we’ll all be sat down doing our work, and someone will say ‘ooh, shall we go and buy some chocolate?’... and we will all go over to the shop... It’s like having a drink, but with chocolate... a treat, or foods you shouldn’t have. Or something naughty... It’s really indulgent.”

Others said that chocolate is eaten on impulse, and that it “just happens”.

Next, the participants were asked to think about whether they would eat chocolate as their evening meal. This question was asked to consider whether chocolate is consumed for its high calorific value, and so would be considered an appropriate way to gain energy, or whether it is considered inappropriate for eating as a meal. Most said that you would still be hungry after eating chocolate, even if a large quantity had been consumed. This suggests that when chocolate is eaten it is not to sustain hunger.

Chocolate was thought to be too rich to eat as a meal, and would make you feel sick. Furthermore, participants said that meals are normally savoury, and not sweet, and chocolate is also not nutritious, balanced or fresh and contains the ‘wrong’ sort of calories. Participants also said that they would feel disgusted with themselves. Furthermore, they described it as socially unacceptable, and so they would feel like others were judging them. For these reasons, participants thought they would feel guilty afterwards:

“You’d feel so guilty. You’d wake up the next day a stone heavier.”

Others said that it would spoil the fun of eating chocolate as a treat, and so they would no longer look forward to it. They said that eating it as a main meal would not be as pleasurable as eating it after the main meal.

“It spoils the fun. It’s a treat, so if you’re having it all the time you’d get bored of it.”

Next, participants were asked when they would choose a solid chocolate bar over a chocolate bar with a filling, such as a caramel, nougat or wafer. Their answers related to the size and quantity of chocolate: solid chocolate is bought in a larger quantity, and so it lasts for longer and more can be consumed. Solid bars of chocolate were thought to be more appropriate as gifts, and were thought to be ideal for sharing. Furthermore, solid chocolate is thought to be more lavish, and more of a treat:

“If you are going to treat yourself I think solid chocolate is more luxurious.”

It was thought that solid chocolate is craved more than other bars.

“If you have a really bad chocolate craving you don’t want anything in it, you just want chocolate.”

Participants were asked when they would choose to eat chocolate over other snacks. Chocolate would be eaten when one was not massively hungry. This choice also depended on the time of day, as chocolate is eaten mostly in the afternoons or evenings. Chocolate was thought to be craved more than other snacks, possibly because it is sweet, and therefore not eaten as much as savoury food, and because makes you feel happier. The choice to eat chocolate depended on the person’s mood: chocolate would be chosen over other snacks if the person had had a bad day. Finally, chocolate was said to be eaten because it is naughty and rebellious.

“It’s naughty... you know you shouldn’t but you do anyway. And that makes it more fun.”

It is also thought that chocolate is marketed as an indulgence, and romantic.

“Chocolate is to do with Valentine’s day... romance. It’s a women thing. Women have chocolate. Women lie in baths and eat chocolate... It seems like an indulgent treat.”

Participants were asked about portion size, and how much they would consume in one sitting. There was thought to be no regularity, with portion size depending on many things, including whether you are trying to ‘be careful’ or not, whether you have

already eaten or expect to be eating soon, whether you have exercised (i.e. whether you deserve it), whether something ‘naughty’ has already been eaten during the day and whether you are stressed or upset.

“It depends on how much exercise I have done in the day. If I have been to the gym and worked out then I will think about the calories I have worked off... so I deserve that much. I feel I can eat it. If I haven’t worked out I don’t feel I deserve it.”

“I don’t really have any routines. If I’m in the mood for chocolate I will eat it. Unless I am in a stressful situation, in which case I will buy a big [bar] and go to town!”

Participants described eating more than they know they should, and eating the whole packet to get it out of the way.

“I always find I eat more than I should do. I always have a little taste, and it’s good, so [I] just plough on through until it’s gone and [I] feel sick.”

When asked about their motivations for eating chocolate, participants said chocolate was eaten as a treat, or a reward. It was also eaten for comfort, or as a pick-me-up. Others said it was out of routine, or boredom. Participants said eating chocolate makes them feel reminiscent and reminds them of childhood.

“Sometimes it’s a bit reminiscent... I pick up chocolate buttons because it reminds me of when I was little.”

Next, participants were asked when they would avoid chocolate. Most said they would avoid it before an occasion when it was important what they looked like, such as before the summer, or an event where photos of them will be taken, as chocolate causes bloating and bad skin. They also said they would avoid chocolate during a health kick, whilst on a diet, or after eating a salad. Similarly, if they had not done anything all day, chocolate should be avoided, as they do not deserve it.

“I won’t eat chocolate if I don’t feel like I have done anything productive with my day.... If I have achieved something... or just been out all day [I will eat it]... But if I’ve just sat in bed all day and done nothing I don’t want to eat chocolate.”

Participants thought that chocolate should be avoided before sport, as it does not give them enough energy or sustain them for long enough, and energy drinks were thought to be more beneficial before sport. Similarly, participants thought chocolate should be avoided on a long day, as it may cause an energy crash. Participants also said they avoid chocolate in the mornings, so would not eat it for breakfast, and when they have a headache or are feeling run-down.

Participants were asked if there were any downsides to eating chocolate. Many answers were given in relation to health, such as diabetes, weight gain, depressed immune system, bad skin, and tooth decay. Chocolate was said to be high in fat, and

include no nutrients. Other downsides included guilt, cravings, temptation and addiction.

“The guilt you feel after eating it. It says in magazines not to eat chocolate.”

Finally, participants were asked what they feel like after eating chocolate. This was thought to depend on the quantity of chocolate eaten. Feelings can vary between feeling more relaxed, happy and satisfied, and on the contrary feeling guilty, sick, regretful, disgusted, bloated, lethargic, physically heavier and headachy.

“I think there is a fine line between satisfied and sick. You don’t want to go too far.”

“Happy whilst you are eating it, but then afterwards you think ‘oh no, I shouldn’t have done that.’”

“If I do eat the right amount, say I have a 5 o’clock lecture, it does give me an energy boost. And it makes me concentrate more... it makes me feel happier about the fact I have a 5 o’clock lecture. It makes it a bit easier.”

There was a split between those that are left wanting more, and those that hate the thought of consuming any more.

To conclude, the focus groups indicated that chocolate consumption is extremely complex, being consumed on many different occasions and for many different reasons. It is clear that chocolate is not eaten for its high energy content, or to sustain hunger, as it contains the ‘wrong’ sort of calories, and conversely, is eaten in the absence of hunger. Chocolate is highly related to mood, and its consumption provokes many emotional words such as ‘guilt’, ‘naughty’ and ‘rebellious’. A lack of control over its consumption also exists, with the words ‘craving’ and ‘addiction’ being used. Whilst having many positive attributes, including pleasurable sensory attributes, a lot of downsides were recognised, including its high fat and sugar content, which were related to health issues and body consciousness (i.e. ambivalence). Similar conflicts of belief have been cited in the literature for sweet and fatty foods that have a high sensory appeal, but have implications for health and body image (Cartwright & Stritzke, 2008; Rogers & Smit, 2000). Thus, many restricted their intake, especially when trying to ‘be careful’, supporting H₄.

3.3.2 Health and Indulgence

In the second part of this study, participants were firstly asked to define the words ‘health’ and ‘healthy’. ‘Health’ was thought to encompass the absence of disease, sickness, ailments or injury, with being a healthy weight, having ‘normal’ body fat levels, ‘normal’ blood pressure, and having a positive outlook, being happy, and not stressed. Having a longer life expectancy was thought to be a good indicator of health. The explanations given by the participants for ‘healthy’ incorporated diet, exercise, mental state and the environment. Examples related to food consumption included having a balanced diet, with a variety of foods being consumed. Eating five portions

of fruit and vegetables every day was given particular importance. Eating fresh, or organic food, drinking plenty of water, and taking supplements were also mentioned. Avoiding convenience food, or food that is high in fat, sugar or salt was also considered important in order to be healthy, as was eating the 'correct' amount during the day. This may be related to calorie consumption, or bulk of food, but was not specified by the participants. Keeping active was mentioned by all groups as crucial. It was thought that regularly participating in sport, visiting the gym, or jogging were important. Having regular medical check-ups, taking medication and having vaccinations were believed to be important, as were hygiene and cleanliness. Lifestyle choices were also mentioned, with not smoking, not taking illegal drugs, having safe sex, avoiding binge drinking, having enough regular sleep, and avoiding sun exposure all being essential in order to be healthy. The environment that one lives in was thought to have an impact on health, with being near the countryside and getting fresh air, and avoiding car fumes, being ideal. Finally having plenty of social time, and avoiding stress were thought to imperative.

Participants were asked to name foods, or food groups that one should eat in order to be healthy. Fruit, particularly oranges, apples, watermelon, kiwis, berries and pomegranates, should be eaten for vitamin C. Vegetables should be eaten for vitamins, minerals and fibre, with broccoli and spinach being especially important. Carbohydrates (such as rice, pasta, couscous, corn, potatoes, bread and cereals), protein (such as lean meat, fish, pulses, lentils) and dairy products (including milk, eggs, yogurt, pro-biotic drinks, cheese for both calcium and protein) were all thought to be essential. Further examples included nuts and seeds, essential fats and oils, salts

and sugars, if in moderation, and water and herbal teas. Red wine and dark chocolate were also given.

Participants were then asked to define 'indulgent' or 'indulgence'. The words 'indulgence' or 'indulgent' were used as it they are thought to be suitable words to describe products such as chocolate. They are words that are not only used extensively by confectionary companies (for example Nestle and Lindt), but are thought to be familiar to consumers. The words are related to gratification, pleasure and desire. To 'indulge' is to allow yourself or another person to have something enjoyable, especially more than is good for you (Cambridge Dictionary Online). As all three groups were able to produce similar definitions of the words, and gave chocolate as the very first example of a food that is indulgent, the words are thought to be suitable. In contrast to the definition of 'health' and 'healthy', in which examples of diet, activity levels, lifestyle choices, and mental state were all given, the definition of 'indulgent' or 'indulgence' mainly related to diet and eating habits. The questions were worded in exactly the same way (i.e. "think about what the words 'health' and 'healthy' mean to you" / "think about what the words 'indulgent' or 'indulgence' mean to you"), suggesting that health does incorporate the whole of one's lifestyle, whereas indulgence is related more to consumption. There seemed to be two main definitions of the words indulgent and indulgence. The first related to a treat, that is eaten infrequently, mostly during special occasions, and when socialising (all groups became more animated when discussing which foods they considered to be indulgent):

“My immediate thought was ice-cream. It’s anything that feels like a bit of a treat. Or something you wouldn’t normally do, that’s quite nice to allow yourself once in a while.”

“Something you shouldn’t have a lot of, but it’s a treat.”

“But it is a good reason to gather with friends... And it is linked with celebration. So you have birthday cake. Or a big feast.”

The second definition related to excess, guilt and regret, gluttony, shame, stress and depression:

“You start feeling guilty, and think “I shouldn’t have done that”!”

“It is also sometimes quite secretive. And if someone finds you, you have to explain yourself, as to why you have eaten a tub of Ben and Jerry’s to yourself!”

“I think it is psychological, because if you know you have eaten 2000 calories, you feel like you can picture it on your body, “ooh, it has just popped up on my thighs””

Participants were asked to list a number of foods that they considered to be indulgent. Each group constructed a similar list, which included chocolate, ice-cream, biscuits and cakes, sweets, pizza, crisps, chips, take-aways and convenience foods amongst others. Participants were asked to describe how these foods were similar. These foods

were said to be addictive, tasty, moreish, immediately satisfying, highly enjoyable, but highly calorific and only short term investments.

The participants were then asked to draw upon their own definitions of the words 'health', 'healthy', 'indulgent' and 'indulgence', and imagine that a company has introduced a product that is both indulgent and healthy, giving their thoughts and opinions. There were a number of themes to the answers given. Participants considered that the company may be lying, or fabricating the truth, believing they would be suspicious, assuming there was a catch.

“...they probably use things like sweeteners. Which taste like sugar, but are actually not very good for you.”

It was also thought that 'healthy indulgence' is an oxymoron, or a contraction, so that it is impossible to be both healthy and indulgent, or it defeats the object of being indulgent.

“I would be disappointed in a way, because I might feel like I wasn't indulging as much. If you want something you don't normally have...”

“I don't know what I would think. Because it is healthy but indulgent, so am I meant to indulge more?”

“You indulge in something because you know that you shouldn't. It makes it better because you know it is bad for you.”

Participants also thought that the taste of the product would be inferior, and may taste subtler, or plainer, never comparing with an equivalent product. Finally, some of the participants said they would be curious and interested to find out more about the product, and thought that there would be a market for the product.

“Everyone wants chocolate to be healthy don’t they. And I’m sure if it was it would do very well.”

“...could sell it as a really indulgent product, that you wouldn’t feel guilty about.”

Although some members of the group believed there was potential for a ‘healthy indulgent’ product, the majority found the concept confusing, or were disapproving. This apparent negativity may not be related to the concept of a product being both healthy and indulgent. Instead these opinions may be as a consequence of the reality that most low-fat or low-calorie alternatives on the market are inferior in taste and texture to the higher calorie equivalents. If it was stressed that the healthy indulgent product had identical taste to the original product then opinion may have been different.

To conclude, whilst the words ‘health’ and ‘healthy’ encompassed many things including diet, exercise and mental state, the words ‘indulgent’ and ‘indulgence’ mainly related to diet and eating habits. Indulgence had two definitions, one that related to treats, and foods that are consumed during special occasions and celebrations, the second was related to stress, depression, excess, guilt and regret.

Chocolate was given as the very first example of an indulgent food. Indulgent foods were said to be tasty, highly enjoyable, addictive, but highly calorific. When asked to imagine a healthy indulgent product many thought that this concept was an oxymoron, and confusing, although some saw the potential.

3.4 Overall Conclusions

A number of hypotheses were identified:

H₁. Chocolate will be described as highly palatable, with pleasurable sensory characteristics;

H₂. Craving towards chocolate will be apparent in a proportion of chocolate eaters;

H₃. Ambivalence towards chocolate will be reported as it has high sensory appeal, but is highly calorific and is not consumed to sustain hunger. As such its consumption is detrimental to health and body image;

H₄. Consumption of chocolate will often be restricted;

H₅. Opinions towards a healthy indulgent product will differ to opinions of standard indulgent products.

The focus groups indicated that ambivalence is commonly experienced towards chocolate, as it has a desirable taste, but is also high in fat, sugar and calories, so its consumption often results in stress, depression, guilt or regret, supporting both H₁ and

H₃. This negative affect was related to body consciousness (weight-gain, bloating and bad skin) and health (diabetes and tooth decay). Furthermore, a lack of control was described, with many describing craving towards chocolate, or an addiction to it, supporting H₂. Many restricted their intake of chocolate, especially when trying to ‘be careful’, supporting H₄. Although many downsides to consuming chocolate were described, when presented with the concept of a ‘healthy indulgence’ product during the Health and Indulgence focus group, it was thought to be a confusing and suspicious contradiction. Furthermore, it was thought that it is impossible to be both healthy and indulgence. There were two reasons for this: i) foods are indulgent because they are bad for you; ii) the taste is often sacrificed in healthy alternatives. In line with H₅, opinions towards a healthy indulgent product differ from opinions of healthy or indulgent products.

When marketing a reduced-fat product, focusing on a reduction in fat and sugar the consumer may believe the taste will be inferior, as this appears to be how low-fat and low-calorie equivalents are perceived. However, by either focusing on the health benefits that can be gained by consuming less fat and calories, or by introducing the product as an indulgent product, with calorific values or health benefits playing a minor role, opinions towards the product may be more positive.

Di Monaco, Ollila and Tuorila (2005) used a focus group to discuss chocolate consumption, and the importance of both health and taste in chocolate consumption. The results suggested that the main purpose of eating chocolate is for pleasure, or to reward oneself. Thus, health issues are irrelevant as it is eaten only occasionally and cannot harm one’s health, and buying a ‘healthy’ chocolate bar would be

disappointing. These findings are very similar to the current study, indicating that this belief is common among different populations (their study was conducted in Finland).

The focus groups highlight the need to understand the impact that expectations about a reduced-fat chocolate bar have upon liking, and other aspects of the consumption experience. Thus, the next chapter (Chapter 4) addresses this in more detail.

4. Using labelling to explore consumer expectations of reduced-fat chocolate

4.1 Introduction

Many studies have investigated how label information influences expectations or sensory and hedonic ratings of foods, in addition to energy intake (typically *ad libitum* i.e. self regulated) and willingness to pay for the product (a review can be found in Chapter 1). A number of authors have specifically investigated chocolate, and found that reduced-fat information negatively affects acceptance (Stubenitsky *et al.*, 1999), pleasantness ratings and buying probability (Kähkönen & Tuorila, 1999), and resulted in lower expected melting rate (Kähkönen, Hakanpää & Tuorilla, 1999) and decreased fattiness, flavour intensity and melting rate during consumption (Kähkönen, 2000). Conversely, participants may rate low-fat products higher due to impression management or social desirability (Light *et al.*, 1992), or may choose a lower fat product against their hedonic preference (Tuorila *et al.*, 2001) for the health benefits. It has been suggested (Wansink & Chandon, 2006) that nutrition labels could create ‘health halos’, in which consumers believe that it is acceptable or appropriate to consume more of a food labelled as being lower in fat. Low-fat claims may lead consumers to eat more because it reduces the conflict between the hedonic goal of pleasure gratification, and the long-term goal of health preservation (i.e. feelings of ambivalence). Wansink and Chandon (2006) found that consumers trade-off taste reductions for increased consumption of low-fat foods, thus offsetting the lower-energy density.

In the previous chapter the results of two focus groups were described. They indicated that ambivalence is commonly experienced towards chocolate as it has a desirable taste, but also has a high fat, sugar and calorie content, and so its consumption often results in stress, depression, guilt or regret. However, the reduction of fat in chocolate was expected to result in an inferior taste, and the concept itself was thought to be a confusing oxymoron. The intention for this section of work was to explore in more depth opinions towards a reduced-fat chocolate. This was achieved by manipulating label information given during the consumption experience, in order to determine how expectations may change the perception of the product. Such an approach was used to establish the value of producing a reduced-fat chocolate, and consequently the most effective way to market it.

More specifically, the aim was to investigate whether labelling solid milk chocolate as either 'Reduced-fat Milk Chocolate' or 'Milk Chocolate' affects expected liking, hedonic and sensory ratings, expected consumption amount, purchase intent and price perception. On two separate occasions, four weeks apart, participants were given two identical samples of chocolate, accompanied by the different labels. Participants were asked to rate the chocolates according to liking: expected liking ratings were taken prior to consumption, and actual liking ratings were taken following consumption. Following consumption, participants were also asked to rate different sensory attributes: melting rate, creaminess, richness, thickness, milkiness, smoothness and sweetness. Participants were also asked how much of the chocolates they expect they would consume during a normal situation, and during a situation where they would

not be restricting their consumption amount, whether they would purchase the chocolate, and if so, for how much money.

The study also aimed to explore individual differences (gender, age, BMI, normal consumption amount, willingness to try low fat foods, external, emotional or restricted eating, and ambivalence) and how they may affect ratings, consumption amount, purchase intent or price perception.

4.2 Hypotheses

A number of hypotheses were identified:

H₁. The label condition will have an impact on ratings of expected liking, with ‘Reduced-fat Milk Chocolate’ being rated lower than ‘Milk Chocolate’;

H₂. Actual liking of the ‘Reduced-fat Milk Chocolate’ will be higher than expected liking;

H₃. When the chocolate is labelled ‘Milk Chocolate’ the expected and actual hedonic ratings will be similar;

H₄. Sensory attributes perceived as being related to fat content in chocolate will be rated lower, or higher (depending upon the attribute), when the chocolate is labelled as ‘Reduced-fat Milk Chocolate’ than when labelled ‘Milk Chocolate’;

H₅. When the chocolate is labelled as ‘reduced-fat’ a higher quantity will be consumed than when labelled as a standard chocolate.

4.3 Results

Specific information about the procedure used, methods of data analysis and presentation of data can be found in Chapter 2.

4.3.1 Carryover Effects

The data obtained was first analysed to investigate whether the session order had an effect on liking (i.e. any carryover effects). ANOVAs were conducted to consider whether ratings of expected or actual liking were affected by the session in which the chocolates were tasted. Significant effects (i.e. p -values $< .05$) were not observed for expected liking [$F(1,85) = .508, p = .478$] or for actual liking [$F(1,85) = .802, p = .373$] (mean ratings and standard deviations can be found in Table 4.1). This suggests that participants were not influenced by any experience gained during the first session when tasting the chocolate during the second session. Furthermore, when asked to indicate what they thought the purpose of the study was, none of the participants correctly guessed the aims. Typical answers included discovering opinions of taste and quality of different, newly developed chocolates, and investigating individual differences (for example, eating habits, health consciousness or mood) and the relationship with chocolate consumption.

Table 4.1 Mean ratings of expected and actual liking of the chocolates labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’ tasted during session 1 and session 2. In brackets standard deviation.

	Expected Liking		Actual Liking	
	Milk Chocolate	Reduced-fat Milk Chocolate	Milk Chocolate	Reduced-fat Milk Chocolate
Session 1	73.1 (2.6)	62.9 (3.1)	69.8 (2.7)	69.9 (3.3)
Session 2	62.1 (3.1)	54.8 (2.6)	64.2 (2.8)	60.8 (2.4)

4.3.2 Expected and Actual Liking

Next the effect of label type on expected and actual liking was analysed. As can be seen in Figure 4.1, a significant main effect of label type was found for expected liking, with participants having lower expectations of liking for the chocolate labelled ‘Reduced-fat Milk Chocolate’ ($\bar{\chi} = 58.88$, $\sigma = 18.8$) than the chocolate labelled ‘Milk Chocolate’ ($\bar{\chi} = 67.68$, $\sigma = 19.4$) [$F(1, 86) = 18.11$, $p < .0005$]. However, a significant effect of label was not found for actual liking, where the ratings of the chocolate were similar (‘Milk Chocolate’: $\bar{\chi} = 67.04$, $\sigma = 18.1$; ‘Reduced-fat Milk Chocolate’: $\bar{\chi} = 65.41$, $\sigma = 19.5$) [$F(1, 86) = 7.16$, $p = .4$], suggesting that the experience of the chocolate’s taste outweighed any expectations generated by the label.

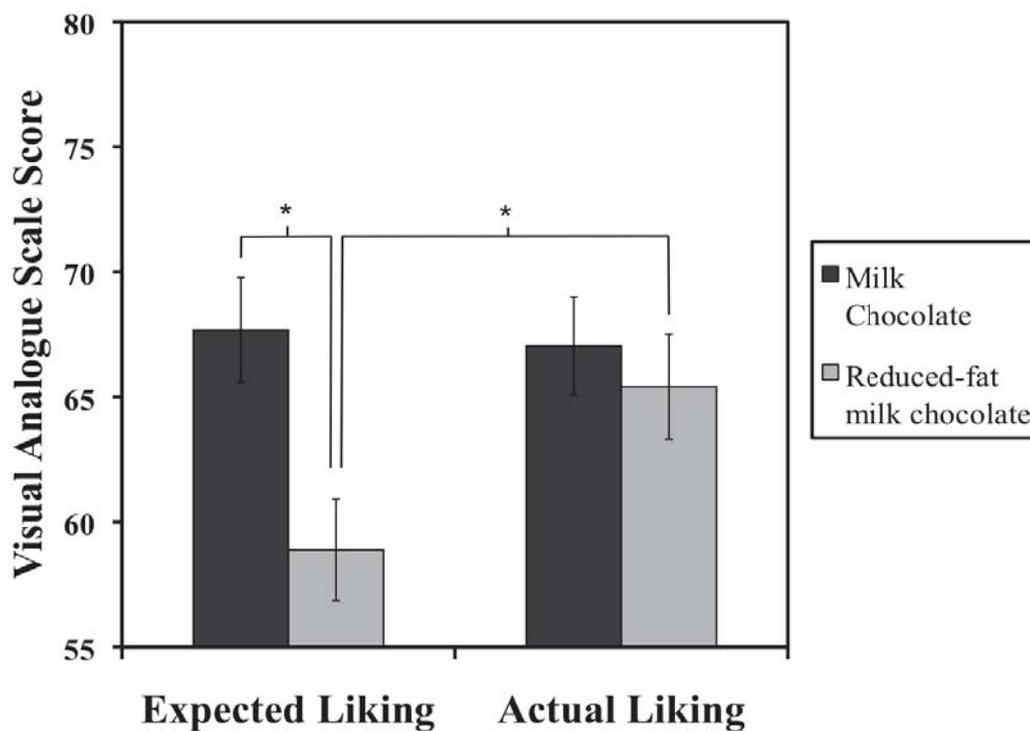


Fig. 4.1. Rated scores for expected and actual liking of chocolate samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’, showing score out of 100 where 0 represents “like not at all” and 100 represents “like extremely”. Data are means, and error bars represent one standard error of the mean. ‘*’ denotes a significance value ≤ 0.05 .

As can be seen in Figure 4.1, although a significant difference was not observed between expected and actual liking for ‘Milk Chocolate’ [$F(1, 86) = .121, p = 7.29$], a significant difference was observed for ‘Reduced-fat Milk Chocolate’ [$F(1, 86) = 14.35, p < 0.0005$], with higher ratings for actual liking. This indicates evidence of positive disconfirmation (see Chapter 1, Section 1.4.2) i.e. the chocolate tasted better than expected. However, participants did not like the reduced-fat chocolate more than the milk chocolate following tasting, so no expectancy-contrast effects were observed i.e. the participants did not exaggerate the disparity between the expected taste and actual taste, and thus rate it higher even than the standard chocolate. Thus, it appears

that the label was more powerful for expected liking prior to tasting and had less of an effect on actual liking, as a result of experience of the chocolate's taste.

Participants were also asked to explain why they expected to like, or dislike the product, and explain their ratings of actual liking. Summaries of consumer comments can be seen in Appendices 17 and 18.

When asked to explain expected liking ratings, descriptions for 'Milk Chocolate' and the 'Reduced-fat Milk Chocolate' were similar. These included general remarks, such as:

- a like, or dislike, of milk chocolate (in comparison to dark chocolate);
- the chocolate looked typical of other chocolate tasted before;
- visual aspects of the chocolate such as colour (how light or dark it looked), shininess, and an impression of its texture.

One positive reason for rating the 'Reduced-fat Milk Chocolate' highly was that it would be less fattening, which was thought to be a bonus. A number of negative explanations, not observed for 'Milk Chocolate', were noted as reasons for rating the 'Reduced-fat Milk Chocolate', including:

- it did not appear as creamy;
- it might be less rich;
- it might be less sweet;
- it will not taste as flavoursome or as tasty;

- it looked like a diet food.

These opinions were not given for the 'Milk Chocolate', so were as a result of experiencing the reduced-fat label.

When asked to explain actual liking ratings, reasons were variable, and indicative of personal taste. For example, actual liking scores of both the 'Milk Chocolate' and the 'Reduced-fat Milk Chocolate' were affected by perception of sweetness, where some believed the products to be too sweet, and others not sweet enough. This was observed for many of the attributes, including chocolate flavour, milkiness, smoothness, melt-in-the-mouth characteristics and creaminess. Interestingly, some participants were of the perception that the reduced-fat chocolate tasted 'light' or 'reduced-fat', though no differences in sensory attributes between the two chocolates were described i.e. no explanation for this description can be observed in the comments given. It may be that although the same shortcomings were observed for the standard milk chocolate, when the product was thought to be reduced in fat, the word 'light' was used to describe these deficiencies. It is interesting to note that although during the first session participants had not been familiarised with the vocabulary that would be used during the rest of the study questionnaire, many used words such as 'milkiness', 'creaminess', 'richness', 'smoothness', 'sweetness' and 'melting in the mouth' when describing the chocolates themselves, suggesting that this vocabulary can be used with confidence.

4.3.3 Sensory Attributes

Next, participants were asked to rate the chocolate according to eight sensory attributes: melting-in-the-mouth, creaminess, richness, thickness, milkiness, smoothness and sweetness. Participants were given a glossary of terms to standardise the questions, and ensure that all the participants understood the sensory words given (see Chapter 2). Each attribute was asked on a visual analogue scale (VAS), and as Just About Right (JAR) questions (see Chapter 2 for a description).

For the visual analogue scale questions, trends toward significant differences (i.e. p values $< .1$) were observed for:

- melting rate, with reduced-fat chocolate melting quicker ($\bar{\chi} = 44.76$, $\sigma = 20.3$) than milk chocolate ($\bar{\chi} = 40.78$, $\sigma = 18.3$) [$F(1, 86) = 3.05$, $p = .085$];
- creaminess, with Milk Chocolate being rated higher ($\bar{\chi} = 59.06$, $\sigma = 18.7$) than Reduced-fat Milk Chocolate ($\bar{\chi} = 54.83$, $\sigma = 19.6$) [$F(1, 86) = 2.48$, $p = .119$];
- smoothness, with milk chocolate being rated as smoother ($\bar{\chi} = 71.6$, $\sigma = 21.3$) than reduced fat milk chocolate ($\bar{\chi} = 67.1$, $\sigma = 23.3$) [$F(1, 86) = 2.714$, $p = .103$].

No significant effects of labelling condition, or trends, were observed for richness, thick mouthfeel, milkiness or sweetness. Furthermore, for the JAR questions, significant differences or trends were not observed for any of the sensory attributes. Figure 4.2 shows the percentage of consumers who rated the two chocolates from ‘much too much’ to ‘not at all enough’ according to each of the sensory attributes.

Very little difference in the intensity of each of the attributes is observed between the 'Milk Chocolate' and the 'Reduced-fat Milk Chocolate'.

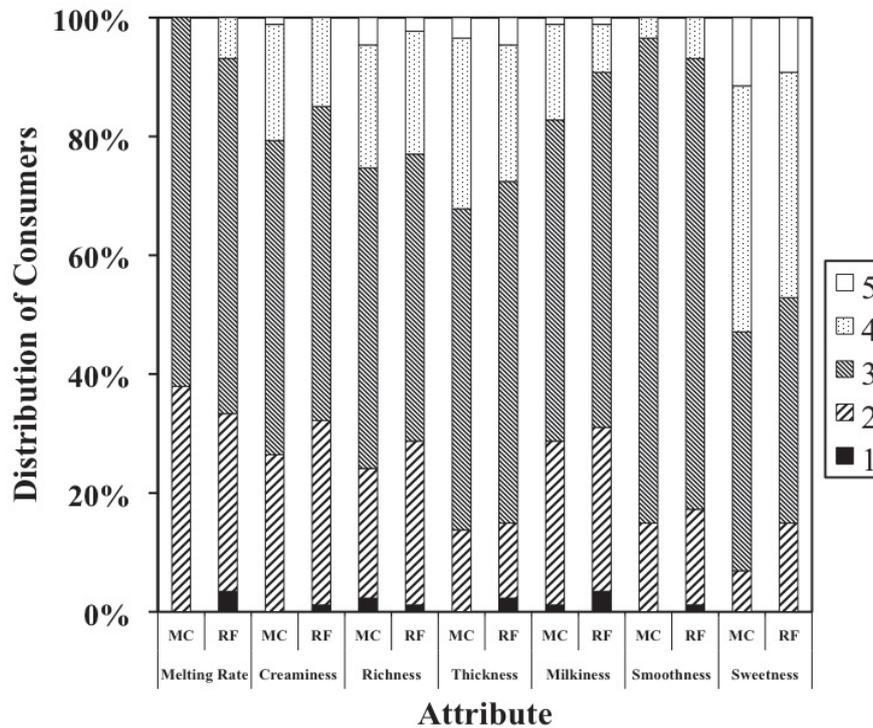


Fig. 4.2. Distribution of JAR scores for each attribute, where 1 = much too much, 2 = slightly too much, 3 = just about right, 4 = not quite enough, 5 = not at all enough. 'MC' refers to the labelling condition 'Milk Chocolate', and 'RF' refers to the labelling condition 'Reduced-fat Milk Chocolate'.

4.3.4 Individual Differences

As a significant effect of label was observed for expected liking, further analyses examined the interaction between this variable and individual differences (interactions between ratings and individual differences are not described if the finding was not significant).

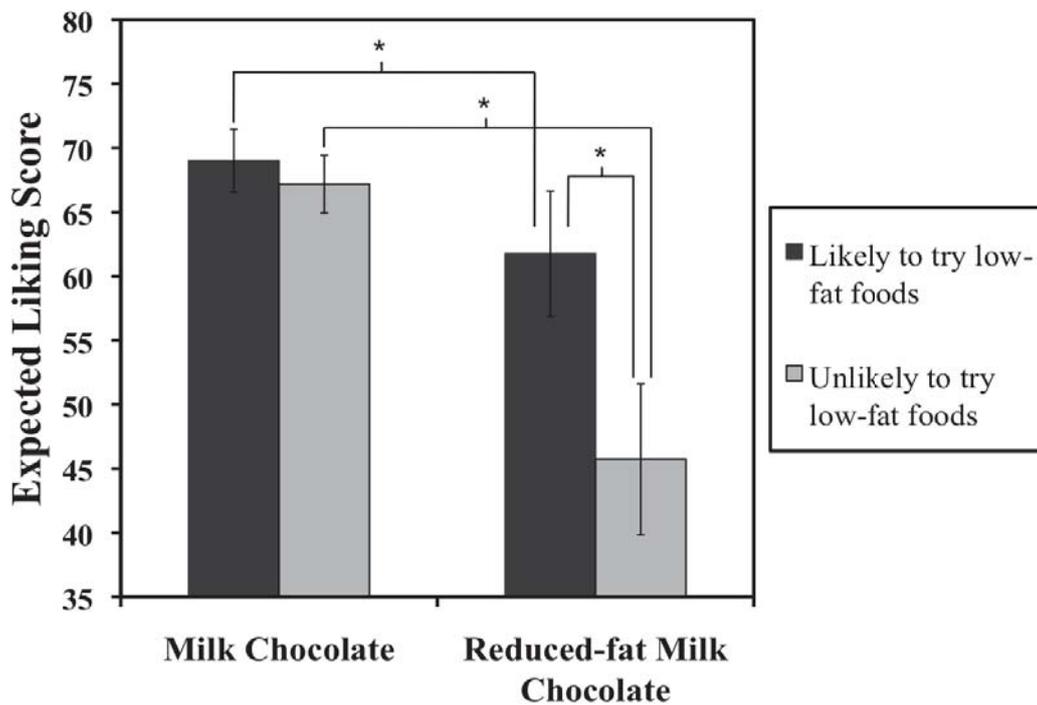


Fig. 4.3. Ratings of expected liking of chocolate samples labelled 'Milk Chocolate' and 'Reduced-fat Milk Chocolate' for those participants who said they were likely or unlikely to try low-fat foods. Data are means, and error bars represent one standard error of the mean. '*' denotes a significance value ≤ 0.05 .

A significant interaction between expected liking and willingness to try low-fat foods [$F(1,87) = 5.601, p = .020$] was observed (see Fig. 4.3). Firstly, in order to discover whether expected liking differed across labelling conditions, analysis was conducted separately on the two groups: those likely and those unlikely to try low fat foods. As can be seen in Figure 4.3, significant differences in expected liking scores in the two labelling conditions were observed for both those likely to try low fat foods [$F(1,67) = 10.7, p = .002$], and those unlikely to try them [$F(1,11) = 9.57, p = .01$]. This may suggest that chocolate is different from other low-fat products on the market, as even those who are normally willing to consume low-fat foods expected to like the 'reduced-fat' chocolate less than the standard chocolate. This may also be a result of

the experience that low-fat foods taste inferior to their higher fat equivalents. Independent samples t-tests were conducted to compare expected liking scores for the two groups, separately for the two labelling conditions. Although for 'Milk Chocolate' no significant difference in expected liking scores was observed between those likely to try low fat foods ($\bar{\chi} = 69.01, \sigma = 20.12$) and those unwilling to try low fat foods [$\bar{\chi} = 67.18, \sigma = 16.22; t(85) = .297, p = .767$], for 'Reduced-fat Milk Chocolate' a significant difference was observed between the two groups [$\bar{\chi} = 61.76, \sigma = 18.35$ & $\bar{\chi} = 45.72, \sigma = 19.49$ respectively; $t(78) = 2.767, p = .007$], with those participants with general unwillingness to try low-fat foods rating expected liking of 'Reduced-fat Milk Chocolate' significantly lower than those participants more open to low-fat foods. This indicates a greater negativity from participants unlikely to normally try low-fat foods.

Further analysis was conducted for expected liking and external eating scores, i.e. eating in response to external cues such as the sight, smell or taste of food, as measured using the DEBQ (van Strien *et al.*, 1986). Average expected liking scores for those with below median and above median external eating scores can be seen in Figure 4.4. Although a significant difference between expecting liking scores for 'Milk Chocolate' and the 'Reduced-fat Milk Chocolate' was not observed for those people with low levels of external eating [$F(1,36) = 2.278, p = 0.14$], a significant difference was observed for those with high levels of external eating [$F(1,49) = 21.09, p = < .0005$]. External eaters have elevated responsiveness to food-related cues in the immediate environment (van Strien *et al.*, 2009). This is thought to be a result of external eaters being aroused by, and getting pleasure from food, and the desire and

anticipation of eating it. It is thought that these people would not expect to like the ‘reduced-fat’ chocolate, as it would not excite them to the same extent as a full-fat equivalent. Independent samples t-tests indicated that there were no significant differences in expected liking between those with low and high levels of external eating for either the ‘Milk Chocolate’ [$\bar{\chi} = 64.57$, $\sigma = 19.82$ and $\bar{\chi} = 69.99$, $\sigma = 18.96$; $t(85) = -1.29$, $p = .2$] or for ‘Reduced-fat Milk Chocolate’ [$\bar{\chi} = 59.35$, $\sigma = 19.87$ and $\bar{\chi} = 58.54$, $\sigma = 18.16$; $t(85) = .197$, $p = .845$].

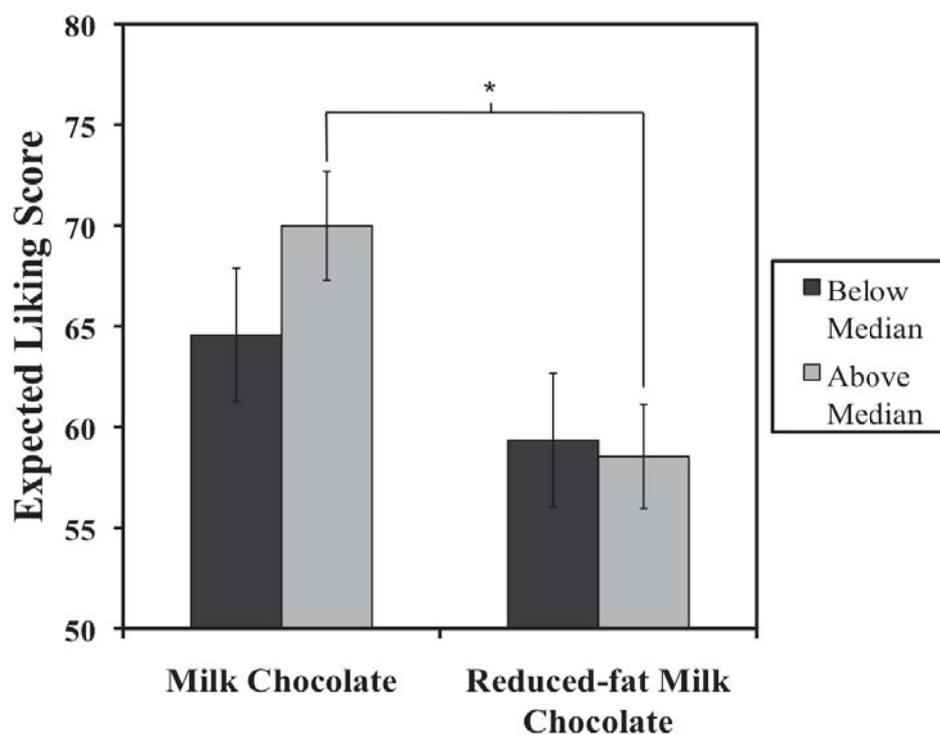


Fig. 4.4. Rated scores for expected liking of chocolate samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’, for participants with below median, and above median levels of external eating, as measured using the DEBQ (van Strien *et al.*, 1986). Data are means, and error bars represent one standard error of the mean. ‘*’ denotes a significance value ≤ 0.05 .

Although individual differences (i.e. willingness to try low-fat foods, and external eating) had an effect on expected liking score, no significant interactions, or trends

were observed for individual differences and actual liking. This is surprising as other researchers find that individual differences affect liking scores following tasting, for example Westcombe and Wardle (1997) report that health concern affected the pleasantness ratings of cheese.

4.3.5 Expected consumption amount and individual differences

Next, ratings of both expected normal consumption, and expected unrestricted consumption were analysed; no significant differences were observed for either. Although a significant main effect was not observed for normal consumption amount, a significant interaction was seen for normal consumption and ambivalence [$F(1,85) = 5.254, p = .024$] (see Fig. 4.5). Ambivalence refers to the existence of both positive and negative beliefs about an object, and is associated with conflicts between the sensory appeal of sweet and fatty foods, and their implication for health and body image. Ambivalence was measured using the chocolate consumption questionnaire (Sparks *et al.*, 2001; see Appendix 10). The two ambivalence groups (separated by a median split) were analysed separately.

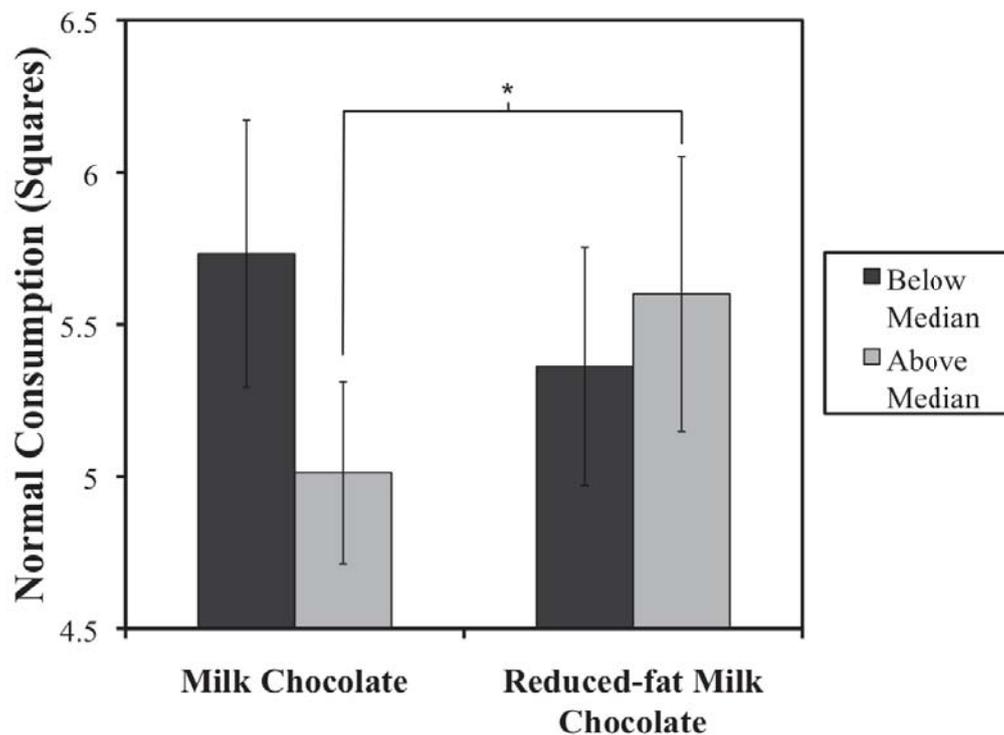


Fig. 4.5. Ratings of expected normal consumption amount (in squares of chocolate) of samples labelled 'Milk Chocolate' and 'Reduced-fat Milk Chocolate' for participants below, and above, the median ambivalence, as measured using the chocolate consumption questionnaire (Sparks *et al.*, 2001). Data are means, and error bars represent one standard error of the mean. '*' denotes a significance value ≤ 0.05 .

Although a significant difference was not observed between the two labelling conditions for those participants with a lower level of ambivalence [$F(1,46) = 1.42, p = .24$], a significant difference between the chocolates was observed for participants with a higher level of ambivalence [$F(1,39) = 4.81, p = .034$], who anticipated that they would consume significantly more of the 'Reduced-fat Milk Chocolate' than the standard chocolate. This would be expected, and may be a result of decreased conflict in feelings when consuming reduced-fat products as the implications for health and body concern are reduced (Sparks *et al.*, 2001), resulting in the belief that a higher quantity can be consumed. Differences in projected consumption were not observed for those with lower than median ambivalence, possibly because the incidence of

mixed feelings when consuming chocolate is lower, so a reduced-fat chocolate does not offer as much of an advantage to these consumers.

Although no significant main effect was observed for anticipated unrestricted consumption, a significant interaction was seen for unrestricted consumption and BMI [$F(1,85) = 5.011, p = .028$] (see Fig. 4.6).

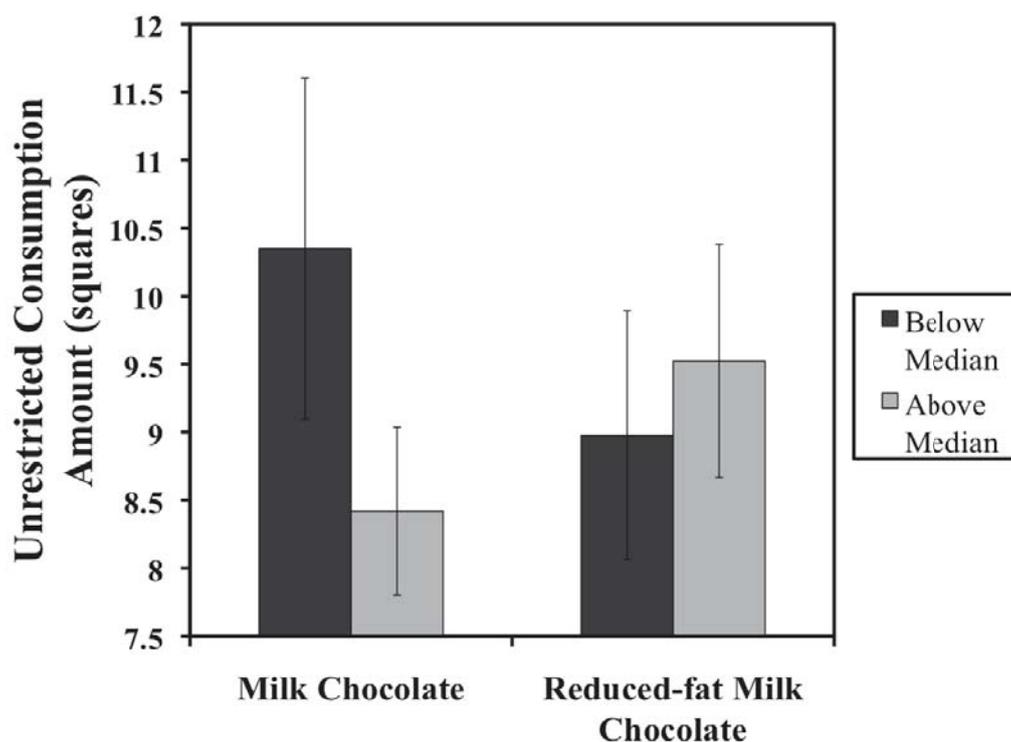


Fig. 4.6. Ratings of expected unrestricted consumption amount (in squares of chocolate) of samples labelled 'Milk Chocolate' and 'Reduced-fat Milk Chocolate' for participants below, and above, the median BMI. Data are means, and error bars represent one standard error of the mean.

The two BMI groups (i.e. above median and below median BMI) were analysed separately. No significant differences were observed between the two labelling conditions for either those with a lower BMI [$F(1,42) = 2.574, p = .116$], or those with a higher BMI [$F(1,43) = 2.453, p = .125$]. Furthermore, an independent samples

t-test indicated no significant difference between those with lower BMIs ($\bar{\chi} = 10.35$, $\sigma = 8.12$), or those with higher BMIs ($\bar{\chi} = 8.42$, $\sigma = 4.05$) for 'Milk Chocolate' [$t(85) = 1.406$, $p = .163$], or for 'Reduced-fat Milk Chocolate' [$\bar{\chi} = 8.97$, $\sigma = 5.93$ and $\bar{\chi} = 9.52$, $\sigma = 5.62$, respectively; $t(85) = -.441$, $p = .66$]. Whilst none of the individual t-tests showed a significant difference, Figure 4.6 suggests that the overall significance is being driven by high unrestricted consumption of 'Milk Chocolate' by those with a lower than median BMI. It is likely that those with a lower BMI are less concerned with their weight, and so do not see the benefits of consuming a reduced-fat chocolate. Instead it appears that they would like to gain maximum pleasure from its consumption, and so would prefer to eat more of the standard chocolate and not restrict their consumption. There also may be a slight increase in consumption of the 'Reduced-fat Milk Chocolate' by those with an above median BMI. This would be consistent with literature, such as Wansink and Chandon (2006) who observed that overweight consumers ate more snacks labelled as low fat than normal weight consumers. However, here this is not a statistically significant finding.

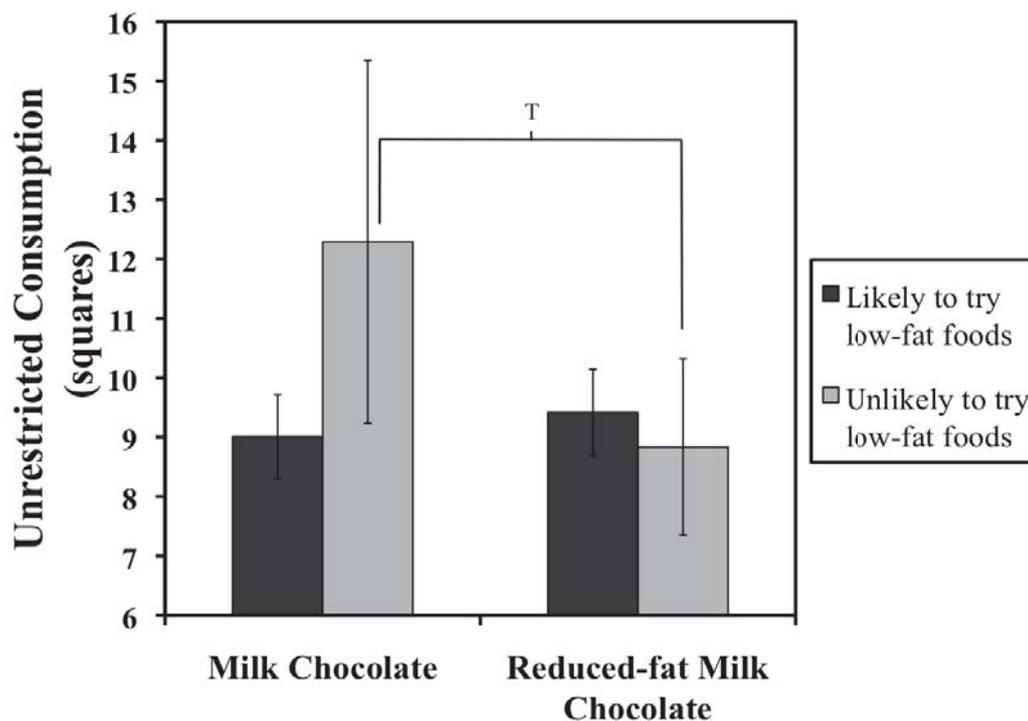


Fig. 4.7. Ratings of expected unrestricted consumption amount (in squares of chocolate) of samples labelled 'Milk Chocolate' and 'Reduced-fat Milk Chocolate' for participants who are likely, and unlikely to try low-fat foods. Data are means, and error bars represent one standard error of the mean. 'T' denotes a trend towards significance at a value ≤ 0.1 .

A significant interaction was also observed for unrestricted consumption and willingness to try low-fat foods [$F(1,78) = 5.646, p = .020$] (see Fig. 4.7). Post hoc analysis indicated that although there was no significant difference between scores of unrestricted consumption for those willing to try low fat foods [$F(1,67) = .462, p = .499$], there was a trend towards significance between the two labelling conditions for those unlikely to try low fat foods [$F(1,11) = 3.59, p = .085$]. Furthermore, an independent samples t-test indicated that there was no significant difference between those willing ($\bar{x} = 9.01, \sigma = 5.79$) and unwilling ($\bar{x} = 12.29, \sigma = 5.79$) to try low-fat foods for 'Milk Chocolate' [$t(12) = -1.09, p = .296$]. There was also no significant difference between the two groups for 'Reduced-fat Milk

Chocolate' [$\bar{\chi} = 9.42$, $\sigma = 5.95$ and $\bar{\chi} = 8.83$, $\sigma = 4.93$ respectively; $t(78) = .322$, $p = .748$]. Whilst those likely to try low-fat foods had similar consumption amounts for the two chocolates, for participants unlikely to try low-fat foods there was a trend towards higher consumption of the standard chocolate than the 'reduced-fat' chocolate. Expected consumption of the standard chocolate was greater for this group, possibly because they are not only unlikely to consume low-fat foods, but enjoy consuming high fat foods. Interestingly, being likely to try low-fat foods normally did not affect consumption amount, possibly because chocolate is thought to be within a different product category to other low-fat foods experienced, or because low-fat foods are chosen by this group to aid healthy living and a desired body image, and not to allow increased consumption of those foods.

4.3.6 Willingness to pay and individual differences

Next, the effect that labelling condition has on price was considered. Participants were first asked whether they would be willing to purchase the product, and if so, were asked for how much (using standard chocolate prices as a reference; see Chapter 2 for details). A significant effect was also not observed for price, although a significant interaction was observed for price and BMI [$F(1,71) = 4.130$, $p = .046$]. The two BMI groups (i.e. below and above median BMI) were analysed separately, finding that there was not a significance difference in price between the two labelling conditions for those with a lower than median BMI [$F(1,34) = 1.508$, $p = .228$]. No significant difference was observed for those with a higher than median BMI [$F(1,37) = 2.779$, $p = .104$]. Furthermore, an independent samples t-test indicated that there was no significant difference between the low and high BMI groups for price of

‘Milk Chocolate’ [$\bar{\chi} = 44.09$, $\sigma = 11.6$ and $\bar{\chi} = 43.93$, $\sigma = 15.09$ respectively; $t(76) = .055$, $p = .957$].

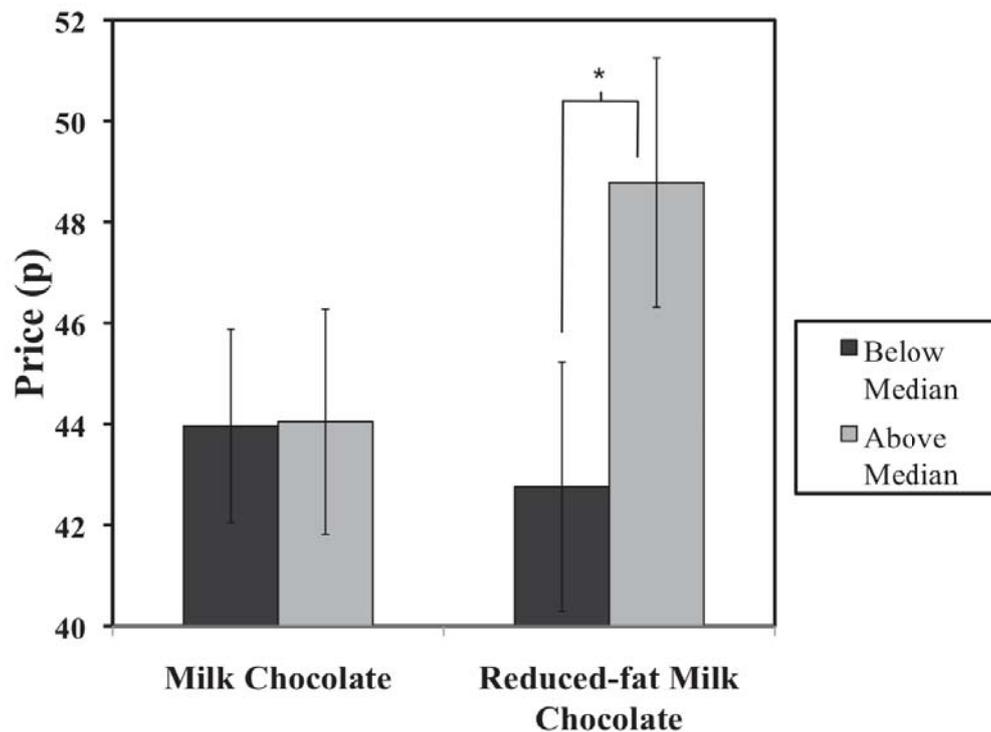


Fig. 4.8. Ratings of price of samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’ for participants below, and above, the median BMI. Data are means, and error bars represent one standard error of the mean. ‘*’ denotes a significance value ≤ 0.05 .

However, a significant difference was observed between the low and high BMI groups for ‘Reduced-fat Milk Chocolate’ [$\bar{\chi} = 42.03$, $\sigma = 14.36$ and $\bar{\chi} = 49.26$, $\sigma = 16.06$ respectively; $t(76) = -2.087$, $p = .04$], with participants with higher BMIs willing to pay significantly more for this chocolate than participants with lower BMIs (see Fig. 4.8). This may indicate that those with a higher BMI are concerned with their weight, and so are willing to pay more for this product.

A significant interaction was also observed for price and normal chocolate consumption [$F(1,71) = 5.961, p = .017$].

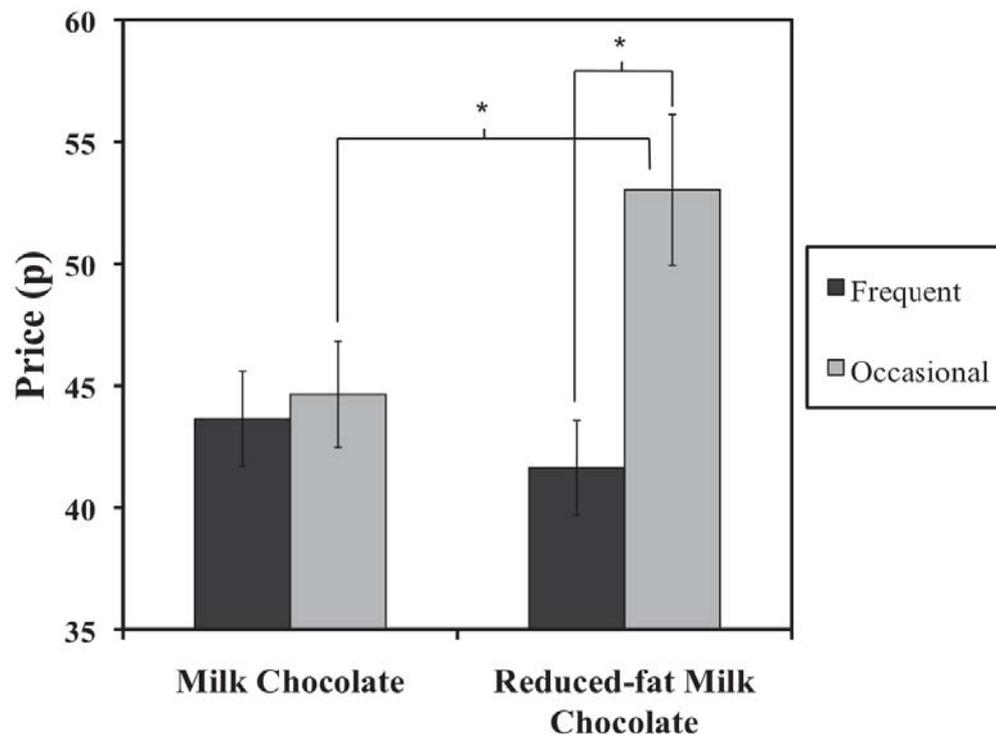


Fig. 4.9. Ratings of price for chocolate samples labelled ‘Milk Chocolate’ and ‘Reduced-fat Milk Chocolate’, for frequent and occasional consumers of chocolate. Data are means, and error bars represent one standard error of the mean. ‘*’ denotes a significance value ≤ 0.05 .

The two chocolate consumption groups were analysed separately, and while no significant difference between label condition was seen for frequent consumers of chocolate [$F(1,46) = .753, p = .39$], a significant difference in price was seen for consumers who normally consume chocolate once a week or less [$F(1,25) = 5.003, p = .034$]. Furthermore, an independent samples t-test indicated that while there were no significant difference in price of ‘Milk Chocolate’ between frequent and occasional consumers of chocolate [$\bar{\chi} = 43.4, \sigma = 14.07$ and $\bar{\chi} = 45.15, \sigma = 12.26$ respectively; $t(76) = -.544, p = .588$], there was a significant difference in price between the groups

for 'Reduced-fat Milk Chocolate' [$\bar{\chi} = 42.23$, $\sigma = 14.27$ and $\bar{\chi} = 51.9$, $\sigma = 16.12$ respectively; $t(76) = -2.75$, $p = .007$] (see Fig. 4.9). Although it is not clear why this group eats chocolate only occasionally, it is possible that this is a result of its high fat content, and so a reduced-fat chocolate would be preferable and worthy of a higher price tag. Conversely, these occasional consumers may normally purchase higher quality, more expensive chocolates and so are used to, or happy to, spend more on it.

Finally, a trend towards significance was observed for price and restriction [$F(1,71) = 2.833$, $p = .097$]. Low restriction and high restriction were analysed separately, finding no significant difference between label conditions for those with low restriction [$F(1,35) = .366$, $p = .549$], and a trend towards significance for those with high restriction [$F(1,36) = 3.618$, $p = .065$]. An independent samples t-test indicated that there were no significant difference between price of 'Milk Chocolate' for those with low restriction ($\bar{\chi} = 45.58$, $\sigma = 11.9$) and high restriction ($\bar{\chi} = 42.51$, $\sigma = 14.72$) [$t(76) = 1.009$, $p = .316$]. There was also no significant difference between the two groups for 'Reduced-fat Milk Chocolate' [$\bar{\chi} = 44.13$, $\sigma = 15.8$ and $\bar{\chi} = 47.53$, $\sigma = 15.47$ respectively; $t(76) = -.961$, $p = .34$] (see Fig. 4.10). It has been suggested that people with higher levels of cognitive dietary restraint are more likely to choose reduced-calorie or reduced-fat foods (Rideout *et al.*, 2004). Being reduced in fat might increase the attractiveness of this normally forbidden food for these consumers, deserving of a higher price tag.

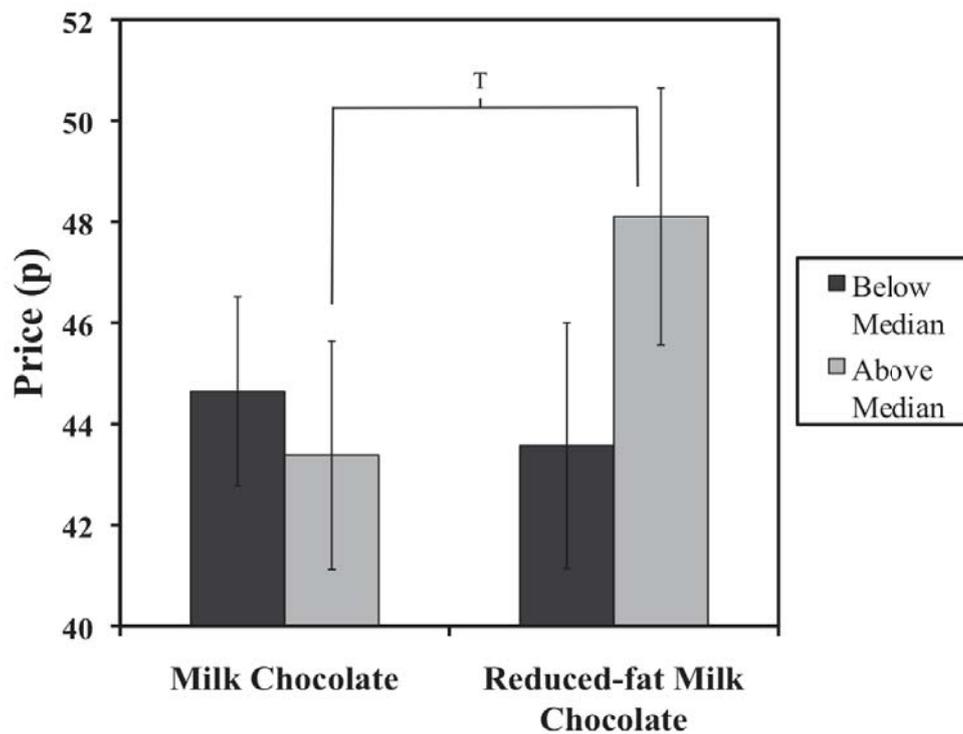


Fig. 4.10. Ratings of price of samples labelled 'Milk Chocolate' and 'Reduced-fat Milk Chocolate' for participants below, and above, the median restriction. Data are means, and error bars represent one standard error of the mean. 'T' denotes a trend towards significance at a value ≤ 0.1 .

4.4 Discussion

The effect that information about fat content has on expected liking, actual liking, ratings of sensory attributes, consumption amount, purchase intent and price perception of chocolate has been investigated. The hypotheses set up for this investigation were presented in section 4.2. The following hypotheses were supported by the results:

H₁. The label condition had an impact on ratings of expected liking, with 'Reduced-fat Milk Chocolate' being rated lower than 'Milk Chocolate' (see Fig. 4.1);

H₂. Actual liking of the ‘Reduced-fat Milk Chocolate’ was higher than expected liking, as a result of positive-disconfirmation (see Fig. 4.1);

H₃. When the chocolate is labelled ‘Milk Chocolate’ the expected and actual hedonic ratings were similar (see Fig. 4.1);

H₆. A person’s eating style and health concerns affected their ratings of the chocolate (see Fig. 4.3, 4.4, 4.5 & 4.10);

H₇. BMI had an effect on expected consumption amount (see Fig. 4.6).

However, the following hypothesis were not upheld:

H₄. Sensory attributes perceived as being related to fat content in chocolate were not rated as lower, or higher, when the chocolate was labelled as ‘Reduced-fat Milk Chocolate’ than when it was labelled ‘Milk Chocolate’ (see Fig. 4.2);

H₅. When the chocolate is labelled as reduced-fat, a higher quantity would not be expected to be consumed.

4.4.1 Liking

In summary, an effect of labelling condition was observed for expected liking, with lower expectations of liking for ‘Reduced-fat Milk Chocolate’, although no difference in scores was observed for actual liking (see Fig. 4.1). Similar observations are cited in the literature, where a reduced-fat version was expected to be less pleasant (Kähkönen & Tuorila, 1998). It is unclear in this case whether this is due to i) participants’ prior experience of other low-fat foods, and their associated inferior

tastes, or ii) an expected loss of pleasure because the reduced-fat chocolate would no longer be indulgent. This may be of concern to those aiming to manufacture a low-fat chocolate, as consumers may be less willing to buy the product due to reduced expectations. However, purchase intent is not completely driven by expectations about taste, but is also determined by the health benefits gained by purchasing and consuming the product.

A significant effect of label was not observed for actual liking, indicating that experience during consumption plays a role in judgements, and subsequent ratings. Assuming that a genuine reduced-fat chocolate could be formulated to be similar in taste to a standard version, ratings of liking should not be significantly lower just because consumers are aware that it is reduced-fat. This is a positive outcome: if a reduced-fat chocolate can be produced, sensory attributes can be matched, and consumers can be encouraged to purchase the product, actual liking should not be affected by the knowledge that the product is reduced in fat. Furthermore, a difference in ratings of expected and actual liking was observed for reduced-fat chocolate, with higher ratings for actual liking. This indicates positive disconfirmation, with a large discrepancy between expected and actual experience, which has been reported to lead to consumer satisfaction and repeated use (Deliza & MacFie, 1996).

Furthermore, an interaction between expected liking and general willingness to try low-fat foods was observed (see Fig. 4.3), with both groups of participants expecting to like the reduced-fat chocolate less than the standard chocolate, but with greater negativity from those people who are normally unlikely to try low-fat foods. Additionally, a trend was seen for the interaction between expected liking and

external eating (see Fig. 4.4), with those with higher than median levels of external eating expecting to like the ‘reduced-fat’ chocolate less than the standard chocolate, possibly as a result of a loss of excitement and expected pleasure.

4.4.2 Sensory attributes

Sensory attributes were measured using VAS questions and JAR questions. Significant results were not observed for any of the attributes, although a number of trends were observed for melting rate, creaminess and smoothness (see Section 4.3.3). Reduced-fat information has previously been found to lead to lower expected melting rate in chocolate (Kähkönen *et al.*, 1999), and decreased melting-rate ratings during tasting (Kähkönen, 2000). However, the results presented here suggest an opposite relationship, with quicker melting, not a decreased melting rate. Given the lack of significant results for actual liking, it appears that actual experience played a greater role than labelling condition after tasting.

4.4.3 Consumption Amount

When considering anticipated, or projected consumption, no significant differences were observed for normal consumption, or for unrestricted consumption (see Section 4.3.5). A concern for the production of a reduced-fat chocolate has been the knowledge that the product is reduced in fat may result in increased energy intake, both in terms of the product itself, and also during subsequent meals (Shide & Rolls, 1995). This result suggests that, in general, energy intake would not differ, and the reduced-fat chocolate would not be eaten to excess, which is a positive result for

obesity prevention and reduction. However, this result may result from a limitation of the method used in this work. Participants were asked to self-report envisaged consumption of the two chocolates, both during a normal occasion, and if they were not going to restrict their behaviour. It is questionable whether this method is predictive of actual behaviour. Not only does it require self-reporting (where observation would be preferable), but it is also a prediction of future behaviour (online measurements may be more accurate). It would be interesting to explore *ad libitum* consumption to determine whether the findings presented here are accurate, to determine whether a reduced-fat chocolate would be an effective means of reducing energy intake.

Despite these overall consumption findings, some interactions were observed between consumption amount and participant characteristics. A significant interaction was observed for normal consumption and scores of ambivalence (see Fig. 4.5), with participants with higher levels of ambivalence predicting that they would consume significantly more reduced-fat chocolate during a normal sitting than they would consume a standard chocolate. This is consistent with the idea that ambivalence is associated with conflict between the sensory appeal of sweet, fatty foods and concern of health and body image (Cartwright & Stritzke, 2008); people with high levels of ambivalence should experience less conflict with reduced-fat products, and thus allow themselves to consume greater quantities.

A significant interaction between unrestricted consumption and BMI was observed (see Fig. 4.6). It appeared that whilst those with a lower than median BMI believed they would consume less 'Reduced-fat Milk Chocolate' than 'Milk Chocolate', those

with a higher than median BMI thought they would consume more of the ‘reduced-fat’ bar than the standard bar. Those with a higher BMI are possibly more concerned with their weight, and see consumption of a reduced-fat chocolate as a route to consuming chocolate without negative health or weight related outcomes. Alternatively, this may also be a result of ‘impression management’, in that those with a higher BMI believe that they should be consuming less of the standard chocolate as it is higher in fat, so state that they would consume less regardless of whether this is an accurate prediction of their true behaviour. An obstacle in obesity research is the inaccuracy of food intake data; obese people tend to provide biased food intake records (i.e. food diaries), and eat more than they claim to, especially when regarding dietary fat (Blundell, 2000).

An interaction was also observed for unrestricted consumption and general willingness to try low-fat foods (see Fig. 4.7), with those unlikely to try low-fat foods expecting to consume less of the reduced-fat chocolate when not restricting consumption.

4.4.4 Willingness to pay

Although an overall effect of labelling condition on price was not observed (see Section 4.3.6), a significant interaction between price and BMI was observed (see Fig. 4.8), with a higher price being given for ‘Reduced-fat Milk Chocolate’ for those with a higher BMI. Furthermore, those with a higher BMI were willing to pay more for this chocolate than consumers with a lower BMI. A significant interaction was also observed for price and normal chocolate consumption (see Fig 4.9). Whilst no

difference between labelling condition was seen for frequent consumers of chocolate, a difference was seen for consumers who consume chocolate once a week or less. This group would spend more on the 'Reduced-fat Milk Chocolate' than the standard chocolate. Finally, a trend towards significance was observed for price and restriction (see Fig. 4.10), with those with high restriction being willing to spend more on the 'reduced-fat' chocolate than on the standard chocolate.

4.4.5 Additional Individual Differences

Surprisingly, no effect of gender was observed. It was expected that gender would have an impact on liking, as women report liking or craving chocolate more than men (Rozin *et al.*, 1991), and consume more energy-reduced and fat-reduced products than men do (Fagerli & Wandel, 1999). Women are likely to be more open to consuming a reduced-fat chocolate, and are perhaps more likely to be calorie conscious (Kiefer *et al.*, 2005). It was also expected that age may have an effect upon liking, as not only do reports of cravings for sweet foods decline with age (Pelchat, 1997), but snack-related comfort foods made older people feel less unhealthy than they made younger people feel (Wansink *et al.*, 2003). Finally, no effect of emotional eating score was observed. Emotional eating has also been related to a higher consumption of sweet foods in both men and women (Konttinen *et al.*, 2010).

4.5 Conclusions and Future Work

In summary, this study provides evidence that labelling a chocolate as 'reduced-fat' affects expected liking, but has little effect on actual liking or ratings of sensory attributes. Furthermore, individual differences (such as BMI, external eating,

restriction, ambivalence, and opinions toward low-fat foods) are important determinants of expectations and eating attitudes. This information can provide insight into the mechanisms by which information can be communicated to consumers (and specific cohorts of consumers) for successful marketing of reduced-fat milk chocolate.

Future work should consider other labelling options, such as more realistic chocolate packaging concepts that maximise liking (this will be discussed in Chapter 5). Furthermore, the effect of labelling condition on actual consumption of the chocolates, and on consumption during subsequent meals should be considered, to determine whether *ad libitum* consumption amount results from expectations about the foods energy density (feeling of fullness, or feelings of guilt), or the post-ingestive experience (for example, the energy absorbed by the body following consumption). Once a reduced-fat chocolate has been produced it would be interesting to consider the interaction between labelling condition and energy density or sensory acceptance on liking and consumption.

5. Exploring packaging of reduced-fat chocolate

5.1 Introduction

The aim of the work presented in this chapter was to gain a better understanding of current chocolate packaging, and determine aspects of packaging that highlight how healthy, or unhealthy, the bar is. Furthermore, the intention was to explore approaches to packaging a reduced-fat chocolate bar, and to consider differences in opinion between male and female consumers. This was achieved using a) two focus groups b) a rank-rating task. Focus groups were conducted to explore chocolate packaging and generate ideas for the packaging of a reduced-fat chocolate bar. The rank-rating task was then used to measure the relative importance of each of the attributes recognised during the focus groups on expected liking, to discover the ideal packaging combination.

Information presented on packaging is critical for the success of the product. Packaging protects contents from contamination and spoilage, makes it easier to transport and store goods, and provides a uniform measure of contents (Robertson, 2005). Packaging is also used to communicate information to the consumer, and enable the consumer to recognise the product amongst similar products (Robertson, 2005). The majority of purchasing decisions are made at the point of sale (Ampuero & Vila, 2006), so packaging should create differentiation and identity, serving as a “vehicle for promotion” (McDaniel & Baker, 1977). Thus, consumers are exposed to packages in the same way that they are exposed to advertisements. Product packaging also plays an important role in consumer product perception and acceptance, for

example, background colour and information had a significant effect on all rated attributes and upon expected liking (Deliza, MacFie & Hedderley, 2003).

A combination of both qualitative and quantitative methods has been used by other researchers (Raz *et al.*, 2008), who used focus groups and conjoint analysis to create innovative food products through sensory marketing. Similar methods have been utilised in this work. Group discussion was used to explore current successful packaging options and generate package ideas for a reduced-fat chocolate. Information gained during the focus groups was used to determine the attributes, and levels of these attributes, for investigation during the rank-rating task. Individual differences, specifically gender related, were also considered. Studying individual differences may help identify a possible target market. It is likely that differences in opinions will be observed as not only do women report liking and craving chocolate more than men (Rozin *et al.*, 1991), but they are more positive towards low-fat foods (Solheim & Lawless, 1996), consume more energy-reduced and fat-reduced products (Fagerli & Wandel, 1999), and are more likely to be calorie conscious (Kiefer *et al.*, 2005). This may affect perception towards a reduced-fat chocolate, and preference towards packaging type, and packaging information. Thus, prior to running the focus groups two hypotheses were made:

H₁. Packaging attributes will differentially influence perceptions of healthiness;

H₂. Differences in packaging preferences will be observed for male and female participants.

This study was also exploratory in that it sought to identify ways in which packaging can promote a ‘healthy indulgent’ message for a chocolate bar, with the aim of increasing acceptance.

Specific details of the experimental design and methods used can be found in Chapter 2.

5.2 Focus Groups

5.2.1 Results

5.2.1.1 Female Focus Group

During the female focus groups the participants were given a selection of 24 photographs of chocolate bars (see Appendix 14). When asked to separate the bars according to healthiness, the female group indicated a number of important factors. When asked to explain why bars were placed in the ‘unhealthy’ pile, reasons included the name, bright, dark colours, the size of the bar (i.e. square bars rather than long, thin bars), the font and photographic image on the packaging:

“...the names are different... there are two [in the unhealthy pile] with ‘max’ on them or ‘chunky’, compared to ‘twirl’ and ‘ripple’ [in the healthy pile] which sounds a bit less fatty... It makes them sound bigger, or more substantial.”

“I think the more unhealthy ones are square and chunky, whereas the healthier ones are long and thin.”

Often, bars in the ‘unhealthy’ pile were thought to be targeted towards children, who are unconcerned with their health or weight, with the use of bubble writing or bright colours being used to attract attention. Similarly, the female participants thought that some of the bars were targeted towards male consumers, who are not as weight conscious as women.

When asked to explain reasons for placing photographs of the bars into the ‘healthier’ pile reasons included the name, the use of lighter colours, and the slim-line shape. Bars in this pile were thought to be more feminine:

“The healthier bars are more elegant, whereas the unhealthier bars are more childish.”

Additional reasons included information given on the package (i.e. ‘organic’, ‘Fairtrade’, and the percentage of cocoa or calorie content) or a logo. Knowledge that the chocolate bar is Fairtrade or organic indicated to the consumers that it is high quality, and gave emphasis to additional health claims:

“... organic... makes me think of healthy... it’s less likely to have rubbish in it... man-made or concocted type ingredients in it.”

“...Fairtrade sign... It’s about the quality of what is going in to it, rather than maybe what is actually in it... if they are good to the people they get their ingredients from then they care about the people buying it too... you wouldn’t feel as guilty buying it.”

The use of logos on the bars was heavily discussed, with the conclusion that even if the meaning of the logo is unknown, or ambiguous, if it looks official, it suggests that the company is accredited or approved, which instils trust:

“... it’s also got a little leaf on there which must mean natural or nature.”

“It’s got a logo on the bottom as well. I wouldn’t really know what that was, but it just feels like it is more trustworthy. It looks official.”

Participants were then asked to discuss how a new reduced-fat milk chocolate could be packaged. The female participants highlighted possible issues surrounding the introduction of a reduced-fat chocolate bar, including that consumers may not wish to advertise to others that they are on a diet, or being health conscious. Furthermore, although chocolate is often purchased as a gift, this product could not be bought for someone else, as they may be offended. These issues suggest a need for subtle packaging, with less emphasis on the products fat and calorie reduction, but more focus on taste and health benefits. Furthermore, the group suggested that packaging should include a positive, uplifting slogan. Participants also thought that colour was important, with the use of a light colour, accompanied by a light, airy sounding name. There was uncertainty surrounding the size of the bar, with opinions that it should be thin to emphasise its health properties, and reduce the calories even further, or that it should be sold as a bigger bar as it has already been reduced in fat and calories. All agreed that the bar should be elegant.

5.2.1.2 Male Focus Group

When the male group was asked to place the photographs into a pile of ‘unhealthier’ bars, reasons included the use of health facts (which were believed to be a result of ‘overcompensating’ for something else), an unattractive name, a picture of the bar, a childish font and a yellow colour:

“They seemed to be overcompensating with health facts, almost as if they miss out the fact that it is full of lard.”

“... the picture... it looks a bit disgusting!... looks stodgy inside.”

“The yellow colour is putting me off as well. It’s sort of fatty... I think that yellow makes you feel hungry.”

“We tended to put lighter coloured products in the healthier pile. This is dark, and it kind of signifies that there is something dark inside. That it’s denser. And richer.”

The male group also highlighted that the unhealthier bars tend to be marketed towards men:

“I think that the more unhealthy ones are probably marketed towards men. You’re a man, you can take the calories!”

Reasons for placing bars in the ‘healthier’ pile included the use of logos or association with Fairtrade or being organic, which emphasised the health claims by indicating quality:

“The little green symbol on the side. Whatever it says it is still a little green symbol... it’s nice.”

“The gold leaf in the corner is like a seal of approval, or a standard... I think that the writing looks quite refined to me... it’s sophisticated, and fits in with the gold leaf.”

“I like the tick. This is a chocolate bar that is good for me. I associate the tick with active lifestyle.”

This group also thought that paper packaging indicated that the company is concerned with the environment, and so in turn concerned about the welfare and health of its consumers:

“If the packaging is made out of paper it is better for the environment and better for you. It is made for healthier people.”

When asked to indicate possible packaging options for a reduced-fat chocolate, the male group thought that colour was important (and suggested light colours, such as lilac), as was size, with a bigger bar being preferential. The use of a logo was stressed, with suggestions of a tick, a chocolate heart, or a green symbol, all of which were thought to increase the persuasiveness of the health message. This group questioned

whether the reduced-fat information should be used, or emphasised, on the packaging. The male group believed that a reduced-fat chocolate bar would be for women, so suggested that the packaging should be feminine. A possible limitation exists for the introduction of a reduced-fat bar as men believe that this type of product is for women, who are more concerned with their weight. They feel they would risk embarrassment buying a woman's chocolate bar, as they are not supposed to care about their weight or health.

5.2.2 Overall Conclusions

To conclude, it was clear that packaging attributes influenced perceptions of healthiness (H_1 was upheld). The focus groups highlighted which aspects of the chocolate bar packaging indicate that the bar is more healthy or unhealthy, irrespective of previous knowledge of the bar. Furthermore, although many ideas were similar between the gender groups, both men and women believed that a reduced-fat bar would be aimed towards women, which may restrict its potential success. Thus, as hypothesised, differences in opinions were observed (H_2 was supported).

5.3 Rank – Rating Task

5.3.1 Packaging Concepts

Information gained from both the focus groups was used to produce 16 packaging concepts. A number of important factors were recognised during discussion sessions, resulting in four components being selected: colour; size / shape; slogan; logo.

5.3.1.1 Colour

Two colours were selected; the manipulations chosen were light blue and caramel. Blue was observed as a colour used for healthier bars, whilst yellows and caramels were seen as an indication of unhealthier, fattier bars. These are labelled ‘B’ (blue) and ‘C’ (caramel) throughout the chapter.

5.3.1.2 Size / shape

It was observed that the size and the shape of the bars was an indicator of healthiness. Participants were divided in opinion when discussing the size of a reduced-fat chocolate bar. Some would prefer a long, thin bar as it further emphasises the healthiness, whilst others thought a wider, chunky bar would be preferential as the bar is already reduced in calories, allowing for a greater quantity to be consumed. Thus, two manipulations were chosen: a long, thin bar and a wider, chunky bar. These are labelled ‘L’ (long) and ‘S’ (square) throughout the chapter.

5.3.1.3 Slogan

Two slogans were selected to reflect both a taste-oriented message (“*same smooth and creamy taste*”) and a pleasure-oriented tagline (“*enjoy a guiltless pleasure*”). Both the focus on taste, and a positive uplifting message were thought to be important; these slogans were thought to address this observation. They are labelled ‘T’ (taste oriented) and ‘P’ (pleasure oriented) throughout the chapter.

5.3.1.4 Logo

Logos were thought to increase the persuasiveness of health messages, so two were selected: a heart was chosen to represent ‘heart-healthiness’ (as a result of reduction in fat), as well as love of the product, whilst the tick was used to indicate healthiness, related to an active lifestyle, and making the ‘right’ choice. These are labelled ‘H’ (heart) and ‘T’ (tick) throughout the chapter.

Each of the four components had two levels, resulting in sixteen combinations. The sixteen packages, with details of the corresponding features, and codes used throughout the chapter, can be found in Appendix 19.

The bars were created using Adobe Photoshop CS4, where two stock images (taken from photographs of existing chocolate bars on the market), of the correct size and shape, were used as templates. All identifiable information was removed, and the new information added. Elegant fonts (Perpetua Titling, Lucinda Calligraphy and Zapfino) in gold lettering were selected, as was a light sounding name: ‘Delight’. Each of the bars was given a random 3-digit code. The images were printed on a photographic paper using a commercial photographic printer. All were 5 x 7 inches in size. Two examples of the images used in the study are shown in Figure 5.1.



Fig. 5.1 Two of the bars used for the rank-rating task. Left: a blue, square bar with the pleasure oriented slogan, and a heart logo (BSPH). Right: a caramel, long bar with a taste oriented slogan and a tick logo (CLTT). All 16 of the bars can be found in Appendix 19.

Participants were given all the packages in a random order, and asked to rank them according to how much they expected to like the product. They are then asked to give the least liked bar a score of one, the most liked a score of 100, and score all the other bars in between.

5.3.2 Hypotheses

It was hypothesised that differences in the liking scores of the packaging concepts would be observed, however it was unclear whether participants would prefer the components that most related to healthier bars, or unhealthier bars. Thus:

H₃. Liking of the packages will be significantly different, representing differences in the components;

H₄. Differences in liking will be observed for male and female participants, reflecting the observation that reduced-fat chocolate bars are aimed at women.

5.3.3 Results

A significant effect of packaging type was found [$F(15) = 10.38, p < .005$], indicating that significant differences exist between the packages. Post hoc analysis indicated where the differences in scores lie.

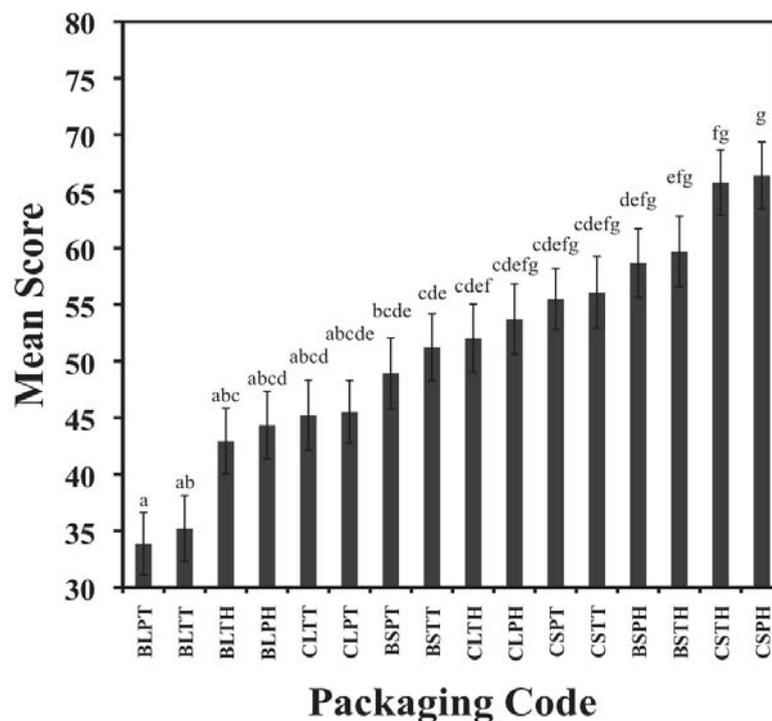


Fig. 5.2 Average liking scores for each of the packaging concepts (for all participants). The packages are coded, according to the components displayed on the packages: 1) Colour: 'B' = blues, 'C' = caramel; 2) Shape: 'L' = long, 'S' = square; 3) Slogan: 'T' = taste oriented, 'P' = pleasure oriented; 4) Logo: 'H' = heart, 'T' = tick; for example 'BLPT' is a blue, long, pleasure oriented package with a tick logo. All the packages can be found in Appendix 19. Lower case letters indicate where significant differences exist: when the same letters are observed, the two bars are not significantly different in liking scores; two bars with distinct letters are significantly different in liking scores.

The results in Figure 5.2 show a fairly linear ranking for liking. The average scores of all consumers indicate two distinct groups, where the letters 'a' and 'b' are not common to letters 'f' and 'g'. All the other groups overlap. The majority of the bars in

groups 'a' and 'b' are blue, long and contain the tick logo; these have the lowest scores of expected liking. The majority of the bars in groups 'f' and 'g' are caramel in colour, square and contain the heart logo. These bars have the highest expected liking scores.

ANOVAs were also conducted to compare scores for females and males separately. Significant effects were found for both females [$F(15) = 10.214, p < .005$], and males [$F(15) = 3.476, p < .005$]. Very similar patterns of liking were seen for male and female consumers. Post hoc analyses indicated where differences in liking scores lie; Table 5.1 and 5.2 show liking scores for each of the bars, and indicate which scores were significantly different.

Table 5.1 Mean rating scores for each of the packages for female consumers. All the packages can be found in Appendix 19. Lower case letters indicate where significant differences exist: when the same letters are observed, the two bars are not significantly different in liking scores; two bars with distinct letters are significantly different in liking scores.

BLTT	BLPT	CLTT	CLPT	BLTH	BLPH	BSTT	BSPT
32 ^a	33.42 ^a	40.28 ^{ab}	41.8 ^{ab}	46.92 ^{ab}	48.65 ^{ab}	50.68 ^{abc}	50.93 ^{abc}
CSTT	CLTH	CSPT	CLPH	BSTH	BSPH	CSTH	CSPH
51.47 ^{abc}	54.85 ^{bcd}	55.13 ^{bcd}	55.42 ^{bcd}	68.62 ^{cd}	70.20 ^{cd}	72.07 ^d	72.93 ^d

Table 5.2 Mean rating scores for each of the packages for male consumers. All the packages can be found in Appendix 19. Lower case letters indicate where significant differences exist: when the same letters are observed, the two bars are not significantly different in liking scores; two bars with distinct letters are significantly different in liking scores.

BLPT	BLTT	BLTH	BLPH	BSPT	BSPH	CLTH	CLPT
34.33 ^a	38.47 ^{ab}	38.98 ^{abc}	40.07 ^{abcd}	46.95 ^{abcd}	47.18 ^{abcd}	49.18 ^{abcd}	49.25 ^{abcd}
CLTT	BSTH	BSTT	CLPH	CSPT	CSTH	CSPH	CSTT
50.18 ^{abcd}	50.77 ^{abcd}	51.78 ^{abcd}	52.05 ^{abcd}	55.85 ^{bcd}	59.48 ^{cd}	59.9 ^d	60.68 ^d

The mean ratings for each of the components used on the packaging (see Table 5.3) were used to calculate part worths for all consumers, and separated results for male and female consumers (see Fig. 5.3). The part worth calculation (see Section 2.6.2.3 for the equation) is used to determine the relative importance of each of the attributes, along with combined effects, in order to determine the optimum combination of attributes for the highest expected liking score.

Table 5.3 Mean rating scores for each of the factors, for all consumers, females and males.

Attribute	Level	All Consumers	Female	Male
Colour	Blue	46.87	50.18	43.57
	Caramel	55.04	55.49	54.57
Shape	Long	44.12	44.17	44.06
	Square	57.79	61.50	54.07
Slogan	Pleasure	50.88	53.56	48.20
	Taste	51.07	52.11	49.94
Logo	Heart	55.46	61.21	49.70
	Tick	46.45	44.46	48.44

The component with the largest part worth for all consumers was the size and shape of the bar, where the square bar was preferred. Furthermore, this effect was stronger for female consumers. An effect was also seen for colour, where caramel was liked more than blue. Here the effect was stronger for male consumers. An effect was seen for logo, with consumers preferring the heart to the tick. However, here men and women differed drastically. Whilst male consumers showed only a small preference for the heart, for women the effect was far greater. Finally, for the slogan, no overall effect was observed, and analysed separately, again only a small effect was seen, although women preferred the pleasure oriented slogan, and men preferred the taste oriented tagline. Male participants showed less of a preference than female consumers for all of the attributes, except for colour.

Calculating part worths highlighted the ideal packaging combination, with the highest score for expected liking (and the maximum utility), which was a caramel, square bar with a heart logo, with either of the slogans (as no apparent preference was observed). This ideal packaging combination was the same for male and for female consumers.

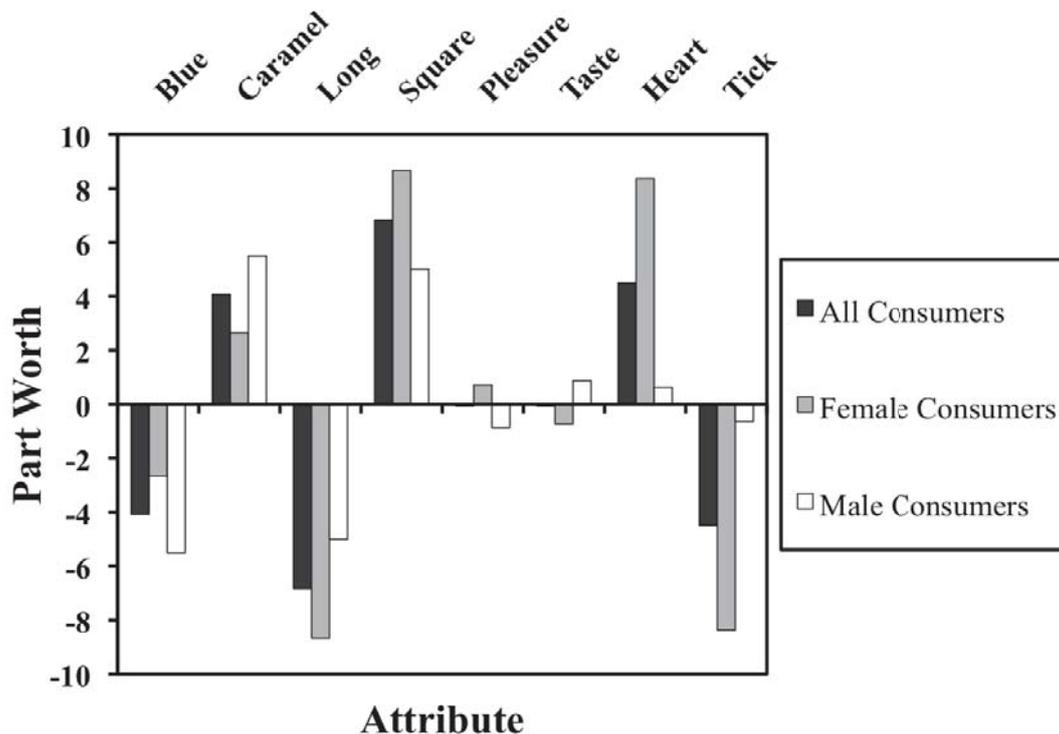


Fig. 5.3 Calculated part worths for each of the components (see Section 2.6.2.3 for the part worth equation).

Participants were also asked to explain their reasons for their choice of favourite, and least favourite bars. For females, some thought that the pleasure-oriented slogan was not correct, as the product was reduced-fat and not fat-free, so it is not a “guiltless pleasure”. It was thought that the taste-oriented slogan was preferential as it gave reassurance that the product will taste the same as regular chocolate. Some thought that the long bar was not appealing as it suggested a smaller volume of chocolate, which combined with a low fat content implies a controlled portion size. The caramel

colour was thought to be more warm, expensive, and luxurious, whilst the blue was thought to be garish, too bold and ‘trying too hard’ to grab one’s attention. The heart was thought to be soft and welcoming, and better on the eye, along with indicating that one would love the taste, and enjoy it. Although some thought that the tick indicated that this was the right choice to make, others thought that it implied a healthy option rather than a treat, and was not as subtle as the heart.

Male participants thought that the caramel indicated that the bar was expensive, having a deluxe look. Conversely, the blue was not thought to be a colour associated with chocolate, and was thought to be unappetising, cold, clinical and cheap. Many of the males thought that the heart was childish, twee and too feminine. Although some thought that the tick was less feminine, others thought it was patronising.

5.3.4 Conclusions

To conclude, 16 packaging concepts were produced, consisting of four components: colour, size/shape, slogan and logo. These packages were assessed according to expected liking, with significant differences in scores being observed (supporting H₃); caramel, square bars, with a heart logo had the highest liking scores. Similar patterns of liking were observed for male and female consumers, and the ideal packaging combination was the same for both males and females (H₄ was not upheld). However, male participants showed less of a preference than females for all the attributes, except for colour.

5.4 Discussion

The objective of the work presented in this chapter was to explore possible styles of packaging a reduced-fat chocolate bar for maximised liking. The focus groups highlighted important cues that exist on current chocolate packaging that may suggest that the product is healthier or unhealthier. Furthermore, the discussion sessions encouraged idea generation surrounding the packaging of a reduced-fat chocolate bar. This resulted in four important components being recognised (colour, size / shape, slogan and logo), each with four manipulations (caramel or blue, long or square, pleasure or taste oriented, and heart or tick, respectively), to result in sixteen packaging concepts, which were assessed during the rank-rating task according to expected liking. The results indicated significant differences between the packaging concepts; the ideal packaging combination was a caramel, square bar, with the heart logo, and either of the slogans (although the taste-oriented slogan was described as preferential by some participants).

Both the male and female focus groups recognised colour as an important determinant of healthiness, suggesting a light colour, such as blue or lilac, for a reduced-fat bar. However, during the rank-rating task all participants showed a preference for caramel over blue bars. This contradiction in responses between the focus group and rank-rating data may be as a result of explicit versus implicit processing systems. Sanfey, Loewenstein, McClure and Cohen (2006) suggest that behaviour can be interpreted as choosing alternatives with the goal of maximising utility, and whilst operations involved in controlled processes are often accessible to introspective, explicit description, those involved in automatic processes are much less so. It is likely that

during the rank-rating task implicit instincts were relied upon (Raz *et al.*, 2008). However, findings from the rank-rating task should be given primacy, as it is what consumers do, and how they behave in the marketplace, that is important.

Colour has been recognised as important in food consumption, affecting the ability to recognise flavour (Blackwell, 1995; Lavin & Lawless, 1998; Morrot *et al.*, 2001; Zampini *et al.*, 2008), and an important feature in food choice (Marshall, Stuart & Bell, 2006). It has also been found that both colour and shape affect expected liking and willingness to purchase milk desserts (Ares & Deliza, 2010), where colour was particularly related to flavour, whereas shape was more related to texture characteristics. Smets and Overbeeke (1995) found that people are able to match packaging with desserts using both form and colour information. Shankar, Levitan, Prescott and Spence (2009) investigated whether colour and label information affected ratings of intensity of chocolate flavour and likability of candy shell coated chocolate, where the exterior colour of the candies is independent of the flavour of the chocolate interior. Authors found that brown candies were rated as significantly more chocolatey than green candies. Thus, the preference seen in this study may reflect the appropriateness of the colour caramel for indicating the taste of the chocolate bar. It has also been found that expensive, elegant and refined products directed at the upper classes require cold, dark coloured packaging, whilst products directed at price sensitive consumers require light coloured packaging (Ampuero & Vila, 2006). This could also explain the lower scores of expected liking for the blue bars. As other information presented on the packaging may have suggested an elegant product (i.e. the font and gold lettering), consumers may have felt that the light blue colour

contradicted this image. Both groups expressed that caramel indicated a deluxe, warm, luxurious expensive product, whilst the blue looked clinical and cheap.

Discussion within the groups indicated that the size and shape of the bar is important, although participants disagreed on the ‘perfect’ size and shape. The results of the rank-rating task indicated that the size and shape of the bars played a significant role in expected liking for females. The square bars were preferred, possibly as the bar is already reduced in fat and calories one can ‘afford’ to eat more, and so it should be sold as a larger, or apparently larger, bar.

The results of the rank-rating task did not indicate a preference for a taste oriented or a pleasure oriented slogan. The findings from the focus groups would suggest a preference for the taste-oriented slogan, as it does not highlight the product as a diet or health product. However, it may be argued that the “enjoy a guiltless pleasure” also does not heavily suggest that the product is for dieters, and is instead an uplifting tagline.

Although the female group discussed the use of logos on the packaging of the ‘healthier’ bars, they did not express an opinion when asked to discuss the packaging of a reduced-fat bar. However, they expressed a significant preference for bars containing a heart over a tick during the rank-rating. They did stress that the bars should be elegant, and may believe that this logo was more elegant. Furthermore, the heart logo may be more feminine, and so favourable for this group. The male group did not show a preference for either of the logos. Although the use of a logo was thought to be very important as it both emphasised any health claims, and indicated

quality, the type of logo, or in fact the meaning behind the logo, were not thought to be important.

Many ideas expressed during the focus groups were similar between the gender groups, and scores for the rank-rating task were comparable. However, both men and women believed that a reduced-fat bar would be aimed towards women, who are more weight and health conscious. Furthermore, men felt they would risk embarrassment buying a woman's chocolate bar, which may restrict the potential success of this type of product. This suggests that the female market should be targeted for this product, or further work should focus on exploring packaging options in order to more successfully market to men.

Future work should consider additional factors, as liking is not likely to be limited to the four factors addressed here. Real packaging includes other information, such as branding, which is likely to influence liking. Furthermore, other measures such as purchase intent and price propensity could be considered in order to more accurately determine behaviour in the market place. Finally, in order to more clearly define a possible target market other individual differences such as age, dieting status, levels of restricted eating and ambivalence should be considered, in order to further direct marketing efforts.

5.5 Conclusions

In conclusion, several important attributes, and levels of these attributes were identified during the focus groups, resulting in sixteen packaging concepts being produced. Conjoint analysis was used in order to discover the relative importance of

each attribute, and to calculate the optimum packaging concept. Overall, consumers exhibit relatively consistent responses to the type of packaging required for a reduced-fat chocolate to succeed in the marketplace. Although large differences in scores did not exist between male and female consumers, there were gender differences relating to attitudes towards energy and fat reduced products. Overall, these data suggest that specific marketing approaches will aid the successful introduction of a reduced-fat chocolate into the market.

6. Creation of cocoa butter emulsions and reduced fat chocolates:

Materials and Methods

6.1 Introduction

In this following section the materials used and procedures performed and described in Chapters 7, 8 and 9 will be detailed. The aim of the work presented in the chapters was to investigate formulation engineering routes for producing a reduced-fat chocolate, that has a similar flavour, texture and mouthfeel to full fat equivalent. Firstly, the materials used during lab scale and pilot plant scale experiments will be detailed. Following this, the processing methods used, and analytical techniques employed will be explained. Details of how the measurement techniques work can be found in Chapter 1.

6.2 Materials

Pure cocoa butter, commercially sourced sugar (icing sugar with tricalcium phosphate (E341), Silver Spoon, UK), lecithin (L- α -Phosphatidylcholine from soybean, Type II-S, Sigma, UK), polyglycerol polyricinoleate (PGPR, HLB 1.5 +/- 0.5, Kerry Bio-Science, UK), gelatin from porcine skin (250 bloom, Fluka Analytical, Germany) and distilled water were used in the experiments described in Chapters 7 and 8.

During scale up experiments, described in Chapter 9, the materials were sourced by the external company. The emulsions described in Chapter 9 were made with cocoa butter (type PPP, Cargill, The Netherlands), polyglycerol polyricinoleate (PGPR, Sugin 476/M, Cargill, Spain), milk fat (Campina, The Netherlands), porcine gelatin

(250 bloom, Rousselot, France), and potassium sorbate (E202, Brenntag, Belgium). Potassium sorbate was used as it is a preservative that prevents microbial growth, in particular bacterial growth, so is essential when producing food grade products. The emulsion was added to a dry mix of sugar (Tienen, Belgium), skimmed milk powder (Lactoland Trockenmilchwerk, Germany), cocoa powder (type 10 B2, Cargill, The Netherlands) and vanilla (LC 12153 P, Cargill, France). This dry mix was produced by blending and dry-grinding the ingredients to produce a powder with very little fat (0.1 – 0.5%), and a particle size of 20 – 24 μ m.

6.3 Preparation of emulsions

Emulsions were produced using three different methods: a high shear mixer (results displayed in Chapter 7); a bench-top margarine line (results displayed in Chapters 7 and 8); a Schröder Kombinator (results displayed in Chapter 9).

6.3.1 High Shear Mixer

A Silverson L4RT (Silverson Machines Ltd, UK) high shear mixer was used to produce a range of cocoa butter emulsions with varying water contents (from 10 to 60% water by weight). Temperature control was limited during this process. The emulsifier (either 2% soya bean lecithin or 1% PGPR) was added to the cocoa butter (where it is most soluble), and was heated to 45, 55 or 65°C using a bain-marie (placing the beaker inside another beaker of water which is heated on a heated plate). The distilled water was heated to the same temperature by placing the beaker on a magnetic hotplate stirrer (Stuart, UK), and stirring with a magnetic stirrer. If sugar was being used, it was added at this stage to make a sugar solution. The aqueous

phase was then added to the lipid phase, and the whole mixture was emulsified for 3 minutes using the high shear mixer, fitted with a fine emulsifier screen, at approximately 7400 rpm. Following emulsification, a sample of the emulsion was taken, which was sealed, and immediately refrigerated.

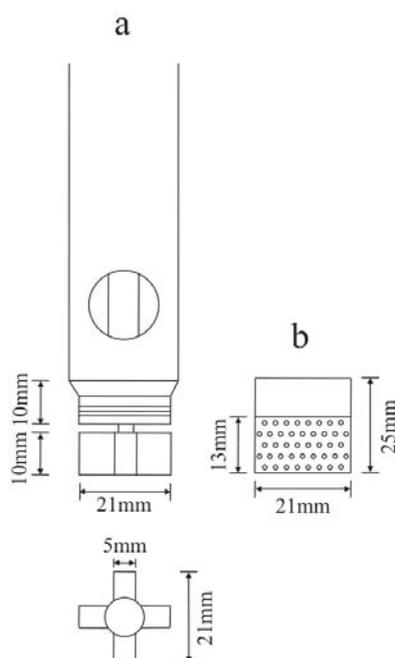


Fig. 6.1 A schematic of **a.** the Silverson high shear mixer, and **b.** the emulsion screen used. The screen screws on to the bottom of the shaft. The holes of the emulsion screen had a diameter of 1mm. The diagram serves an illustration, but key dimensions have been labelled.

6.3.2 Margarine line

The ‘margarine line’ comprises a scraped surface heat exchanger (A Unit, see Fig. 6.2.a) followed by pin stirrer (C Unit, see Fig. 6.2.b) (supplied by Unilever, Sharnbrook, UK), providing a continuous process (see Fig. 6.3), in which the temperature of the two jackets can be manipulated so that tempering can occur during the emulsification stage.

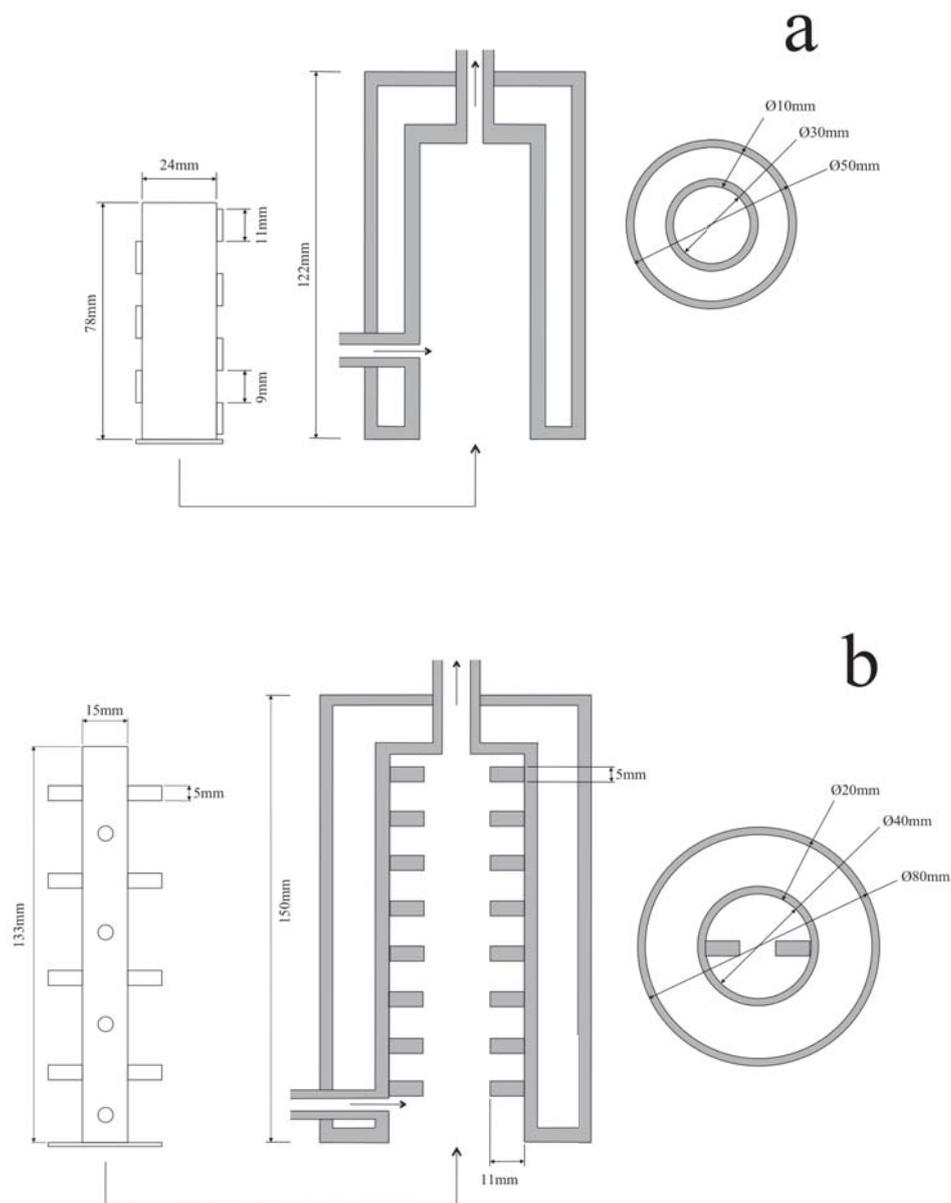


Fig. 6.2 A schematic of **a.** the scraped surface heat exchanger (A unit), and **b.** the pin stirrer (C unit). The diagrams serve illustrations, but key dimensions have been labelled. The schematic shows inlets and outlets (represented with arrows), but does not illustrate the jacket inlets or outlets. Shaded areas represent the jacket cavity.

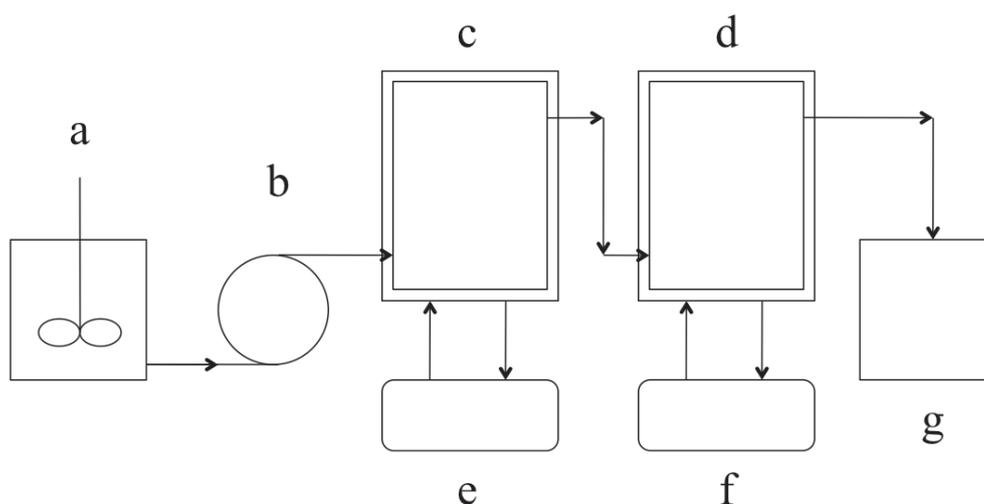


Fig. 6.3 A schematic of the margarine line, where **a.** is a beaker where the lipid and aqueous phases are added, and mixed using an overhead stirrer to produce a coarse pre-emulsion, **b.** is the peristaltic pump, **c.** is the scraped surface heat exchanger (A unit), **d.** is the pin stirrer (C unit), **e.** is the water bath used to control the temperature of the A unit, **f.** is the water bath used to control the temperature of the C unit, and **g.** is the collection vessel.

The cocoa butter and emulsifier (0.1 - 5% PGPR within the lipid phase) were heated using the water bath to approximately 60°C. This temperature was selected as it is above the final melting point of the cocoa butter crystals in all polymorphic forms (Loisel *et al.*, 1998). Initial experiments considered the addition of sugar to the aqueous phase (1, 10 and 20% by mass), whereas subsequent experiments investigated the addition of gelatin. When working with sugar, it was added to the aqueous phase and heated to 60°C on a hotplate stirrer (Stuart, UK), and was stirred using a magnetic stirrer. When using gelatin, the crystals were dissolved in the water at a temperature of 60°C, using the hotplate stirrer with a magnetic stirrer. The sugar / gelatin solution was then added to the cocoa butter, and stirred using an overhead stirrer (IKA RW20 digital) fitted with an anchor-shaped impeller until the mixture visually appeared homogeneous (judged by eye), which took approximately three minutes. This coarse pre-emulsion was then pumped through the margarine line (Masterflex L/S Digital Economy Drive with a Masterflex Easy-Load II L/S pump,

Cole-Parmer Instrument Company, UK), through silicon tubing (outer diameter 6.3mm, inner diameter 3.2mm; ESCO, SLC, UK). The A unit had a volume of 40ml, and had an 80mm stirrer with two 70mm blades. The C Unit had a volume of 150ml and had a 140mm stirrer with sixteen 5mm pins, and had sixteen 5mm pins in the shaft. T-junctions were inserted before the A unit to measure inlet temperature, and after the A unit, and after the C unit, to measure outlet temperatures. A Digi-Sense® dual type thermocouple and thermometer (Cole-Parmer® Instrument Company, USA) was used to measure the temperature at these points.

During the initial experiments (results presented in Chapter 7), the A unit jacket temperature was held at 30°C and the C unit at 40°C for all the samples. These jacket temperatures were selected to start fat crystallisation in the A unit and control the polymorphic form of the cocoa butter in the C unit. The exit temperature was measured and was approximately 35°C for all the samples. Settings were manipulated: shaft speeds (pin stirrer: 800, 1230 and 1350 rpm and scraped surface heat exchanger: 800 and 1320 rpm) and throughput (30, 50 and 70 mL/min). Following emulsification, samples of the emulsion were taken, sealed, and immediately refrigerated.

During subsequent studies (results presented in Chapter 8) a number of formulation changes (percentage of gelatin, percentage of aqueous phase, percentage of emulsifier), and processing changes (A and C units shaft speeds, A and C unit temperatures, and pump speed) were investigated.

6.3.3 Schröder Kombinator

The cocoa butter was melted in an oven (Jouan, UK) overnight at 60°C. The lipid phase ingredients (cocoa butter, PGPR, and milk fat, depending on formulation) were blended in a Schröder feed vessel, held at 60°C and run at a shaft speed of 120 rpm. The aqueous phase ingredients were prepared by heating the water to 60°C, and mixing the gelatin and potassium sorbate into the water using a laboratory mixer (Silverson AXR, UK) at maximum speed for approximately 5 minutes, until all the particles were dissolved. The aqueous phase was then added to the lipid phase, and homogenised. This pre-emulsion was then passed through the Schröder Kombinator (Belgium): 2 scraped surface Freon cooled heat exchangers, and through the pin stirrer. The pump was kept at a constant 110 rpm, keeping the product flow at a constant throughput of 0.75 kg/min. The shaft speeds of both Freon units were kept constant at 600 rpm. The temperatures of the scraped-surface heat exchangers were altered, as was the shaft speed of the pin stirrer (see Chapter 8). Samples of the prepared emulsions were immediately taken, and stored at both ambient (approximately 20°C) and chilled (approximately 6°C) temperatures. A dry mix of sugar (65.15%), skimmed milk powder (26.16%), cocoa powder (8.68%) and vanilla (0.01%) was also added to some of the emulsions following emulsification. This was carried out in a planetary mixer (Hobart, USA), until the solid particles were fully mixed into the emulsions. No temperature control was possible at this stage.

6.4 Analytical Techniques

6.4.1 Nuclear Magnetic Resonance (NMR)

The droplet size of the water within the final emulsions, and the percentage of emulsified water, was measured by NMR analysis, using both a Maran 20 (MHz) NMR spectrometer (Resonance Instruments, UK) and a minispec mq series pulsed-time domain NMR (Bruker, UK), with a water droplet size application specifically for W/O emulsions. The samples were put into 10mm NMR tubes, using a metal plunger, and filled to a height of 10mm. Unless otherwise specified, all NMR analysis was conducted within two weeks of emulsion manufacture. The NMR computes $d_{3,3}$, sigma and free water. $d_{3,2}$ can be calculated using the following formula (van Duynhoven *et al.*, 2002):

$$d_{3,2} = d_{3,3} \times e^{-0.5\sigma^2} \quad 6.1$$

where $d_{3,2}$ is the surface-weighted mean droplet diameter, $d_{3,3}$ is the volume-weighted mean droplet diameter, e is the exponential function, and σ is the standard deviation of the logarithm of the droplet diameter.

6.4.2 Differential scanning calorimetry (DSC)

A Perkin Elmer DSC Series 7 was used to determine the melting temperature (T_{peak} , °C) and the melting enthalpy (J/g) of the cocoa butter within the emulsion. Cooling was achieved with an Intracooler III (Perkin Elmer), that allowed reliable cooling or heating over the temperature range of -10°C to 100°C. The equipment was used with

thermal analysis software (Pyris). Nitrogen was used as a purge gas, at a flow rate of 30ml/min to prevent condensation occurring on the head. The DSC was firstly calibrated for temperature using indium and zinc standards, with an aluminum pan as a reference. Emulsion samples were loaded into Perkin Elmer 40 μ L capacity aluminum pans, and sealed with aluminum covers. An empty pan was used as a reference. The results were then analysed taking account of the sample weight and the fat content of the sample. Melting properties were compared with polymorphic information found in the literature (Loisel *et al.*, 1998). Different scan rates were investigated (0.5, 1 and 2°C/min). As the heating rate is doubled from 0.5°C/min to 1°C/min and from 1°C/min to 2°C/min the peak height approximately doubles (see Fig. 6.4). The energy of melting is then calculated, and shown to be constant across heating rates, indicating that the heating rates could be used interchangeably. A heating range of 2°C/min being preferred, not only for speed, but also for smaller error across samples.

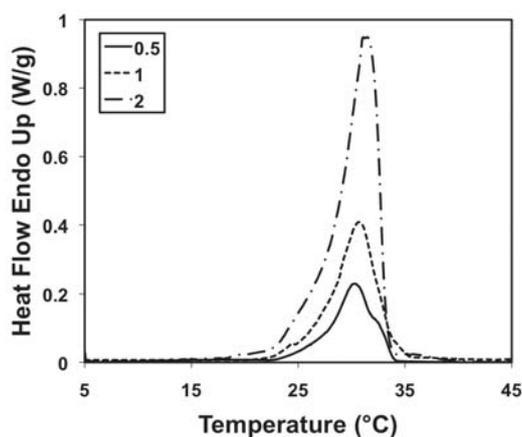


Fig. 6.4 DSC data indicating melting profile of the same emulsion (containing 30% water and 1% PGPR), heated from 5°C to 50°C at a rate of 0.5, 1, or 2°C/min. Results have been normalised for both sample size and fat content. An empty pan was used as a reference.

Table 6.1 Melting enthalpies of replicates of the same cocoa butter emulsion (containing 30% water), heated at a rate of 0.5, 1, or 2°C/min. Results have been normalised for both sample size and fat content.

Sample	Sample 1 (J/g)	Sample 2 (J/g)	Sample 3 (J/g)	Sample 4 (J/g)	Average (J/g)	Std. Dev. (J/g)
0.5°C/min	154.55	120.36	131.46	-	135	17
1°C/min	132.37	105.03	145.86	121.21	126	17
2°C/min	132.3	139.82	142.8	135.63	138	5

During scale-up trials (see Chapter 9) DSC analysis was conducted using a TA Instruments DSC Q100, with a TA instruments DSC Refrigerated Cooling System, and TA QSeries Software. Aluminium pans (Tzero, TA Instruments) and lids (Tzero Hermetic, TA Instruments) were used. Nitrogen was used as a purge gas. Samples were heated from 5°C to 45°C at a rate of 2°C/min. An empty pan was used as a reference.

6.4.3 Conductivity

A conductivity probe (Mettler Toledo, inlab 710 platinum 4-cell conductivity probe, connected to a Mettler Toledo SevenEasy™ meter) was used to determine whether the emulsions were fat continuous W/O emulsions. A conductivity of $0 \pm 0.1 \mu\text{Scm}^{-2}$ for each sample suggested a fully emulsified W/O emulsion.

6.4.4 Microscopy

Cryo-SEM was performed, using a Philips XL-30 FEG ESEM (Quorum cryo system with a Polaron PolarPrep 2000 cryo preparation chamber), in which the sample was frozen to below -80°C , coated with gold to allow electrical conductivity, and scanned by a focused electron beam to produce an image. Light microscopy was from a Polyvar II microscope (Reichert-Jung, Germany) in bright field mode.

6.4.5 Gravimetric Method

In order to determine the exact amount of water in the emulsion, the sample was weighed before and after it was placed in a dry oven at 110°C . This temperature was chosen as it would allow melting of the fat crystals, thus breaking the emulsion, and for evaporation of the water. The emulsions were placed in the oven for 8, 12 and 30 hours. The difference between the mass before and after heating gives the mass of the water lost. Knowing the amount of water within the original emulsion a calculation of the amount of water lost on storage (i.e. after emulsification and before being placed in the oven) can be made.

6.4.6 X-Ray Diffraction (XRD)

XRD experiments were conducted at the EPSRC National Crystallography Service located at the University of Southampton. The monochromatic beam of X-rays came from Molybdenum (Mo) radiation (Bruker-Nonius FR591 rotating anode ($\lambda_{\text{Mo}} = 0.71073\text{\AA}$), Coventry, UK) focused using 10cm confocal mirrors. Diffracted radiation was then collected on a CCD detector (Bruker-Nonius Apex II KappaCCD, Coventry, UK). The instrument was fitted with non-liquid nitrogen cooling circuit, allowing data to be collected at temperatures in the range of -193°C to 227°C . The sample was mounted on glass fibre, and inserted into a brass tube. All samples were measured at 4°C .

7. Development of cocoa butter emulsions using two processing methods: a high shear mixer and a bench-scale margarine line

7.1 Introduction

As discussed in Chapter 1, chocolate is consumed in large quantities by the British population, but is high in both fat and calories, has limited nutritional benefits and is only eaten as a snack or a treat. As such, producing a reduced-fat chocolate offers a way of reducing the fat and energy consumed, with beneficial effects for body weight and health. The aim of the work presented in this chapter was to identify whether the calorie and fat content of chocolate (by weight) could be reduced by introducing water, in the form of an emulsion. By creating a continuous oil phase (cocoa butter) with a dispersed aqueous phase, where the water droplets are segregated from the additional ingredients, it may be possible to “hide” the water, so that the product continues to function as a chocolate (for example is glossy, snaps and melts in the mouth), but is reduced in fat. Thus, the aim of this investigation was to create W/O cocoa butter emulsions with varying water contents, with small droplets of water which are below the detection threshold of the mouth (typically 10 μ m in size, although at high concentrations particles of 5 μ m in diameter can be detected; detection is affected by size, concentration, hardness, shape and viscosity; de Wijk & Prinz, 2005), which are stable, and do not destabilise or coalesce on storage. Furthermore, the cocoa butter within the samples should be in polymorphic form V (β_2), which consumers find the most attractive, as it results in a chocolate that snaps, melts in the mouth, and does not bloom, as described in Chapter 1. Furthermore, the

method for producing the emulsions should be similar to that used when producing other food emulsions (e.g. margarine), so that a high capital investment is not required. At this stage only the simple emulsion is investigated: the addition of the other ingredients (sugar, cocoa solids, and milk) will be discussed in Chapter 9.

Two methods of emulsification were employed: a high shear mixer (see Section 7.2) and a bench scale ‘margarine line’ (scraped surface heat exchanger and pin stirrer) (see Section 7.3). The different processing methods, in addition to varying water fractions (10 – 60% by mass) and different emulsifiers (soya bean lecithin and PGPR) were studied. Following processing, a number of analysis techniques were investigated to characterise the emulsions. Droplet size was measured using NMR analysis with a water droplet size application specifically for W/O emulsions (see Sections 1.4.7.6.1 and 6.4.1). DSC was used to determine the dominant crystal form of the cocoa butter within the emulsion (considering both the melting temperature, and the melting enthalpy), to determine whether an emulsion with crystals in polymorphic form V had been produced (see Sections 1.4.5.3 and 6.4.2). Finally, images were captured using both cryo-SEM and light microscopy, to determine the emulsion microstructure.

7.2 High Shear Mixer

The high shear mixer offers a batch process in which temperature control is limited, but it is a commonly used method of emulsification (see Section 6.3.1), which has the advantage that emulsions can be produced quickly and reliably. Results and knowledge obtained using this process will then direct production of emulsions using

the margarine line. The high shear mixer was used to produce a range of cocoa butter emulsions, using either soya bean lecithin or PGPR as emulsifiers. Both emulsifiers are used widely in chocolate manufacture to reduce the viscosity of the bulk, but can also be used to stabilise W/O emulsions (see Sections 1.4.7.3.1 and 1.4.7.3.2). A selection of samples, containing different percentages of water, were produced and studied over the course of 24 weeks in order to observe any polymorphic transitions and changes in droplet size.

7.2.1 Emulsifier Type

A range of emulsions were produced with water contents between 10 and 50%. This range of aqueous phase volumes was studied to consider the effect of water content on droplet size, as it should allow fat continuous processing throughout. Samples were produced with either 2% (of overall weight) soya bean lecithin or 1% (of overall weight) PGPR, to consider the effect of emulsifier type on droplet size for all water contents. Samples emulsified with soya bean lecithin resulted in emulsions that appeared visually stable for 24 weeks i.e. showing no creaming or sedimentation on storage and no water pooling at the bottom of the emulsion sample, meaning that all the water was contained in droplets within the emulsion. Measurements carried out by NMR (see Table 7.1) indicated that as the water content increased from 10% to 50% i) the percentage of water in small droplets ($< 100\mu\text{m}$) decreased from 97% to 66%, and ii) the percentage of water in droplets size greater than $100\mu\text{m}$ (termed ‘free water’) increased from 3% to 34%. Furthermore, droplet size increased with water content. It was concluded that only the emulsions containing 10% and 20% water

were fully emulsified, with 97% and 91% respectively of droplets being smaller than 100 μm , with a mean droplet size for both water contents of 1.6 μm (see Table 7.1).

Table 7.1. NMR analysis for cocoa butter emulsions produced using a high shear mixer. Emulsions were processed for 3 minutes at 7400rpm. Samples include 1 % sugar and 2 % soya bean lecithin. NMR measurements were taken in the first week after processing. Sigma (σ) is presented in brackets.

Percentage of water in sample	Percentage water in droplets >100 μm	Percentage water in droplets <100 μm	$d_{3.2}$ (μm)
10	3	97	1.6 (0.4)
20	9	91	1.6 (0.8)
30	21	79	3.4 (1.3)
40	23	77	3.9 (1.3)
50	34	66	7.9 (1.7)

Having only obtained stable emulsions for samples containing 10 and 20% using soya bean lecithin, PGPR was used as an alternative emulsifier. Samples were produced with water contents between 10 and 60% using 1% PGPR. These samples were again visually stable over the 24 week period.

Table 7.2 NMR analysis for cocoa butter emulsions produced using a high shear mixer. Emulsions were processed for 3 minutes at 7400 rpm. Samples include 1 % sugar and 1 % PGPR. NMR measurements were taken in the first week after processing. Sigma (σ) is presented in brackets.

Percentage of water in sample	Percentage water in droplets >100 μm	Percentage water in droplets <100 μm	$d_{3.2}$ (μm)
10	0	100	3.0 (0.7)
20	0	100	4.0 (0.8)
30	62	38	-
40	72	28	-
50	73	27	-
60	90	10	-

NMR measurements (see Table 7.2) indicated that all the water was contained in small droplets (i.e. below 100 μm) for the 10 and 20% water containing emulsions. However, as the water content was increased, an increasing amount of ‘free’ water

(i.e. droplets above 100 μm) was observed, so that an accurate measurement of mean droplet size could not be taken. PGPR is a powerful W/O emulsifier (Wilson *et al.*, 1998), and this is evident here (see Table 7.2), as 100% of droplets were emulsified at 10 and 20% water with a small droplet size of 3 or 4 μm . However, it appears that 1% PGPR is not sufficient to emulsify droplets at higher water contents, possibly as a result of insufficient time for the emulsifier to get to the interface within the process.

To compare the data obtained, the percentage of water in small droplets is plotted as a function of the water content for both PGPR and lecithin in Figure 7.1. As can be seen from this figure, using soya bean lecithin at water contents of 20% or below results in emulsions with more of the water in small droplets (<100 μm), as compared with PGPR. Both emulsifiers produce emulsions with small droplets (<100 μm) for 10 and 20% water content. As the water content is increased i) a gradual decrease in the percentage of emulsified water is observed with lecithin, ii) a decrease in emulsified water occurs between 20% and 30% water with PGPR. It would normally be expected that loss of emulsion stability would occur close to 50:50 phase volumes as the emulsion is likely to phase invert and become O/W. The most likely cause of the observed behaviour is that the emulsifiers are not adsorbed to the interface fast enough during processing, allowing coalescence of the droplets. This might be expected more for PGPR as it is a larger molecule so it will take longer to get to and stabilise the interface during emulsification (see Section 1.4.7.3.2). As would be expected, the results show that for emulsions with 10 or 20% water, the use of PGPR results in small droplets, 100% of which were smaller than 100 μm . Thus, if the emulsifier has sufficient time to get to the interface during processing, it can stabilise

emulsified structures. The rate at which emulsifiers get to the interface is concentration dependent (i.e. the more emulsifier present the quicker it will reach the interface), so future work should consider the effect of different quantities of emulsifier (this will be discussed in Chapter 8).

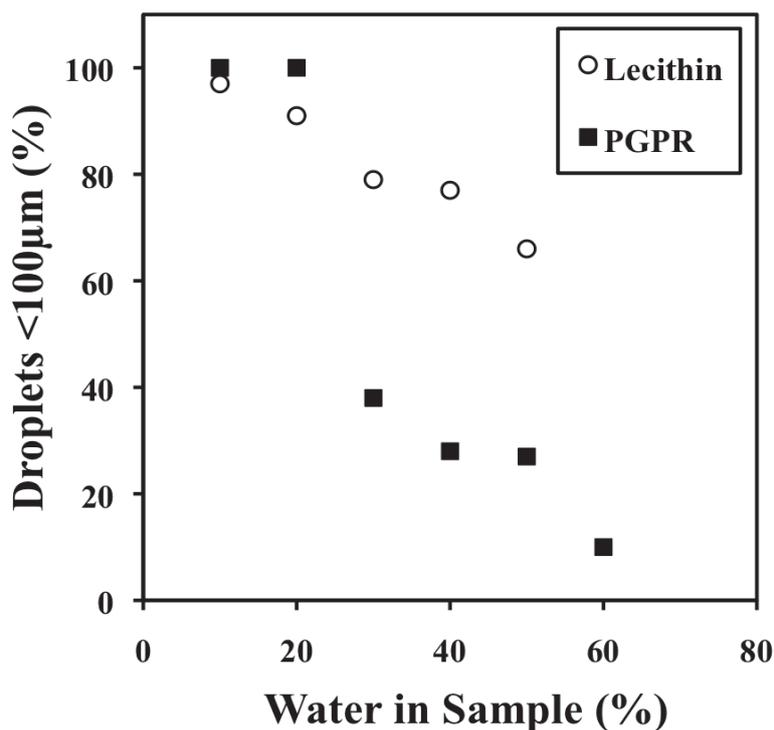


Fig. 7.1. Percentage of water in small droplets (<100µm), determined by NMR analysis, for samples containing soya lecithin and PGPR with different percentages of water. All samples were emulsified using a high shear mixer and then refrigerated. NMR measurements were taken in the first week after processing.

Having obtained stable emulsions with small droplet sizes, the microstructure of the emulsions was investigated using cryo-SEM (see Sections 1.4.7.6.2 and 6.4.4). Images obtained of the cocoa butter emulsions are presented in Figure 7.2. A number of images were obtained for each sample to determine whether the images were representative of the sample. In all the images, a network of fat crystals in the

continuous phase (A), and water droplets, covered by smooth sintered crystalline fat shells (B) can be observed.

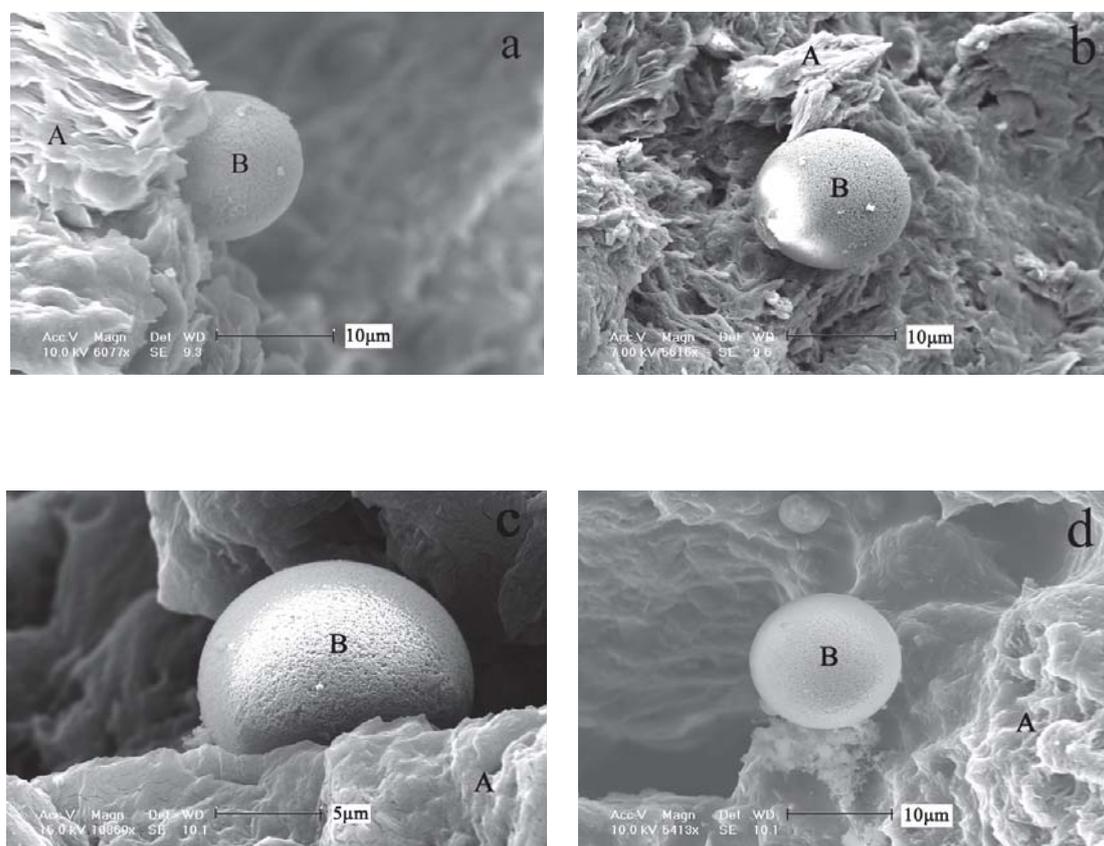


Fig. 7.2. Scanning electron micrographs of cocoa butter emulsions made with soya bean lecithin emulsified using a Silverson high shear mixer: **a.** 10% water phase, **b.** 20% water phase, **c.** 20% water phase and **d.** 40% water phase. All the micrographs indicate a continuous fat phase (A), with water droplets, covered by sintered fat crystal shells (B).

Very similar microstructures have previously been reported for margarine (Heertje, 1993; Heertje, 1998; Norton *et al.*, 2006). It is thought that as the fat crystals within the system begin to grow, they are swept to the interface during the emulsification process and then behave as Pickering particles (Hodge & Rousseau, 2005), stabilising the emulsion. As the crystals are soluble in the oil phase, they sinter together to form solid crystal shells (Johansson & Bergenstahl, 1995) over time (i.e. on storage). This

behaviour has been observed in other systems (Frasch-Melnik *et al.*, 2010), where a combination of mono- and triglyceride crystals acted as Pickering particles to stabilise a W/O emulsion without the addition of emulsifiers. Furthermore, Frasc-Melnik *et al.* (2010) report that the sintered fat crystal shells around the water droplets enable salt to be encapsulated within the droplets, which remained stable even under applied osmotic pressure, achieved by placing the emulsion in distilled water. Having these sintered crystal networks within a cocoa butter emulsion could prevent water migrating to the hydroscopic materials within the final chocolate, i.e. the sugar, cocoa solids or milk powder (this work is presented in Chapter 9). Furthermore, the crystal shells should melt in the mouth on consumption, resulting in a smooth mouthfeel. In margarine it is this fat shell that imparts both the physical and microbiological stability to the emulsion (Rousseau *et al.*, 2003). The water phase is contained in a solid structure even on the application of mechanical forces (i.e. on spreading) and any microbial activity will be isolated within a single droplet, preventing growth. It is therefore very likely that the same behaviour will be observed for the cocoa butter emulsions, making the production of a chocolate containing the emulsion very promising.

The droplets observed in all the cocoa butter emulsions using SEM are comparable across samples produced with different percentages of water. Droplet sizes are similar (between 5-15 μm), irrespective of the water content (see Fig. 7.2). This is in the same order of magnitude as obtained by NMR analysis (between 2 and 8 μm), however the droplets seen in the images are slightly larger. This may be as a result of the SEM

method, where there is a natural tendency to focus on larger structures, and a small number of droplets, while the NMR measures all of the droplets that are present.

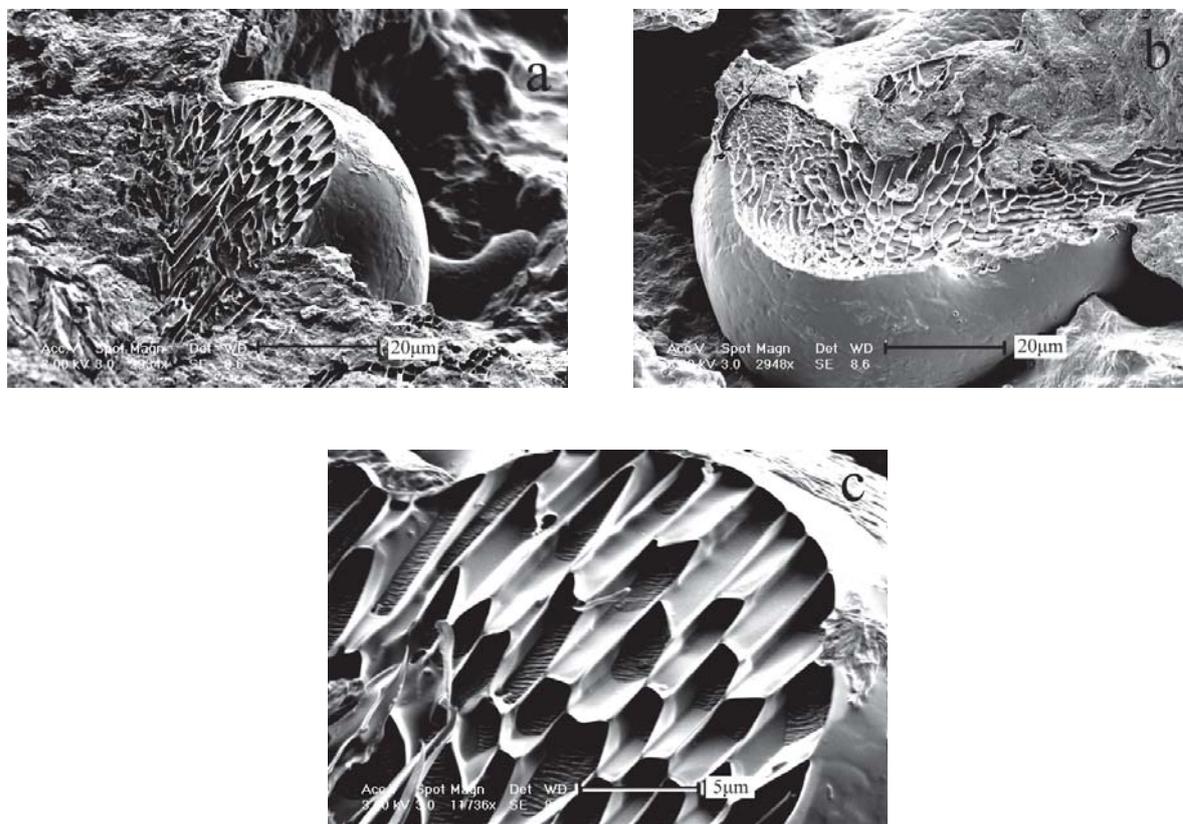


Fig. 7.3 SEM images of cocoa butter emulsions containing 20% water, 1% sugar and 1% PGPR that were processed using a Silverson high shear mixer. All the images show fractured cells.

Occasionally, fractured shells were observed within the emulsions (see Fig. 7.3). Similar fractured cells have been reported in the literature for other microstructures including margarine (Hirokawa, Ueda & Harano, 1994). The structure observed within the droplet (honeycomb or fishbone type structures) may be the sugar network, or an artefact observed following the freeze-process of SEM. The fractured shells suggest that the shell wall is sometimes insufficiently thick to resist the mechanical forces applied when the SEM samples were prepared, or during the emulsification process itself. Having fractured shells is not desirable as it could result in water

release on storage, on consumption or when snapped. It is possible that shell thickness could be controlled during processing, by using different fractions of crystallising fats, or by oscillating the temperature. This should be investigated in future work.

7.2.3 Processing Parameters

As temperature control is limited when homogenising with the high shear mixer, the effect of the temperature that the lipid phase was heated to prior to emulsification was investigated with the fat phase being heated to 45, 55 or 65°C. In all cases stable emulsions were produced. The samples were analysed to determine the polymorphic form of the fat in the emulsion. DSC was used to determine the dominant crystalline form present within the emulsions. All the emulsions, regardless of the temperature that the samples were heated to before homogenisation, had melting peaks that are similar to those typically reported in the literature (Loisel *et al.*, 1998) for polymorphic form V (melting between 30 and 34°C; see Fig. 5.4). This suggests that any fluctuations in temperature during the process (i.e. slight cooling in the time between moving from the hot plate, and heating again during emulsification, and final crystallisation on refrigeration) are not important in determining the polymorphic form of the fat. The fact that form V is observed suggests that the applied shear forces allow form V (or precursors to form V) to be produced spontaneously, which seeds the mass by secondary nucleation. This mimics the tempering process.

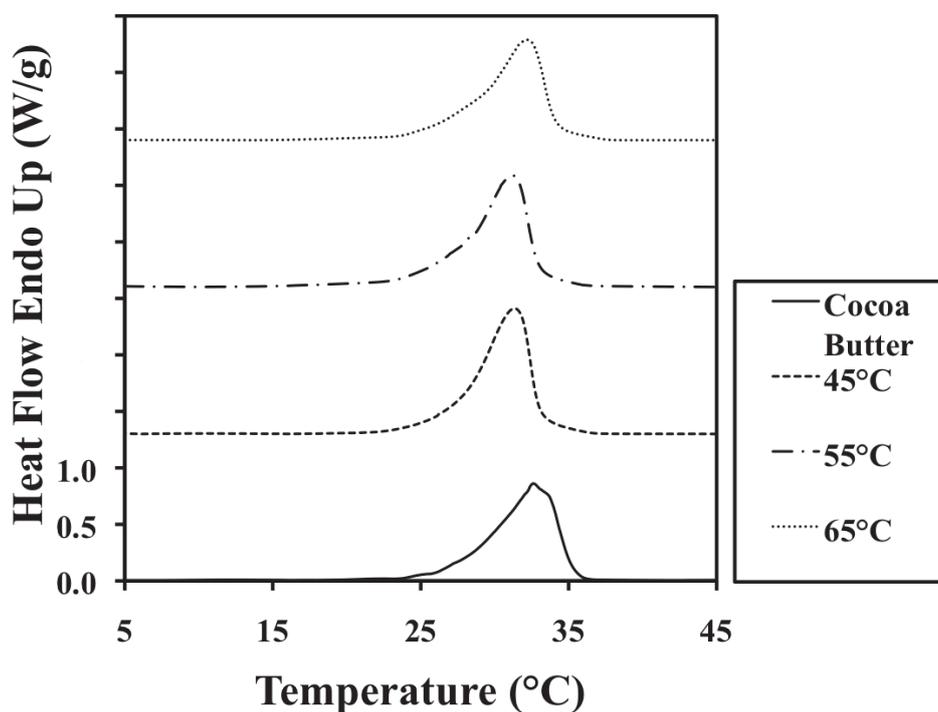


Fig. 7.4 DSC curves indicating melting temperatures of cocoa butter emulsions (all containing 30% aqueous phase) emulsified using a high shear mixer. The fat phase of the emulsions was heated to different temperatures (40, 55 and 65°C) prior to emulsification. Samples were refrigerated immediately after manufacture. Measurements taken 2 months after manufacture of emulsions. Pans were heated from 0°C to 50°C at a rate of 2°C/min. Pure cocoa butter processed in the same way is displayed as a comparison. These results have been normalised for both sample size and fat content. An empty pan was used as a reference.

The peaks shown in Figure 7.4 were analysed to determine the melting enthalpy (i.e. the area under the melting peak). All the samples had slightly higher melting enthalpies (see Table 7.3) than typically cited in the literature for form V (117.5 J/g; Chapman *et al.*, 1971). Instead, enthalpies between 127.9 and 144.8 J/g were observed. This may be as a result of the time delay between emulsification and the DSC analysis, which was two months, possibly allowing the development of crystals in form VI, which have a melting enthalpy of 136.3 J/g (see Table 7.3). However, a melting enthalpy of 143.5 J/g is 7.2 J/g higher than normally observed for polymorphic form VI (Chapman *et al.*, 1971). The higher enthalpies are therefore

almost certainly due to a loss of water on storage, resulting in increased fat content, leading to an inaccurate calculation when normalising for water content. This is the most likely explanation as the melting temperature are those of form V not form VI. This highlights the importance of conducting analysis immediately after emulsification (rather than after a two month period), or conducting DSC analysis over an extended period of time to gain time-dependent comparisons. In order to further study this, a storage time study was carried out, and reported in the next section.

Table 7.3 Melting properties of three cocoa butter emulsions (containing 30% aqueous phase) in which the fat phase was heated to 45, 55 or 65°C. Showing temperature start, peak and end, and melting enthalpy, as measured by DSC. In brackets, the standard deviation of triplicates.

Sample	T _{start} (°C)	T _{peak} (°C)	T _{end} (°C)	Melting Enthalpy (Jg ⁻¹)
45°C	28 (0.9)	31.4 (0.02)	34.2 (0.1)	127.9 (1.5)
55°C	27.7 (1.5)	31.2 (0.1)	33 (1.1)	144.8 (3.5)
65°C	28.1 (1)	32.4 (0.3)	33.8 (0.5)	143.3 (2.3)

The results shown in Table 7.3 indicate that although a formal tempering process was not performed, applying shear to cause nucleation of the fat, followed by cooling to approximately 5°C, is sufficient to ‘temper’ the fat, and produce crystals in form V that melt between 31 and 33°C. However, the transition of the fat crystals over time should be considered, to determine whether form V crystals are produced directly from processing.

7.2.4 Storage Time

Emulsions with different percentages of water (10, 30 and 50%) emulsified with soya bean lecithin, were analysed by DSC at varying time intervals following manufacture, to consider i) whether the presence of water affected the behaviour of the emulsion

over time and ii) whether the percentage of water affected melting properties. The results for the emulsions were compared with data found in the literature, and with measurements taken of pure cocoa butter processed in the same way.

It can be seen (see Table 7.4 and Fig. 7.6) that after one day and one week all the emulsions had similar T_{peaks} (27 – 29°C), which suggests that the cocoa butter within the emulsion is in form IV. Conversely, the pure cocoa butter has a higher T_{peak} than the emulsions after one day (30.8°C), suggesting the presence of form V crystals.

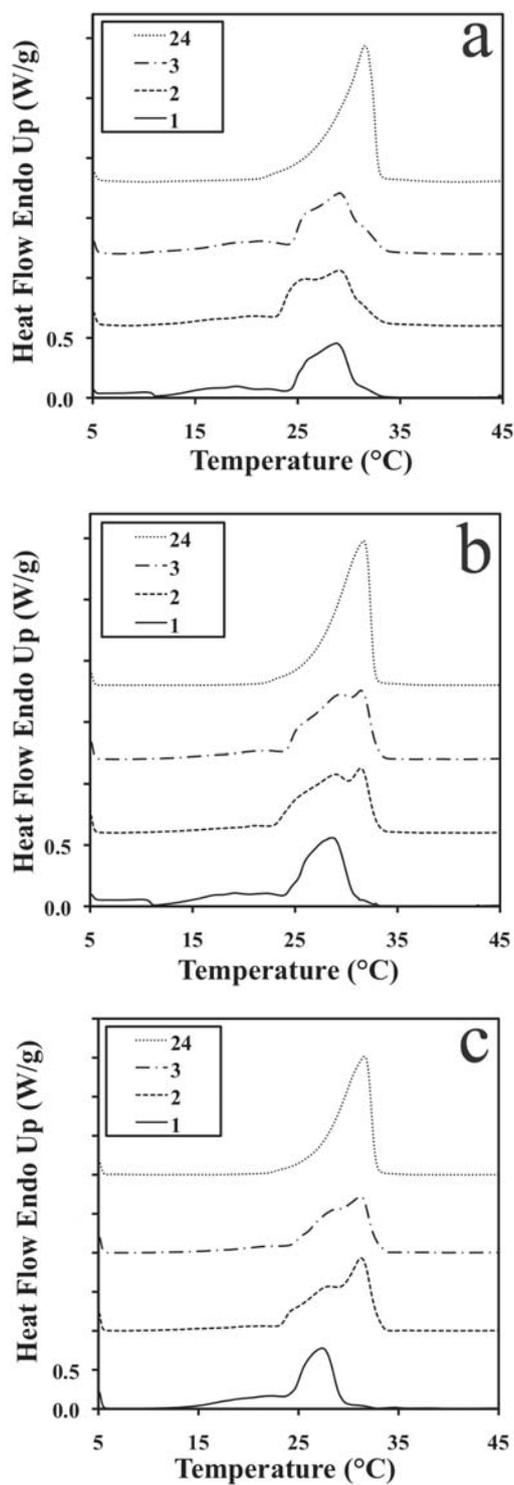


Fig. 7.5 DSC curves indicating the melting temperatures emulsions containing **a.** 10% aqueous phase, **b.** 30% aqueous phase, and **c.** 50% aqueous phase. Measurements were taken after varying lengths of time (1, 2, 3 and 24 weeks, as shown in legend). Pans were heated from 5°C to 45°C at a rate of 2°C/min. These results have been normalised for both sample size and fat content. An empty pan was used as a reference.

Table 7.4 The temperature onset, peak and end (all °C) and enthalpy (J/g) of cocoa butter emulsions over time. Tempered cocoa butter is displayed as a comparison. In brackets, the standard deviation.

Sample	T _{onset} (°C)	T _{peak} (°C)	T _{end} (°C)	Melting Enthalpy (Jg ⁻¹)	Number of peaks
Cocoa Butter					
1 day	10.8 (2)	30.8 (1)	33.5 (1.2)	108.1 (9.3)	1
1 week	9.4 (0.3)	29.9 (1.1)	33.8 (0.1)	109.3 (1.3)	1
2 weeks	9.2 (0.2)	29 (0.1)	34.6 (1)	115.4 (1.5)	1
3 weeks	10.3 (0.8)	29.4 (0.3)	35.1 (0.9)	119.7 (6.7)	1
5 weeks	9.3 (0.1)	31.8 (0.2)	35.5 (2.5)	131.0 (2.4)	1
7 weeks	10.9 (0.4)	31.4 (0.2)	33.1 (0.1)	128.5 (3)	1
12 weeks	12.3 (1.5)	31.5 (0.7)	34.5 (1.3)	136.4 (3.7)	1
24 weeks	15.2 (2.8)	32.4 (0.5)	35.7 (1.1)	133.6 (5.4)	1
10% Water					
1 day	24.6 (0.6)	28.7 (0.1)	30.9 (0.2)	92.5 (8.4)	1
1 week	23.4 (0.1)	29.1 (0.1)	30.9 (0.1)	105.9 (9.7)	2
2 weeks	23.0 (0.2)	29.1 (0.1)	31.5 (0.5)	114.4 (7.3)	2
3 weeks	24.4 (0.2)	29.0 (0.4)	32.2 (1.3)	103.9 (4.6)	1
5 weeks	26.3 (3.4)	32.0 (0.5)	33.5 (0.4)	125.2 (8.6)	1
7 weeks	26.2 (2)	32.3 (0.2)	33.7 (0.2)	118.5 (8)	1
12 weeks	27.6 (2.7)	32.9 (0.3)	34.1 (0.1)	126.9 (7.4)	1
24 weeks	28.3 (0.2)	31.7 (0.4)	33.7 (1.5)	147.2 (5.1)	1
30% Water					
1 day	24.4 (0.6)	28.8 (0.5)	30.3 (0.5)	110.4 (8.5)	1
1 week	24.0 (0.4)	28.4 (0.3)	30.1 (0.4)	107.4 (2.5)	1
2 weeks	24.7 (2.7)	29.8 (1.5)	32.9 (0.2)	118.6 (3.2)	2
3 weeks	24.1 (0.1)	30.0 (1.2)	33.0 (0.1)	119.7 (1.5)	2
5 weeks	28.0 (0.2)	32.6 (0.1)	33.7 (0.2)	139.8 (1.5)	1
7 weeks	28.2 (0.6)	32.9 (0.7)	34.3 (0.8)	142.3 (0.5)	1
12 weeks	28.1 (0.2)	32.9 (0.1)	34.0 (0.1)	140.3 (2.4)	1
24 weeks	26.7 (0.04)	31.7 (0.05)	32.7 (0.04)	147.7 (2)	1
50% Water					
1 day	24.0 (0.7)	27.3 (0.2)	29.3 (0.1)	119.9 (1.7)	1
1 week	23.4 (0.3)	27.6 (0.1)	29.5 (0.2)	123.9 (4.9)	1
2 weeks	26.4 (3)	31.4 (0.01)	32.6 (0.3)	152.6 (8.5)	2
3 weeks	24.5 (0.7)	31.2 (0.04)	32.7 (0.1)	135.8 (5.3)	2
5 weeks	28.1 (0.6)	32.2 (0.01)	33.4 (0.1)	157.9 (6.9)	1
7 weeks	28.2 (0.6)	32.4 (0.4)	34.2 (0.8)	163.9 (13.3)	1
12 weeks	27.7 (0.02)	32.2 (0.2)	33.5 (0.1)	166.5 (9)	1
24 weeks	26.8 (0.1)	31.6 (0.2)	32.7 (0.04)	184.4 (1.7)	1

After two or three weeks all three emulsions appeared to have a mixture of crystal forms, indicated by two different peaks (see Table 7.4 and Fig. 7.5), whilst the pure cocoa butter had a single peak.

After 12 and 24 weeks all three samples had similar T_{peaks} (32.2 – 32.9°C) and all had single peaks, indicating the presence of crystals in form V. The T_{peak} for all these emulsions is plotted as a function of storage in Figure 7.6. This figure shows that crystals in form V are produced after five weeks and are then stable. In contrast to the emulsions, the melting profile of the pure cocoa butter indicated that from five weeks form VI crystals had developed, with a higher melting enthalpy (for example 131 J/g at five weeks). As mentioned previously, on storage some of the melting enthalpies observed for the emulsions are higher than observed for pure cocoa butter, or as cited in the literature. This is true for all emulsions measured, but especially true for the emulsions with 30 or 50% water content, where the highest amount of non-emulsified water is observed. Thus, the high enthalpy is probably due to a loss of water over time, resulting in an inaccurate normalization for fat content. This should form part of any further work.

These results suggest that storing the samples at 5°C over 5 weeks and up to 24 weeks results in a crystal transition, observed by an increase in T_{peak} , to a melting temperature expected for a tempered cocoa butter in form V. Furthermore, it appears that this change is independent of the water content in the emulsion, suggesting that water content has little or no effect on the crystal form of the cocoa butter emulsions. However, it should be noted that the aqueous phase volume might affect the accuracy of the melting enthalpy calculations.

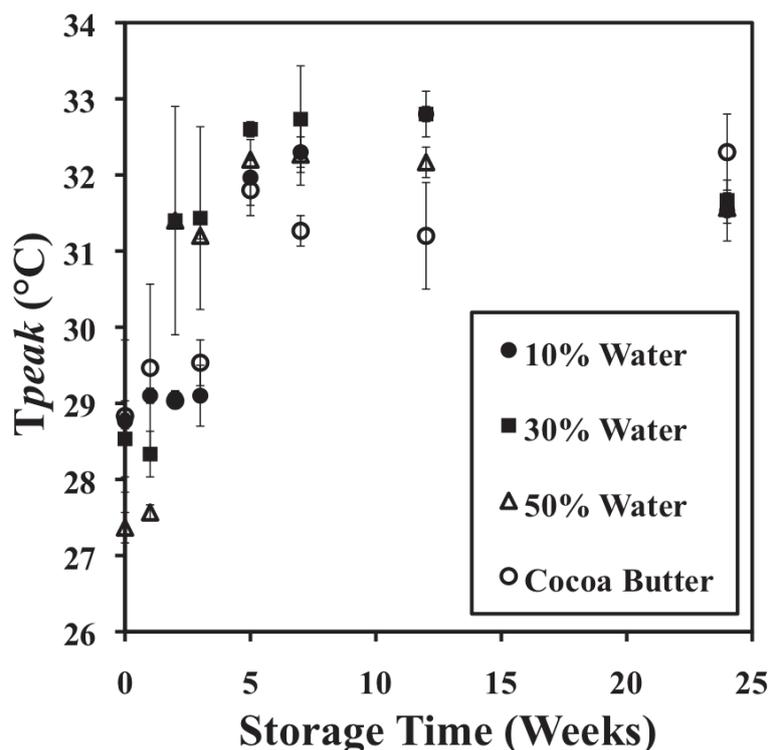


Fig. 7.6 Average temperature peak of cocoa butter emulsions (containing 10, 20 and 30% water) emulsified using a high shear mixer, at varying time intervals (from 1 day to 24 weeks).

7.2.5 Conclusions

It has been shown that the presence of 1% PGPR results in 100% of the water being emulsified in small droplets (3-4 μ m) at 10 and 20% water contents, which is higher than observed for lecithin. At this water content, the droplets are in the size range preventing detection in the mouth (the mouth can typically detect particles of 10 μ m in diameter; de Wijk & Prinz, 2005), or microbial growth (droplet sizes less than 20 μ m do not harbor bacteria in W/O margarines; Duncan, Yaun & Sumner, 2004), although this was beyond the scope of the study. Furthermore, the smooth crystalline shells at the droplet interface suggest sintering of fat crystals has occurred giving greater

physical and microbial stability. Some fracturing of the shells was evident, which probably explains the possible water loss being observed on storage. Future work should investigate whether fracturing can be prevented, resulting in greater stability. Water droplets within the cocoa butter do not appear to affect the melting properties of the fat. However, the lack of accurate temperature control during processing does not reliably produce emulsions with crystals in form V. Although the work presented here shows that form V crystals develop over time, it is preferable to produce an emulsion with crystals in form V directly from processing, and not after a time delay of five weeks or more. By controlling the temperature during processing it may be possible to seed form V directly in the process. As the margarine line offers accurate temperature control, and is a continuous process allowing higher production rates, it was investigated as an alternative processing route, and reported in the next section.

7.3 Margarine Line

A number of cocoa butter emulsions were prepared on a bench scale margarine line as detailed in Table 7.5. The samples produced all had 20% aqueous phase and 1% PGPR. PGPR was selected because as reported in the previous section of this Chapter, it resulted in emulsions which were fully emulsified at 20% aqueous phase when processed with the Silverson high shear mixer (see Section 7.2). Processing parameters, shafts speeds, throughput, and outlet temperatures were investigated (see Table 7.5). As sugar is an ingredient in chocolate it was also decided to investigate the effect of sugar (up to 20%) within the aqueous phase. The reason for doing this was that if sugar comes into contact with water in chocolate the sugar crystals aggregate and form lumps. This increases the friction and viscosity of the chocolate (Afoakwa *et*

al., 2007), and results in an unacceptable product. Thus, the addition of sugar is an important consideration if the cocoa butter emulsion is to be used in chocolate. Here, it was decided that the addition of the sugar to the aqueous phase prior to emulsification might prevent problems post-processing.

Table 7.5 Details of emulsion formulation and margarine line set up, along with measured exit temperatures.

Sample	Sugar Content (%)	A Unit Shaft Speed (rpm)	C Unit Shaft Speed (rpm)	Through put (mL/min)
1	1	1230	1320	50
2	1	1230	1320	30
3	1	1230	1320	70
4	1	800	1320	50
5	1	1350	800	50
6	10	1230	1320	50
7	20	1350	1320	50

As free water had been implicated for the increases in the melting enthalpy measured during the previous study (see Section 7.2), it was decided that the conductivity of the samples following homogenisation would be measured to check for any water continuous regions in the samples. As the samples should be fat continuous (i.e. no water conductivity), the conductivity should always be $0\mu\text{Scm}^{-2}$. All the samples produced had conductivities of $0 \pm 0.1\mu\text{Scm}^{-2}$ (see Table 7.6) showing that they were all fully emulsified, with no water continuity. These results were supported by

NMR analysis (see Table 7.5) that showed that no free water was observed for any of the samples, and all had small droplet sizes and normal size distributions.

NMR analysis (see Table 7.5) supported the conductivity measurements, and indicated that all samples emulsified using the bench scale margarine line were fully emulsified, with no free water, and droplets of between 0.6 and 4.9 μm . These results suggest that the margarine process resulted in smaller droplet sizes for samples 1-6, than the droplets produced using the Silverson (i.e. 1.5 μm and 3-4 μm , respectively). This would be expected, as the residence time in the margarine line is longer, giving greater time for the emulsifier and the crystals to get to the droplet interface as the emulsion droplets are formed. The addition of 20% sugar to the aqueous phase had a small effect on droplet size, with the largest droplets (4.9 μm) being observed for sample 7. The reason for this increased droplet size is unclear; however, this droplet size is still in the range that is expected to result in stable emulsions.

Table 7.6 NMR analysis for cocoa butter emulsions produced using the margarine line. Sample includes 20% water and varying percentages of sugar. Sigma (σ) is presented in brackets.

Margarine Samples				
Sample	Percentage water in droplets >100 μm	Percentage water in droplets <100 μm	$d_{3,2}$ (μm)	Conductivity (μScm^{-2})
1	0	100	1.6 (0.1)	0
2	0	100	1.5 (0.1)	0
3	0	100	1 (0.2)	0
4	0	100	1.4 (0.1)	0
5	0	100	0.6 (0.1)	0
6	0	100	1.3 (0.3)	0
7	0	100	4.9 (0.9)	0

Again, in order to investigate the polymorphic form of the cocoa butter, calorimetry was carried out on the samples; the data in Table 7.7 are averages of triplicates.

Table 7.7 DSC analysis of cocoa butter emulsions made using the bench-scale margarine line, containing 20% water, 1% PGPR and 1% sugar.

Sample	T_{start} (°C)	T_{peak} (°C)	T_{end} (°C)	Melting Enthalpy (Jg ⁻¹)
1	26.4 (0.2)	31.8 (0.1)	33.1 (0.1)	135.6 (2.7)
2	21.9 (0.4)	29.2 (0.4)	31.7 (0.2)	90.4 (0.2)
3	23.2 (0.3)	29.9 (0.4)	31.8 (0.8)	85 (11.3)
4	27.2 (1.2)	31.2 (0.2)	32.4 (0.2)	135.5 (2)
5	20.9 (0.5)	28.6 (0.7)	31.3 (0.4)	93.6 (0.7)
6	21.3 (0.2)	28.9 (0.1)	30.9 (0.2)	102.3 (3.9)
7	21.3 (0.5)	28.6 (0.4)	31.2 (0.2)	82.3 (0.8)

As can be seen from Table 7.6 samples 1 and 4 give a melting peak typical for polymorphic form V i.e. T_{peak} of between 30 and 34°C. For these samples the exit temperature from the process (from the pin stirrer) was between 29 and 32°C i.e. close to the melting temperature of the desired polymorphic form of the cocoa butter. The other samples had exit temperatures at or above 35°C i.e. above the temperature for melting of form V. Thus, any nuclei produced in the scraped surface heat exchanger will have been re-melted in the pin stirrer. Therefore, crystallisation occurred following emulsification into the lower melting polymorphic forms, as would be expected for untempered cocoa butter. These results show that by carefully controlling the temperatures of the two units, it is possible to produce a tempered emulsion during emulsification, without the need for a traditional tempering process. The melting enthalpy typically observed for stable crystals in form V is 117.5 J/g. Sample 1 and 4 had melting enthalpies of 135.6 J/g and 135.5 J/g, respectively. These values are similar to those normally observed for cocoa butter in form VI. However, the DSC measurements were made only two days after emulsification, so it is unlikely that crystals in form VI would have transformed in this time scale. This may have been caused by inaccuracy in determining the curve baseline, which is often noisy.

This will naturally cause errors in the peak area, however for samples containing water this is amplified as the size of the peak decreases. Ideally a micro-DSC would be used as it allows larger samples to be loaded, increasing the size of the peak in comparison to any noise, reducing any errors. The remaining samples (2, 3, 5, 6, and 7), all of which had exit temperatures above the melting temperatures of form V, had unstable crystals, with T_{peaks} , and melting enthalpies lower than expected for form V crystals. This would be expected, as any form V seeds produced in the A unit would have been remelted before the emulsions exited the pin stirrer, as discussed previously.

Cryo-SEM images were taken of a number of emulsions homogenised using the margarine line (see Fig. 7.7). Image a) shows a continuous fat phase with large indentations which are thought to be air bubbles, introduced into the system during emulsification (probably during the pre-emulsification step). The process needs to be improved so that air is not routinely incorporated in to the samples, by improving the inlet connections, and by using a more consistent pump. These air cells are far larger in size than the water droplets measured by NMR for the same sample (approximately 20 μm , rather than 1-5 μm). In image b) a similar continuous fat phase can be observed, with indentations and protrusions of approximately 40-50 μm in size, which are also thought to be air bubbles. Smaller protrusions of approximately 10 μm are also evident, which are the sintered fat crystal shells covering the surface of the water droplets. Similar structures have previously been observed in W/O spreads by SEM (Heertje, 1993; Heertje, 1998; Alexa, Mounsey, O'Kennedy & Jacquier, 2010).

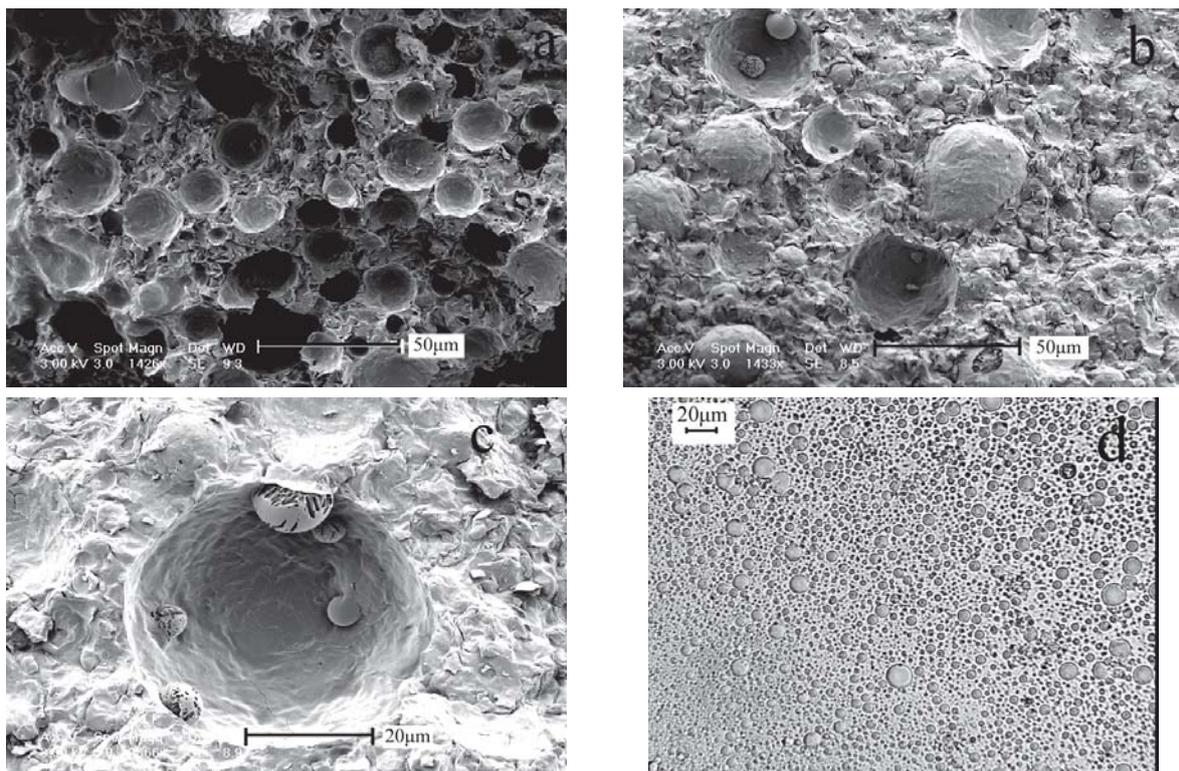


Fig. 7.7 Micrographs of cocoa butter emulsions processed using A and C units: **a.** cryo-SEM image of a continuous fat phase with indentations of air bubbles created during emulsification, **b.** cryo-SEM image of a continuous fat phase with indentations and protrusions of air bubbles, and protrusions of water droplets **c.** cryo-SEM image of a continuous fat phase with an indentation of an air bubble, with water droplets at the interface, and **d.** light microscope image of an emulsion made on the A and C units, showing the distribution of water droplets within the fat matrix.

In image c) a continuous fat phase can be seen, with the indentation of an air bubble. At the interface of the air bubble and the fat a number of water droplets with fat crystal shells can be observed. These have fractured shells, showing similar structures to those reported in the previous section of the chapter, which possibly indicate a sugar structure produced by the freezing process, as previously discussed (see Section 7.2.1). Figure 7.7.d shows a typical light micrograph obtained, in which the distribution of water droplets (approximately 5 μm in size) within the fat matrix can be seen. Although a number of larger droplets are evident they account for only a very small percentage of the phase volume.

7.3.1 Conclusions

All of the samples produced using the margarine line had fully emulsified water, with small droplets (1-5 μ m). It was shown that crystals in form V could be produced during the process, probably as a result of the shear, but the study highlighted the need for careful temperature control in order to successfully mimic the tempering process.

7.4 Overall Conclusions

Using the high shear mixer resulted in stable, fully emulsified W/O cocoa butter emulsions with water contents of up to 20%, that had no 'free' water, and droplets of 1.6 – 4 μ m. The DSC results suggest that by storing the cocoa butter emulsions at 5°C over approximately a 5 week period, emulsions with a melting point and melting enthalpy similar to tempered cocoa butter in form V were produced. Furthermore, this was independent of water content. Samples with 10, 30 and 50% water all followed the same trend, transforming from form IV to form V over a similar number of weeks, suggesting that the water content has little effect on the melting characteristics of the emulsions.

The bench scale margarine line offered temperature control during the process, and providing that the temperatures of the two units were controlled to produce form V crystals, a cocoa butter emulsion with crystals in the desired form for chocolate could be produced successfully. The margarine line has been shown to be more successful for creating an emulsion with fat in the polymorphic form V, as the temperature and shear of the two units can be controlled with more precision. Thus, it offers an

advantage over the high shear mixer because emulsification and tempering can occur during the same processing stage, without the need for a delay of weeks or months. Furthermore, it is more appropriate for large-scale industrial production as it is a continuous process, and if with further work it can be produced in a similar way to margarine, this process could be used to produce chocolate up to 10tonnes/hr on a single line (a typical margarine line capacity).

Thus, this study demonstrates that the production of cocoa butter emulsions can be achieved using technology typically used for margarine manufacture. It shows the potential for the use of cocoa butter emulsions in chocolate formulations for fat reduction. Furthermore, the observation of crystalline shells at the water droplet interface offers potential when adding the other chocolate ingredients. Providing that the crystal shells are thick enough (which should form part of further studies), this would create a barrier to physically separate the water from the cocoa solids and sugar. This could overcome the problem that occurs if water comes in to contact with the dry ingredients: the particles will aggregate, having a dramatic effect on viscosity. If this can be prevented using Pickering stabilised droplets, it will be possible to add the dry particles post-emulsification. Thus, it was decided that the addition of sugar directly to the aqueous phase of the cocoa butter emulsion could be avoided in the future.

The methods of analysis (NMR, DSC and SEM) were successful for gaining sufficient microstructural information about the cocoa butter emulsions. Both NMR and SEM offer comparable indications of droplet size, whilst DSC provides

information about crystal form, which is important when producing an emulsion, or when working with cocoa butter.

Further work should aim at manipulating more variables both in terms of formulation (for example, the percentage of emulsifier used), and processing (shaft speeds, temperature of units, pump speed) in order to optimise the process, and reliably produce an emulsion that contains small droplets, and fat crystals in polymorphic form V directly from emulsification. Further work should also aim at adding the particulates post-emulsification to consider the effect of the water within the emulsion on the final chocolate.

8. Effect of changes in formulation and processing parameters on melting profile and water droplet size

8.1 Introduction

During Chapter 7, studies on the production of cocoa butter emulsions were described. The W/O emulsions were produced using both a high shear mixer, and a margarine line. The use of PGPR resulted in fully emulsified emulsions (100% of the droplets were 3-4 μ m in size), and the temperature control of the margarine line allowed for the production of emulsions with ‘tempered’ cocoa butter. Furthermore, smooth crystalline shells were observed at the droplet interface, suggesting that the fat crystals act as Pickering particles, stabilising the water droplets, resulting in greater physical stability. The aim of the research described in this chapter was to investigate the effect of adding gelatin to the aqueous phase. Gelatin is added to the aqueous phase of low-fat spreads both to improve processing of the emulsion as the water content is increased, and to prevent coalescence by increasing the viscosity of the dispersed phase (Rousseau *et al.*, 2003). In this study the aim was to increase the stability of the emulsion, as defined by the droplet size and ‘free’ water content (percentage of droplets above 100 μ m in size). This was investigated using the margarine line, and by manipulating the shaft speeds and temperatures of both the units and the throughput (flow) in order to optimise the process. Furthermore, changes in the emulsifier concentration were investigated (i.e. the percentage of PGPR).

Biopolymers are often used in food emulsions to gel the aqueous phase and stabilise the emulsion by preventing coalescence, flocculation, sedimentation or creaming (see

Section 1.4.7.5). Gelatin is often added to fat-reduced spreads to hinder coalescence by increasing the viscosity of the dispersed phase (Rousseau *et al.*, 2003). Gelatin is particularly favourable as it has a similar consistency and ‘melt-in-mouth’ behaviour to the fats typically used in foods (Clegg *et al.*, 1996). Figure 8.1 shows the melting profile of three gelatin gels (6, 8 and 10% gelatin in water). The melting peak (31.9°C, $\sigma = 0.8$) is very similar to that cited in the literature for cocoa butter in polymorphic form V (Loisel *et al.*, 1998), making it an advantageous addition to the aqueous phase, as it can be processed at the same temperature as cocoa butter, and should not be detected by the consumer.

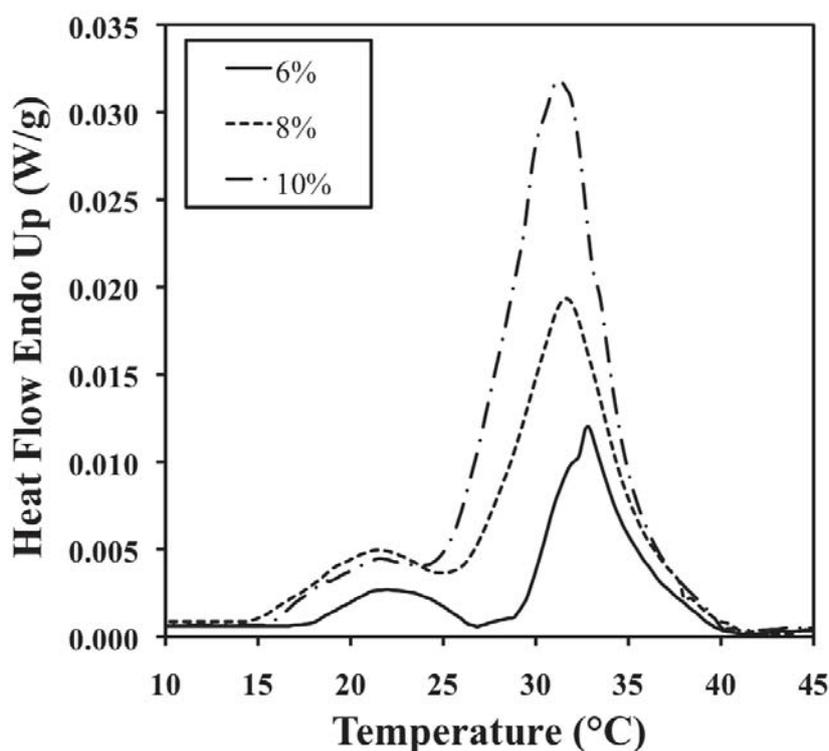


Fig. 8.1 DSC curve indicating the melting profile of solid gelatin solutions, containing 6, 8 and 10% gelatin. Pans were heated from 5°C to 45°C at a rate of 2°C/min. An empty pan was used as a reference. These results have been normalised for sample size. Melting peak for 6% gelatin is 32.3°C (0.5), for 8% is 31.6°C (0.9) and for 10% is 31.8°C (1).

Thus, gelatin could be used in a cocoa butter emulsion to improve stability, and help prevent moisture loss on fracture (e.g. on snapping) by structuring the water.

8.2 Formulation Changes

8.2.1 Percentage of PGPR

The emulsions produced, studied, and described in Chapter 5 contained either soya bean lecithin or PGPR. It was shown (see Chapter 7) that for emulsions containing smaller aqueous phase volumes (i.e. 10 and 20%), PGPR was a more powerful emulsifier, emulsifying 100% of the available water, so that the emulsion contained no 'free' water (i.e. all the droplets produced were smaller than 100 μ m). Furthermore, the droplets produced were small, typically between 1 and 5 μ m in size. However, only one percentage of PGPR was considered (1%). Here, a number of W/O emulsions containing 20% aqueous phase were produced, containing different percentages of PGPR (0.1, 0.5, 1, 2 and 5%) within the fat phase. The emulsions were all made with 20% aqueous phase (of which 5% is gelatin from porcine skin, 250 bloom, i.e. 1% gelatin in the total emulsion), and 80% lipid phase (cocoa butter and PGPR). During processing, the cocoa butter and PGPR were heated to 60°C, and the gelatin was dissolved in the water, also at a temperature of 60°C. The A Unit was set to 920 rpm and held at a constant temperature of 20°C, and the C Unit was set to 900 rpm and held at a constant temperature of 30°C. The throughput was set to 50 mL/min. This gives an average residence time of 48 seconds for the A unit, and 3 minutes for the C unit.

Table 8.1 NMR results for cocoa butter emulsions containing different percentages of PGPR (from 0.1 to 5% within the lipid phase). Analysis was conducted during the week following processing.

Percentage of PGPR	$d_{3.2}$ (μm)	Sigma (σ)	Free Water (%)
0.1	3.9	0.4	0
0.5	3.4	0.5	0
1	3.2	0.5	1.3
2	3.2	0.5	2.5
5	2.9	0.3	0.5

Table 8.1 shows the $d_{3.2}$ (μm) and the percentage of free water as measured by NMR droplet sizing for the emulsions. Slight differences in the droplet size as a function of PGPR content are observed. From 0.1% to 5% PGPR the droplet size decreases from 3.9 μm to 2.9 μm . Slight differences are also observed in the percentage of free water. However, differences in both the droplet size and free water content are not significant, and are within experimental error.

Even at 0.1% PGPR, it is likely that full coverage of the interface occurs. The percentage of emulsifier required to cover the interface of the dispersed phase as a monolayer can be estimated by calculating the surface area of the droplets exposed to the continuous phase per volume of emulsion (AN):

$$AN = \frac{6\phi}{d_{3.2}} \quad 8.1$$

where ϕ is the dispersed phase volume fraction, and $d_{3.2}$ is the surface-weighted mean droplet diameter (McClements, 2005).

The emulsion head group size can then be used to calculate the number of molecules for surface coverage, and thus the percentage of emulsifier required to gain a

monolayer at the droplet interface. This method was used to calculate the percentage of PGPR required to form a monolayer in an emulsion with 20% water (dispersed phase), with 5 μm diameter droplets, assuming that the head group size of PGPR is 1 nm^2 , which is in the range measured for tweens and spans (Apenten & Zhu, 1996; Henry, Fryer, Frith & Norton, 2009). In static conditions 0.01% PGPR would be required to form a monolayer. For an emulsion with 20% water and 2 μm droplets, 0.2% PGPR would be required to form a monolayer.

However, by adding an excess of emulsifier to the system it ensures that full coverage of the interface is achieved during the process, allowing for stable droplets to be produced. The results indicate that PGPR covers the interface more efficiently in the margarine line than the Silverson (see Chapter 7), probably because the residence time is longer, allowing longer for the emulsifier to reach the interface. Furthermore, stability of the emulsion is not limited to the percentage of PGPR, but is also affected by the quantity of gelatin used as it has surface activity, and the crystallisation of the fat during processing (as discussed later).

8.2.2 Gelatin Concentration

The quantity of gelatin used was investigated, in conjunction with the percentage of water within the system. Emulsions with 10 – 50% water were produced, with no gelatin, or with 2.5% or 5% gelatin. During processing, the cocoa butter and PGPR were heated to 60°C, and the gelatin was dissolved in the water, also at a temperature of 60°C. The A Unit was set to 920 rpm and held at a constant temperature of 20°C,

and the C Unit was set to 900 rpm and held at a constant temperature of 30°C. The pump speed was 50 mL/min.

As can be seen from Table 8.2, when the aqueous phase of the emulsion contained no gelatin, droplet size was not affected by the phase volume of water, with all the emulsions (containing 10 – 50% water) having small droplets (3-5µm), and very little, or no, free water (0-1%). With the addition of 2.5% gelatin to the aqueous phase, droplet size and free water content were comparable to that measured for emulsions containing no gelatin, with droplets measuring between and 3-5µm in size, with no free water. However, the addition of 5% gelatin to the aqueous phase resulted in large droplets and a high percentage of free water (i.e. droplets above 100µm) when the emulsion contained high quantities of water (i.e. 40 and 50% water). This was thought to be an outcome of a high phase volume of water, which resulted in a partial phase inversion, producing a bicontinuous system. This effect was not observed for lower quantities of gelatin. It is possible that at this percentage of gelatin, and aqueous phase volume, gelation is occurring within the process, preventing the production of small droplets. Equally, it may be as a result of competition for the interface between the PGPR (favouring oil continuous systems) and gelatin (favouring water continuous systems). For this formulation the processing parameters may need to be adapted.

As can be seen from the *Tpeaks* and melting enthalpies, there are no significant differences between the samples. All the samples have lower melting points than typically observed for crystals in form V (Loisel *et al.*, 1998), suggesting crystals in form IV. This is likely to be as a result of the processing temperatures, highlighting

the need to measure, and manipulate, the A and C unit outlet temperatures (as discussed later).

Table 8.2 Results acquired from both NMR and DSC for cocoa butter emulsions with different percentages of water, and different percentages of gelatin (from porcine skin; 250 bloom). Water content relates to the percentage of water in the overall formulation, whereas gelatin content relates to the percentage within the aqueous phase only. Analysis was conducted during the week following processing. Standard deviation is presented in brackets.

Water content (%)	$d_{3.2}$ (μm)	Sigma	Free Water (%)	T_{peak} ($^{\circ}\text{C}$)	Melting Enthalpy (J/g^{-1})
0% Gelatin					
10	3.7	0.6	0	27.4 (0.7)	95.9 (1)
20	4.8	0.5	0	26 (0.1)	93.3 (1.1)
30	4.3	0.5	0	25.8 (0.04)	91 (2.7)
40	5.0	0.6	1	26.4 (0.6)	88.6 (3.9)
50	3.8	0.4	0	25.7 (0.6)	75.9 (1)
2.5% Gelatin					
10	4.1	0.5	0	28.1 (0.3)	128.3 (3.7)
20	3.2	0.6	0	28.4 (0.7)	117.5 (7.9)
30	4.6	0.6	0	27.1 (1.1)	109.8 (4.1)
40	5.1	0.5	0	26.8 (0.2)	109.2 (4.7)
50	5.2	0.4	0	27.8 (0.8)	83.9 (8.9)
5% Gelatin					
10	4.1	0.7	0	28.7 (0.8)	120.3 (9.1)
20	3.2	0.7	0.4	26.7 (0.2)	94.6 (4.4)
30	2.9	0.6	1.3	26.8 (0.5)	88.6 (1.1)
40	31.3	0.9	19	26.7 (0.5)	93.2 (0.6)
50	35	0	55	26.5 (0.1)	53.1 (0.1)

Other than at higher aqueous phase volumes (i.e. 40 and 50% water), the percentage of gelatin had very little effect on droplet size. Clegg *et al.* (1996) found that the addition of gelatin to low-fat margarine spreads resulted in well emulsified W/O emulsions. The authors found that aqueous phase droplet size (as measured by confocal microscopy) was larger in emulsions containing 2% gelatin than in a control emulsion containing no gelatin (1-5 μm and 1-2 μm , respectively). A further increase to 4% gelatin did not increase droplet size further, although droplets appeared less

defined in shape (i.e. droplets were no longer spherical). It is thought that this occurs as a result of increased viscosity and gelation of the aqueous phase during processing.

Alexa, Mounsey, O’Kennedy and Jacquier (2010) showed that the addition of 7.5 g kg^{-1} of κ -carrageenan to the aqueous phase of a W/O spread resulted in an inhomogeneous droplet distribution, with larger and clustered droplets than observed with the addition of a smaller quantities of κ -carrageenan. The authors suggest that this is a result of increased aqueous phase viscosity. In order to determine whether this was the case in this system, cryo-SEM was carried out on a subset of the samples. Cryo-SEM and light microscope images are presented in Figure 8.2. Figure 8.2 a. b. c. and d. all show a continuous fat phase, with spherical water droplets covered by sintered fat crystals. It is clear from these images that this structure is common both across different phase volumes, and for emulsions containing different percentages of gelatin. Furthermore, these images are similar to the images presented in Figures 7.2 and 7.7, for emulsions containing no gelatin. The two light microscope images (Figure 8.2 e. and f.) give a clear indication of the microstructure, with no evidence of clustered droplets, although an inhomogeneous droplet distribution is evident for both samples (containing 20% and 40% aqueous phase). These images confirm the droplet size obtained via NMR, with the majority of droplets visually appearing to be below approximately $10\mu\text{m}$ in size.

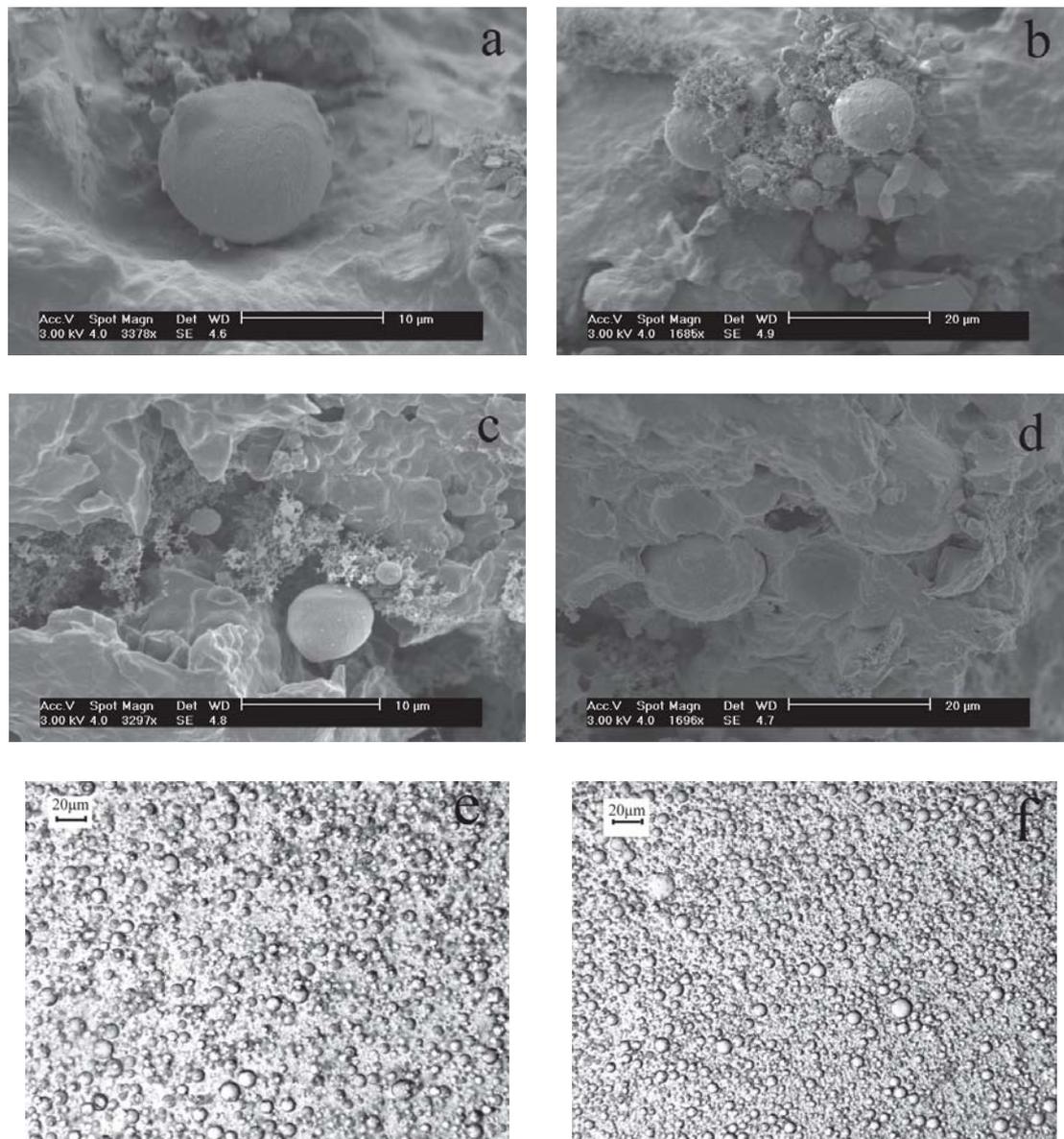


Fig. 8.2 Micrographs of cocoa butter emulsions: **a.** cryo-SEM image of a 30% aqueous phase emulsion containing no gelatin, **b.** a cryo-SEM image of a 30% aqueous phase emulsion containing no gelatin, **c.** a cryo-SEM image of a 40% aqueous phase emulsion containing 5% gelatin, **d.** a cryo-SEM image of a 10% aqueous phase emulsion containing 5% gelatin, **e.** a light microscope image of a 20% aqueous phase emulsion containing 2.5% gelatin, **f.** a light microscope image of a 40% aqueous phase emulsion containing 2.5% gelatin. Analysis was conducted within a week of the emulsion preparation.

8.3 Processing Parameters

8.3.1 A & C Unit Rotation Speeds

To investigate the effect of processing on emulsion and crystal properties a number of parameters were manipulated. All the emulsions produced contained 20% aqueous phase (of which 5% was gelatin), and contained 1% PGPR. The A and C unit rotation speeds were manipulated, from a relatively low speed (170 rpm) to the fastest achievable speed of the units (1300 rpm), to consider the effect on droplet size. Combinations of different speeds of the units were used to consider the relative impact of shear rates of both the units. The A unit temperature was set to 20°C, the C unit temperature was set to 30°C and the throughput was set to 50mL/min.

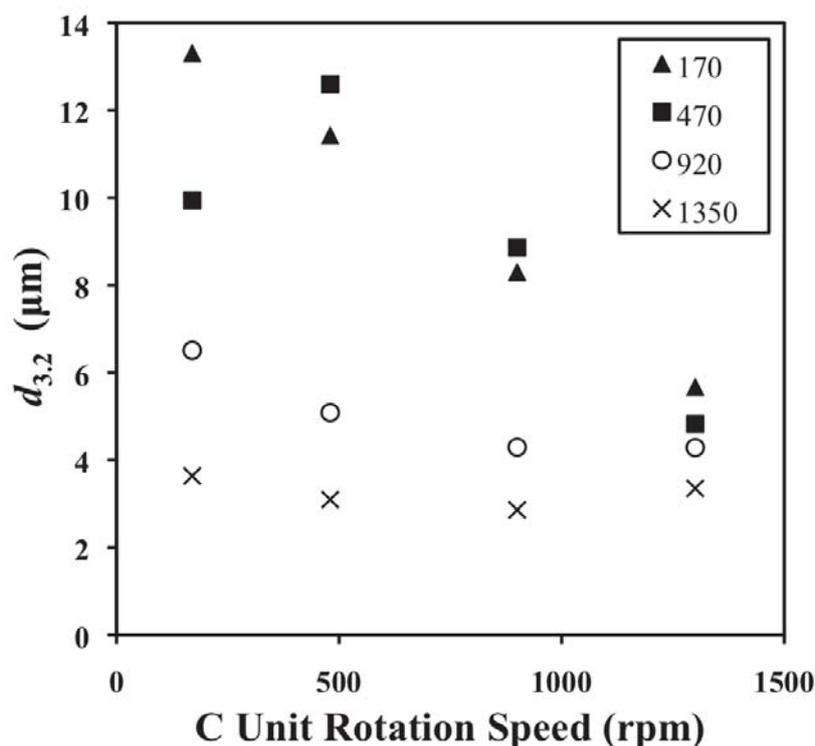


Fig. 8.3 Mean droplet size (μm) as determined by NMR analysis, as a function of varying both the A and the C Unit rotations speeds (rpm). A unit rotation speeds are given in the legend; C unit speeds are given on the X axis.

As can be seen in Figure 8.3 (additional data is given in Appendix 20), the unit rotation speeds had a significant effect on droplet size. By increasing both the A and the C unit rotation speeds, the droplet size is decreased. When the A unit speed was low (i.e. 170 rpm), increasing the C unit speed decreased the droplet size from 13.3 μm to 5.7 μm . However, once the A Unit speed was high (i.e. 1350 rpm), the C Unit speed has little effect on the droplet size. In this case, the small droplets produced in the A unit are comparable to the smallest possible droplets that can be produced in the C unit. Thus, no further reduction in droplet size was possible with the achievable shear rates in the pin stirrer. Heertje (1993) describes the importance of the shear experienced during the margarine line on the size of fat crystals produced, showing that strong shear in the C unit resulted in larger shell formation around the

water droplets. It was suggested that this is as a result of enhanced transport of fat crystals to the O/W interface, in addition to smaller crystals being produced. With no stirring in the C unit the crystals aggregate, and so are no longer available for Pickering. Heertje (1993) emphasises the importance of temperature, as over crystallisation can increase viscosity, resulting in coalescence, and a coarser emulsion; the fat should be in a liquid-like state to produce the smallest droplets.

Whilst changing the A and C unit rotation speeds has an effect on droplet size, it did not seem to affect the melting characteristics of the emulsion, with all samples melting at a similar temperature (between 27.1°C and 28.6°C), with no obvious relationship being observed (see Fig. 6.4).

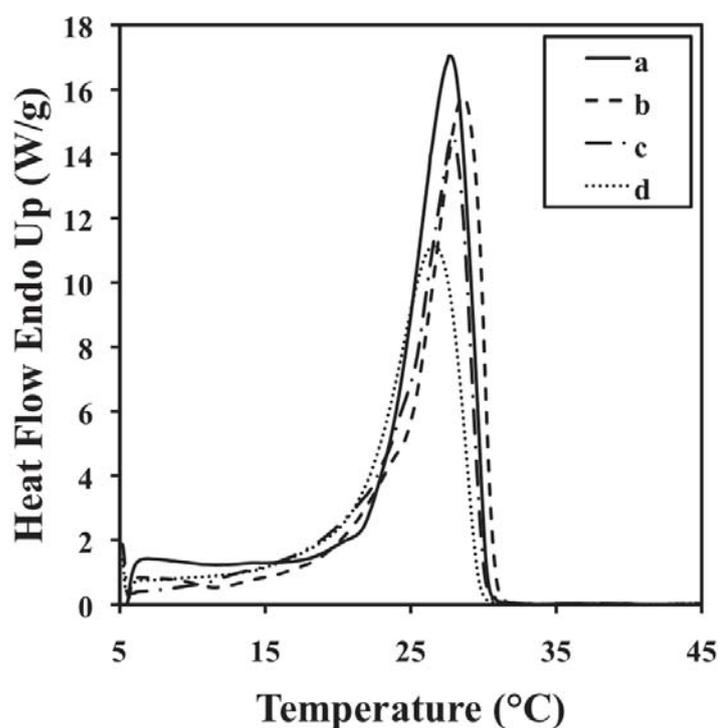


Fig. 8.4 DSC curves for emulsions processed under different conditions, **a.** A unit 170 rpm, C unit 170 rpm, **b.** A unit 470 rpm, C unit 480 rpm, **c.** A unit 920 rpm, C unit 900 rpm, **d.** A unit 1350 rpm, C unit 1300 rpm.

It is possible that a high shaft speed in A Unit would create small fat crystals that will improve bulk crystallisation following tempering; however, this was not observed. It is likely that the temperature of the units is more important in controlling the polymorphic form of the fat, as discussed in the previous chapter. Therefore, this was investigated and reported in the next section.

8.3.2 A & C Unit Temperatures

In order to determine the effect upon melting characteristics and droplet size, the jacket temperatures of the A unit and the C unit were manipulated. Process inlet temperature and unit outlet temperatures were measured in order to accurately record the emulsion temperature during the emulsification process. All the emulsions produced contained 20% aqueous phase (of which 5% was gelatin), and contained 1% PGPR. The A unit was set to 920 rpm, the C unit was set to 900 rpm, and the throughput was set to 50mL/min. In Figure 8.5 T_{peak} (°C) is plotted as a function of either A unit or C unit outlet temperature.

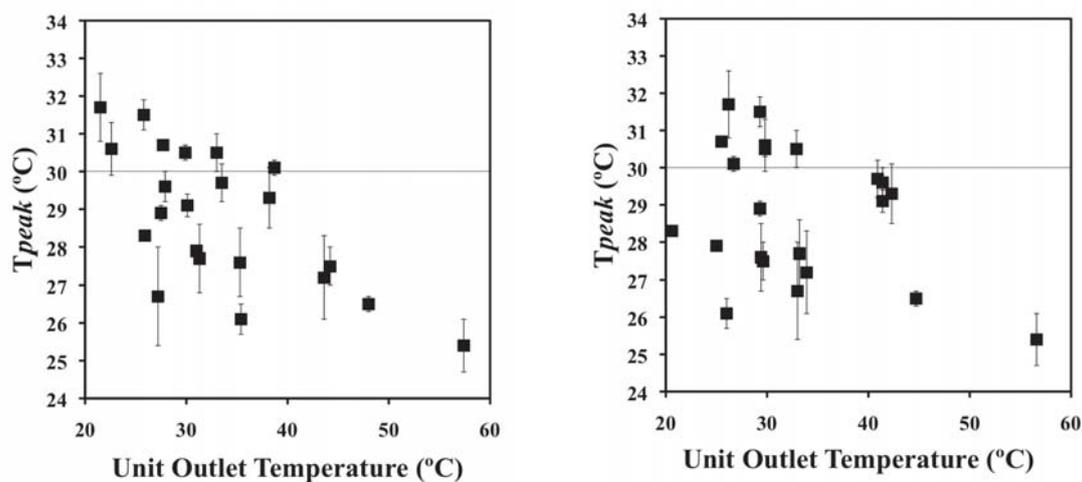


Fig. 8.5 T_{peak} (°C) as a function of A unit outlet temperature (left) and C unit outlet temperature (right). Error bars show one standard deviation of the mean. Values above the solid horizontal line represent samples that have crystals in form V.

As can be seen from Figure 8.5 (additional data is presented in Appendix 20), when the A and C Units are run at very high temperatures (i.e. 50 and 60°C) the melting temperatures of these samples is the lowest (26.5 and 25.4°C respectively). This is because no fat crystals are formed during processing, and are only formed during storage at 5°C. As such, crystals in unstable forms III or IV develop, as would be expected for cocoa butter that has not experienced tempering. Conversely, when the A Unit is run at the lowest temperature (i.e. 15°C) the highest peak temperature is observed (31.7°C), indicating the presence of crystals in form V. It can be seen from Figure 8.5 that there is not a strong relationship between C unit outlet temperature and T_{peak} . A weak relationship between the A unit outlet temperature and the T_{peak} (°C) can be seen. It suggests that an A unit outlet temperature of below 35°C resulted in emulsions that melted between 30 and 34°C, suggestive of crystals in form V. The results suggest that the A unit determines the crystal form, while the C unit maintains the crystal formed in the A unit, or melts any crystals formed in the A unit.

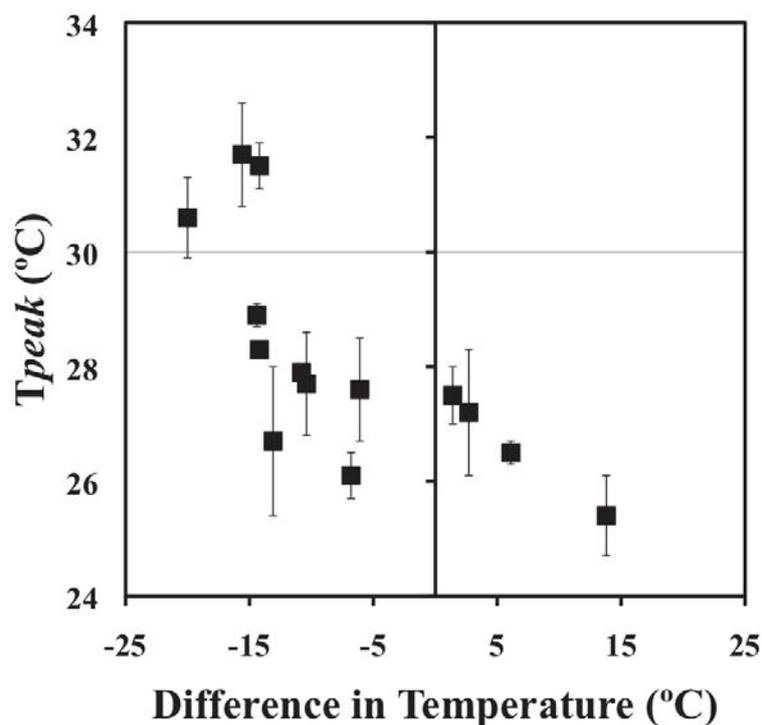


Fig. 8.6 T_{peak} (°C) as a function of the difference in temperature (°C) between the process inlet temperature and the A unit outlet temperature. A positive value refers to an increase in temperature from the inlet to the outlet, whereas a negative value refers to a decrease in temperature between the inlet and the outlet. Error bars show one standard deviation of the mean. Values above the solid horizontal line represent samples that have crystals in form V. DSC was conducted 24-48 hours after emulsification.

This finding was analysed further by considering the difference in temperature between the process inlet and the A unit outlet. The data is presented in Figure 8.6. It can be seen that a decrease in temperature of at least 15°C results in a T_{peak} of between 30 and 34°C. Thus, both the A unit outlet temperature, and the difference in temperature between the A unit inlet and the A unit outlet are important for crystal growth. It seems that if the crystallisation rate is fast, then the crystals formed are more stable and dominate the final polymorphic form. This is probably as a consequence of both the number and size of the crystals formed in the A unit. Therefore, to create a tempered cocoa butter emulsion the A Unit should be run at a

low temperature. However, with the current set-up this causes problems during processing, as cocoa butter solidifies in the pipes and causes pressure build-up.

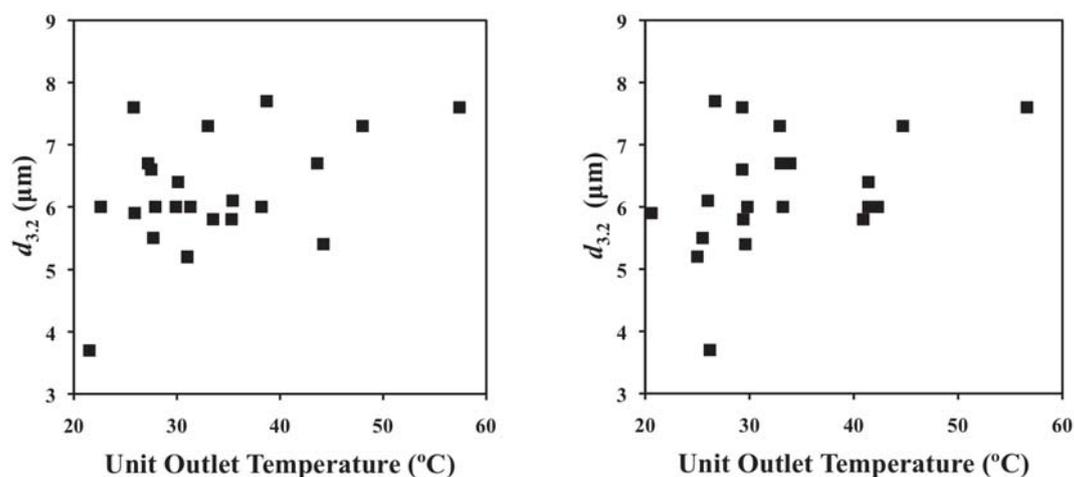


Fig. 8.7 Droplet size ($d_{3,2}$) as a function of A unit outlet temperature (left) and C unit outlet temperature (right).

The dependency of droplet size on outlet temperatures of the A and C units was studied, and is presented in Figure 8.7. At higher temperatures the emulsion should be less viscous, so the droplets would be mobile for longer, colliding and coalescing more than when the temperature is lower, and the emulsion is more viscous. Conversely, during the process the droplets are under shear, so if coalescence occurs droplets will break up again. In practise, all processing temperatures resulted in emulsions with the same droplet size (3 – 8 μm). These results suggest that the PGPR concentration, and shear during the emulsification process affect droplet size, and the crystals present have little effect. The role of the crystals at the interface only occurs after the emulsion is formed, and under static conditions (i.e. on storage).

8.3.3 Throughput

It might be expected that the throughput has an effect on droplet size as the total amount of shear experienced by the emulsion would be affected at any given rotation speed of the A and C units. All the emulsions produced contained 20% aqueous phase (of which 5% was gelatin), and contained 1% PGPR. The A unit was set to 920 rpm and 20°C, the C unit was set to 900 rpm and 30°C. Different pump speeds were investigated, the results of which are presented in Table 8.5 and Figure 8.8.

Table 8.5 Indicating *T_{peak}* and melting enthalpy of emulsions produced using different pump speeds. For all emulsions, the A unit was set to 20°C and 1350 rpm, and the C unit was set to 30°C and 1300 rpm.

Throughput (mL/min)				DSC		NMR		
	Inlet Temp (°C)	A Unit Outlet Temp (°C)	C Unit Outlet Temp (°C)	<i>T_{peak}</i> (°C)	Melting Enthalpy (J/g ⁻¹)	<i>d</i> _{3.2} (µm)	Sigma	Free water (%)
30	39.3	24.5	29.3	27.3 (0.3)	129.3 (2.6)	2.7	0.7	0
60	42.3	25.7	29.5	26.5 (0.2)	100 (1.1)	3.8	0.5	1
100	45	27.5	29.6	26.5 (-)	108 (-)	4.3	0.6	5
150	47.5	29.9	29.8	27 (0.5)	135.8 (1)	4.5	0.7	1.1
200	48.7	32.5	30.2	27.6 (0.5)	127.7 (4.2)	5.7	0.8	0

As can be seen in Figure 8.8, droplet size increases in a linear fashion over the range of throughputs selected. This data shows that the residence time, and the total amount of shear experienced by the emulsion is important in allowing droplet break up, due to the time for the PGPR to get to the droplet interface.

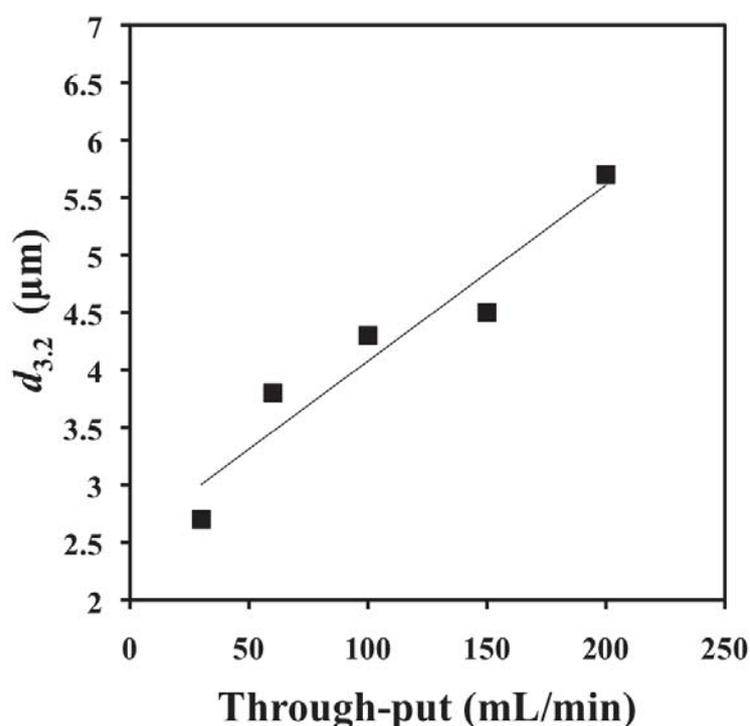


Fig. 8.8 Droplet size as a function of pump speed (mL/min).

The crystal form is less affected by the throughput as can be seen in Table 8.5: only slight differences in T_{peak} are observed as the throughput is increased. This data suggests that crystals in forms III and IV are formed. Although slight differences in melting enthalpy were observed, these are within the experimental error, as discussed in Chapter 7. Furthermore, the data is consistent with that presented in the previous section (see section 8.3.2), where temperature was studied systematically; polymorphic form is dependent on the outlet temperature of the units and the difference in inlet and outlet temperature of the A unit.

8.4 Conclusions

The aim of this the work presented in this chapter was twofold: i) to explore changes in cocoa butter emulsion formulation (i.e. the percentage of PGPR used, and the

addition of gelatin), and ii) to investigate the processing method (the margarine line) by manipulating shaft speeds, unit temperatures and process throughput.

Slight differences in droplet size were observed as a function of PGPR content. From 0.1% to 5% PGPR droplet size decreased from 3.9 μm to 2.9 μm , in addition to some variation in the percentage of free water. However, these differences are not thought to be significant. Even at 0.1% PGPR with 20% aqueous phase, full coverage of the interface is achieved. Further work could consider PGPR contents lower than the 0.1% level investigated here, or could investigate blends of lecithin and PGPR (Schantz & Rohm, 2005). This approach may be favourable when producing the emulsion for the market, as strict regulations exist within the food industry for the use of PGPR.

The addition of gelatin, and the quantity of gelatin used, was also investigated. Very little difference was observed between emulsions containing pure water (i.e. no gelatin) or 2.5% gelatin within the aqueous phase, with small droplets (3-5 μm), and very little free water (0-1%). However, emulsions containing higher phase volumes of water (40 and 50%), and 5% gelatin had large droplets, and much higher percentages of free water, which was thought to indicate partial phase inversion as a result of the surface activity of the gelatin, or gelation during the process. The results suggest that the addition of gelatin does not offer an advantage over using pure water. In fact, at the higher concentration of gelatin the emulsion had larger droplets and more free water, which would inevitably affect the physical and microbial stability of the emulsion, and the taste. Furthermore, there are likely to be issues surrounding the use of animal derived products in foods that are typically vegetarian. It is also important

to try to monitor or reduce the number of ingredients within food products (i.e. clean labelling), and consumers may believe that certain ingredients do not ‘belong’ in chocolate. In Chapter 3, when participants were asked to imagine a product that is both healthy and indulgent, many expressed concern that the company would use additional ingredients that are not good for one’s health, and as such would be suspicious of the product. That said, there may be benefits of using a gelling agent to impart solidity on the aqueous phase: on consumption the water would then no longer flow, giving it a mouth feel closer to fat. This highlights the need to run sensory tests, with a sensory panel or consumers, to determine whether the water within the emulsion can be detected, and whether the use of gelling agents affects the sensory attributes of the emulsion (or chocolates containing emulsions). Thus, future work should continue to investigate the use of hydrocolloids to gel the aqueous phase, but gelling agents preferably should not be animal derived, should have similar melting characteristics to cocoa butter, and should not have a high energy density (in order to keep the calorie content of the aqueous phase low).

A and C unit rotation speeds had a significant effect on droplet size ($d_{3,2}$), with faster rotation speeds resulting in smaller droplets. However, if small droplets were produced in the A unit, the C unit had little effect on droplet size. The rotation speeds of the A and C units did not affect melting characteristics of the cocoa butter.

It has been shown that the A unit outlet temperature, and the difference in temperature between the inlet and the A unit outlet was important for crystal form, with A unit outlet temperatures of below 35°C, and a decrease in temperature of at least 15°C between the inlet and the A unit outlet resulting in a T_{peak} of between 30 and 34°C,

indicative of crystals in form V. No correlation between A or C unit outlet temperature and droplet size was observed, with all temperatures resulting in emulsions with droplets of between 3 and 8 μ m in size.

Finally, the throughput had an effect on droplet size, with slower speeds resulting in smaller droplets as the residence time and amount of shear experienced by the emulsion was increased.

The margarine line shows promise for the production of cocoa butter emulsions, and offers a one-step process for both the emulsification and tempering of the emulsions.

9. Scaling up the emulsification process to pilot plant scale, and investigating the addition of dry chocolate ingredients

9.1 Introduction

In Chapter 7 and 8 the production of W/O cocoa butter emulsions was described, using both a high shear mixer and a margarine line (scraped-surface heat exchanger and a pin stirrer), with varying aqueous phase volumes (10 – 50%), different emulsifiers and emulsifier concentrations, with or without the addition of gelatin. Furthermore, the processing parameters of the margarine line were investigated, including shaft speeds and temperatures, and throughput. It was shown that cocoa butter emulsions can be produced that have small droplets (typically an average $d_{3,2}$ of 1 - 8 μ m). Furthermore, the temperature and shear of the margarine line can be manipulated to produce emulsions with ‘tempered’ fat.

As a result it was decided to carry out a scale-up trial, to i) investigate the scalability of the current emulsification process (to pilot plant scale), ii) to investigate the addition of the other chocolate ingredients (sugar, milk powder and cocoa solids) to move closer toward producing a chocolate, and iii) to consider how the addition of milk fat (to replace part of the cocoa butter) may affect emulsion properties, as milk fat is often used in chocolate formulations. Furthermore, as a food grade pilot plant was used, any products made during the trial could be consumed, in order to consider the taste and texture of the product.

This study was a challenge, as different equipment was used to produce the emulsions: two scraped-surface Freon cooled heat exchangers and a pin stirrer (with no temperature control) were used, as apposed to the two temperature controlled units used during the lab scale experiments. Furthermore, the increase in scale meant that different heat transfer and shear conditions would be experienced during the process, possibly affecting the emulsion produced. Three formulations were used, comprising two percentages of water (20 and 40%), with or without the addition of milk fat (see Table 9.1).

Table 9.1 Recipes used for each of the formulations used during the scale up experiments. Potassium Sorbate (55g) was added to each of the formulations, in addition to the ingredients below.

Emulsion		Formulation 1: 20% aqueous phase		Formulation 2: 40% aqueous phase		Formulation 3: 20% aqueous phase	
		(%)	(kg)	(%)	(kg)	(%)	(kg)
Lipid phase		80	44	60	33	80	44
	Cocoa butter	76.8	42.24	57.6	31.68	64.3	35.365
	PGPR	3.2	1.76	2.4	1.32	3.2	1.76
	Milk fat	-	-	-	-	12.5	6.875
Aqueous phase		20	11	40	22	20	11
	Water	18	9.9	36	19.8	18	9.9
	Gelatin (250 Bloom)	2	1.1	4	2.2	2	1.1
Weight (kg)		-	55	-	55	-	55

These formulations are similar to those investigated in Chapter 8: here 20 and 40% aqueous phase, with 4% PGPR within the lipid phase (at the top end of the range studied in Chapter 8, enabling full coverage of the interface), and 10% gelatin within the aqueous phase (higher than studied in Chapter 8, but allowing for harder droplets to be formed, providing that the temperature within the unit allowed for gelation). The addition of milk fat was not considered in Chapter 8, neither was the addition of

potassium sorbate (a preservative that was required in order to adhere to the food safety regulations of the lab). A full chocolate was produced by adding a dry mix of sugar, skimmed milk powder, cocoa powder and vanilla (see Table 9.2) to the emulsion, following emulsification, using a planetary mixer (Hobart).

Table 9.2 The ratio of dry mix to emulsion, and the percentage of different ingredients within the dry mix.

Milk Chocolate	
	(%)
Dry Mix	63.84
Sugar	65.15
Skimmed Milk Powder	26.16
Cocoa Powder	8.68
Vanilla	0.01
Emulsion	36.16

As detailed in the Material and Methods chapter (see Chapter 6), a number of parameters were kept constant (i.e. the feed vessel was kept at 60°C and 120rpm, the throughput was at a constant 0.75kg/min, and the shaft speeds of the two Freon cooled units were 600rpm). However, other parameters were altered, details of which can be found in Table 9.3.

Table 9.3 Processing parameters used for each of the emulsions produced during scale-up experiments.

	Sample	Flow	Temperature (°C)			Speed (rpm)	
		Pressure (bar)	Pre-emulsion	Freon 1 outlet	Freon 2 outlet	Pin Stirrer outlet	Pin Stirrer
20% aqueous phase	1	4-5	52	21.5	24.9	29.1	400
	2	15	52	18	23.4	-	400
	3	20	52	17.2	-	28.5	400
	4	8-9	49.5	20.5	22	28.3	400
	5	14-15	46.5	17.6	20.8	28.5	400
	6	20	44.5	16.6	19.8	28.3	400
	7	25	44	16.3	-	28	400
40% aqueous phase	8	2-3	59.8	21.8	25.2	29	400
	9	10	57	20.2	23.7	28.4	400
	10	20	56	17.2	20.1	27.8	400
	11	20	56	17.2	20.1	29	800
20% aqueous phase (+milk fat)	12	4	57.5	19	19.5	27	400
	13	7	56.4	16	18	26	400
	14	21	56	16	18	25.1	400
	15	-	-	16	18	-	200

9.2 Emulsions

Emulsions were produced and collected, and stored in both ambient (approximately 20°C) and chilled (approximately 6°C) conditions. They were assessed by a number of analytical techniques. The emulsions were visually assessed by eye to determine whether any free water was present, and observed using cryo-SEM. DSC was used to analyse melting profile, and XRD was used to calculate d spacings (Å), which were compared with data found in the literature (see Chapter 1). NMR was used to assess droplet size distribution, and ‘free water’ content. The moisture content of the samples was also assessed using a gravimetric method.

The emulsions were moulded, and contracted from the mould after being cooled for 12 hours. They were hard, snapped well and were glossy, indicating a well tempered emulsion in crystal form V. All the emulsions were produced successfully, all being fat continuous with no water loss on storage (i.e. no pooling at the bottom of the sample) and no visible water loss when the samples were snapped. These results indicated that the laboratory scale process can be scaled up successfully, even when using different equipment (i.e. two scraped surface heat exchangers and a pin stirrer with no temperature control, rather than the laboratory scale equipment discussed in Chapters 7 and 8).

All of the emulsions were assessed using NMR droplet sizing after 1, 2, 6 and 12 weeks to determine whether any coalescence was occurring over time (all the data obtained are given in the Appendix 21). Droplet size ($d_{3,2}$) and percentage of free water obtained by NMR are shown in Figure 9.1 and 9.2, respectively. Data are means across all processing parameters for formulation 1 (20% aqueous phase), 2 (40% aqueous phase) and 3 (20% aqueous phase, with milk fat in addition to cocoa butter). As can be seen in Figure 9.1, all the samples had small droplets (2.7 – 6.7 μm), which did not change over a period of 12 weeks.

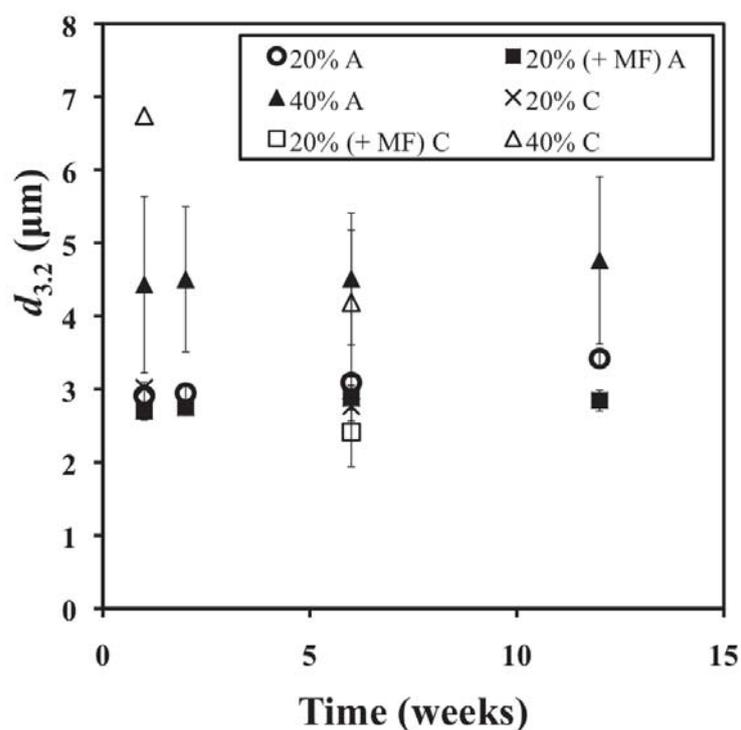


Fig. 9.1 Mean droplet size ($d_{3,2}$) as a function of storage time for emulsions containing 20% aqueous phase (formulation 1), 40% aqueous phase (formulation 2), and 20% aqueous phase with milk fat in addition to cocoa butter (formulation 3), stored in both ambient and chilled conditions. See Table 9.1 for details of the formulations used. The samples were stored at two temperatures: 'A' refers to ambient conditions, 'C' refers to chilled conditions. 'MF' refers to milk fat. Error bars show one standard deviation of the mean.

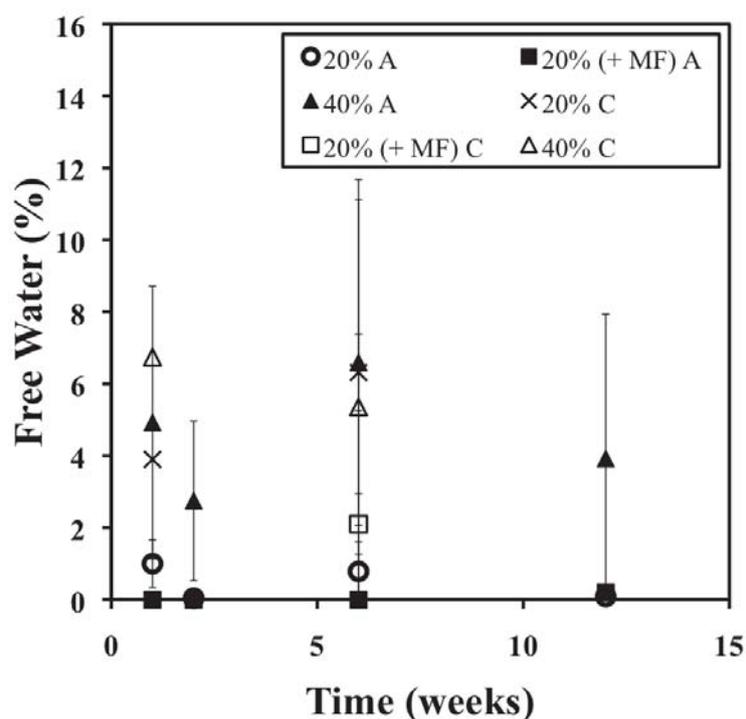


Fig. 9.2 Mean percentage of free water (i.e. droplets above $100\mu\text{m}$ in size) as a function of storage time for emulsions containing 20% aqueous phase (formulation 1), 40% aqueous phase (formulation 2), and 20% aqueous phase with milk fat in addition to cocoa butter (formulation 3), stored in both ambient and chilled conditions. See Table 9.1 for details of the formulations used. 'A' refers to ambient conditions, 'C' refers to chilled conditions, and 'MF' refers to milk fat. Error bars show one standard deviation of the mean.

Greater variation in both droplet size and percentage of free water are observed for samples containing 40% aqueous phase. This is partly as a result of the speed of the pin stirrer being changed (800 rpm for sample 11, as apposed to 400 rpm for the other samples). Smaller droplets are observed for this sample after 1 week ($3.1\mu\text{m}$ as apposed to $3.8\text{-}5.8\mu\text{m}$), and remain smaller over the 12 weeks than the droplet size of the other emulsions containing 40% aqueous phase. However, this does not account for the amount of variation observed; it is thought that the higher water content resulted in samples that were not as homogeneous as emulsions containing 20% aqueous phase.

The droplet size observed during scale up was similar to the droplet size measured during laboratory trials (see Chapters 7 and 8), where droplet size was 3 – 5 μ m when the emulsion contained 20% aqueous phase, and approximately 5 μ m when the emulsion contained 40% aqueous phase.

There was very little difference in droplet size or size distribution for samples stored in ambient or chilled conditions, although some difference in the percentage of free water was observed. The percentage of free water varied somewhat between the samples, with some containing no free water, and others between 7.2 and 8.5%. Larger amounts of free water were observed for samples containing 40% aqueous phase, which is to be expected.

The smallest droplets (between 2 and 3 μ m), and the smallest percentage of free water (0%) were observed for samples containing milk fat in addition to cocoa butter. This is possibly as a result of reduced viscosity of the lipid phase, which allowed emulsifiers to reach the interface quicker. However, the outlet temperatures (see Table 9.3) were also lower for these samples than for the other samples, which would have allowed for more crystallisation of the fat during homogenisation, preventing coalescence.

Droplet size changed very little over the time course of the experiment (i.e. from 1 to 12 weeks; see Fig. 9.1). Any variations might result from sampling differences i.e. parts of the sample had less or more pools of water (characterised as free water), than other parts, resulting in differences in measured free water percentage and droplet size.

Six weeks after emulsification a subset of the samples was heated beyond the melting point of the fats (to 40°C), and held at this temperature over the course of 5.5 hours, with measurements being taken at intervals (see Table 9.4). This experiment was conducted to determine how the sample stability depends upon the crystal shells at the interface, which will melt at this temperature (cocoa butter is molten at 40°C).

Table 9.4 Mean droplet size, sigma and free water content for samples heated to 40°C and held at this temperature for 1, 2, 3 and 5.5 hours. Samples were 6 weeks old. See Table 9.1 for details of the formulations used.

Formulation	1 hour at 40°C			2 hours at 40°C			3 hours at 40°C			5.5 hours at 40°C		
	$d_{3,2}$ (μm)	Sigma (σ)	Free Water (%)	$d_{3,2}$ (μm)	Sigma (σ)	Free Water (%)	$d_{3,2}$ (μm)	Sigma (σ)	Free Water (%)	$d_{3,2}$ (μm)	Sigma (σ)	Free Water (%)
1	2.87	0.2	0.3	3.14	0.101	0	3.17	0	1.2	3.07	0.22	-
2	3.62	0.46	12.7	4.05	0.54	19.9	3.93	0.57	24.8	3.86	0.59	27.5
3	2.98	0	0	2.91	0	0	3.13	0	0	3.07	0	0

The data shows that both samples containing 20% aqueous phase (i.e. formulations 1 and 3) were stable when heated to 40°C, with no increase in droplet size or percentage of free water. The emulsion containing 40% water (i.e. formulation 2) was unstable even after 1 hour at 40°C, with an increase in free water as droplets coalesce due to melting of the fat crystals at the interface. This behaviour suggests that there is a limited availability of PGPR for the amount of water within the sample. Surprisingly, although pooling of cocoa butter on the surface was observed for the emulsion containing milk fat (visually seen as a difference in colour), the droplet size remained constant over the course of 5.5 hours, and the percentage of free water did not increase, suggesting that the emulsifiers are imparting droplet stability into the system.

To determine the exact amount of water in the samples, a subset of the samples was also heated to 110°C in a dry oven for eight hours, twelve hours and thirty hours, to melt the crystals, break the emulsion and evaporate all of the water within the sample. The calculated percentage weight loss is given in Table 9.5.

Table 9.5 Results of the gravimetric method, in which the samples were weighed, before being placed in a dry oven at 110°C for 8, 12 and 30 hours. The total weight loss (%) is given; standard deviation (of duplicates) is presented in brackets. See Table 9.1 for details of the formulations used.

			Total weight loss (%)		
Sample			8 hours	12 hours	30 hours
Formulation 1	1	Ambient	15.4 (0.34)	15.5 (0.34)	15.49 (0.34)
		Chilled	17.63 (0)	17.74 (0)	17.72 (0.01)
	5	Ambient	16.72 (0.02)	16.79 (0.02)	16.78 (0.03)
		Chilled	17.81 (0.1)	17.91 (0.11)	17.9 (0.11)
Formulation 2	9	Ambient	10.5 (7.09)	12.47 (6.89)	23.81 (6.0)
		Chilled	30.37 (1.2)	31.66 (1.2)	32.95 (0.09)
	11	Ambient	30.0 (3.67)	31.05 (2.34)	32.41 (0.41)
		Chilled	16.08 (3.28)	17.61 (3.56)	20.46 (3.28)
Formulation 3	12	Ambient	5.43 (0.22)	6.71 (0.82)	10.84 (2.81)
		Chilled	17.34 (0.27)	17.42 (0.27)	17.4 (0.27)
	14	Ambient	14.89 (1.79)	14.96 (1.8)	14.95 (1.79)
		Chilled	16.5 (0.32)	16.58 (0.32)	16.58 (0.31)

For the samples containing 20% aqueous phase (i.e. 18% water & 2% gelatin), the percentage weight loss was very close to the percentage of water within the emulsion. For the ambient samples, approximately 2.5% of the water added to the emulsion was lost before being heated, whilst the chilled samples lost less than 0.5% water prior to the experiment.

For samples containing milk fat, and 20% aqueous phase, some variability was observed. Sample 12 (stored in ambient and chilled conditions) lost 7% and 0.6% water, respectively, prior to being heated, whilst sample 14 (stored at ambient and

chilled) lost 3.1% and 1.4%, respectively. However, again only a small amount of the water was lost before the experiment.

For samples containing 40% aqueous phase, much less of the original water still remained in the sample prior to being heated. For sample 9, 23.8% water loss was observed after 30 hours, suggesting that over 12% of the original 36% water was lost prior to the experiment. For the same sample stored in chilled conditions, 3% of the water had been lost prior to the experiment. For sample 11, at ambient approximately 3.6% of the original water was lost before the study, whilst nearly 16% of the water was lost from the same sample stored under chilled conditions. This data can be compared with the NMR data, which showed that the emulsions containing 40% aqueous phase had larger droplets and contained lakes of free water. Thus, it would be expected that evaporation would occur on storage, and that sampling would give greater variability.

In order to determine the polymorphic form of the cocoa butter within the emulsion, both DSC and XRD measurements were carried out. DSC experiments were performed after 1 day, 1 week and 1 month.

DSC curves are given in Figure 9.3 for the 20% (with, and without milk fat) and 40% aqueous phase emulsions. This shows that after 1 day all the samples have a melting temperature between 32 and 33°C, which is typical of crystal form V.

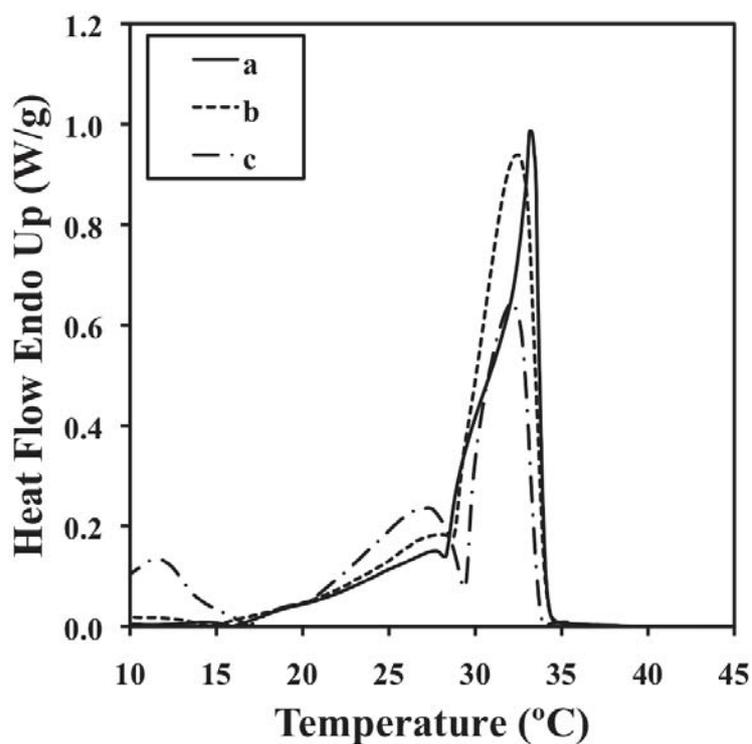


Fig. 9.3 DSC curves indicating the melting profile of three samples, **a.** formulation 1 containing 20% aqueous phase, **b.** formulation 2 containing 40% aqueous phase, and **c.** formulation 3 containing 20% aqueous, with milk fat in addition to cocoa butter. See Table 9.1 for details of the formulations used. DSC was conducted after 1 day stored at ambient temperature. Samples were heated from 5°C to 45°C at a rate of 2°C/min.

Figure 9.4 shows the time dependency of the DSC curves for a range of samples, stored in both ambient and chilled conditions. They all show melting temperatures typical of form V and they are all stable over a month storage time.

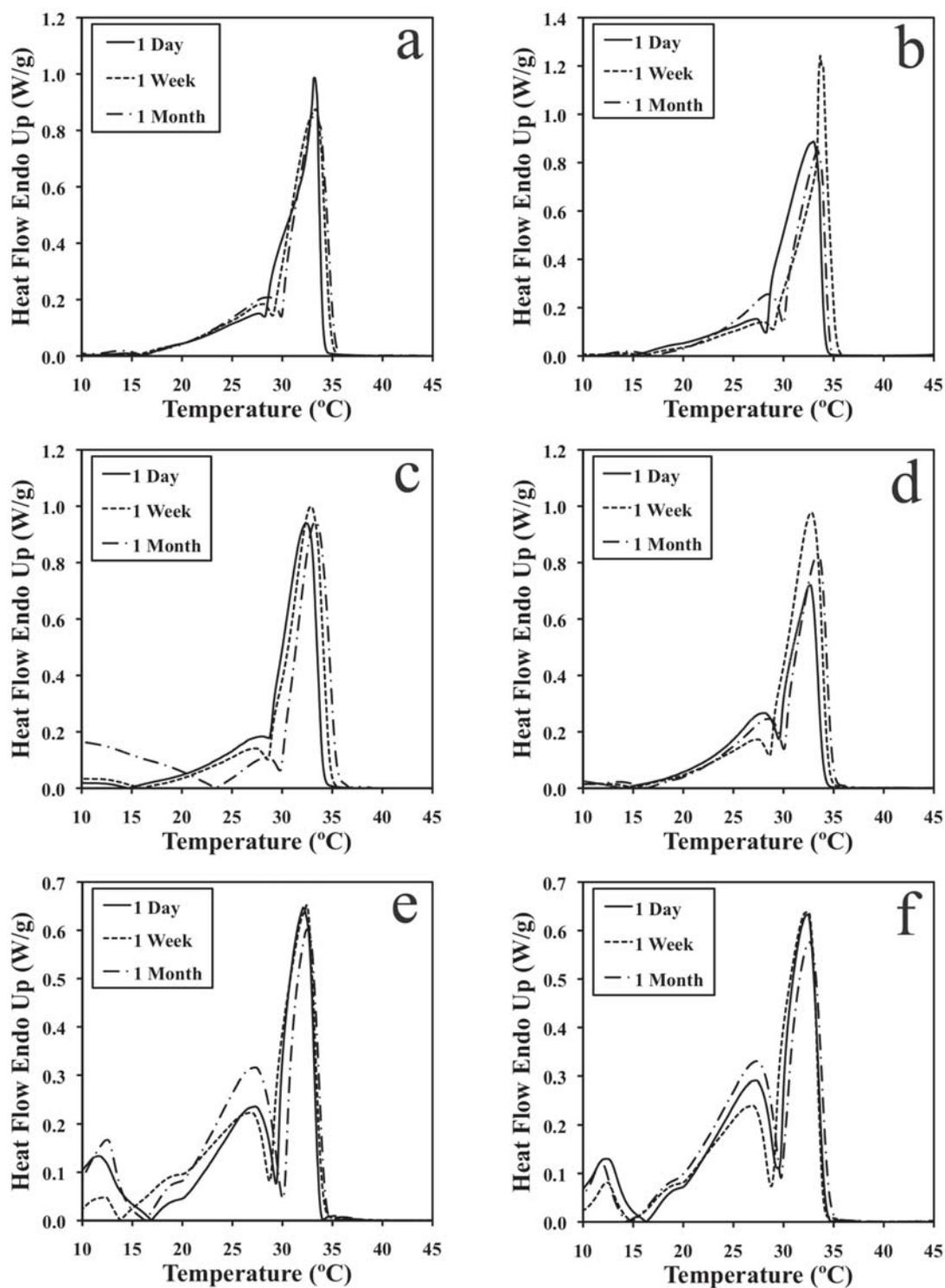


Fig. 9.4 DSC curves for three samples stored in either ambient or chilled conditions. a). sample 1 in ambient; b). sample 1 in chilled; c). sample 8 in ambient; d). sample 8 in chilled; e). sample 14 in ambient; f). sample 14 in chilled. See Table 9.3 for details of the processing parameters investigated. Samples were heated from 5°C to 45°C at a rate of 2°C/min.

Mean T_{peak} ($^{\circ}\text{C}$) and enthalpies (J/g) are given in Figures 9.5 and 9.6, respectively. As shown by Figure 9.5 (and the table presented in Appendix 21) all the samples, regardless of water content, had melting peaks suggestive of form V crystals (i.e. melting between 30 and 34 $^{\circ}\text{C}$). However, some variation in melting enthalpy was observed (see Fig. 9.6). A melting enthalpy of 117.5 J/g is typical of form V (Chapman, et al., 1971). However, the enthalpy calculated is very dependent upon the baseline used, and as can be seen in Figure 9.4, there are large differences in the heat flow. Furthermore, given that it was shown that some samples had lost water following emulsification (see Table 9.5), the melting enthalpy may have been inaccurately calculated when normalising for fat content (i.e. for the weight of cocoa butter within the sample).

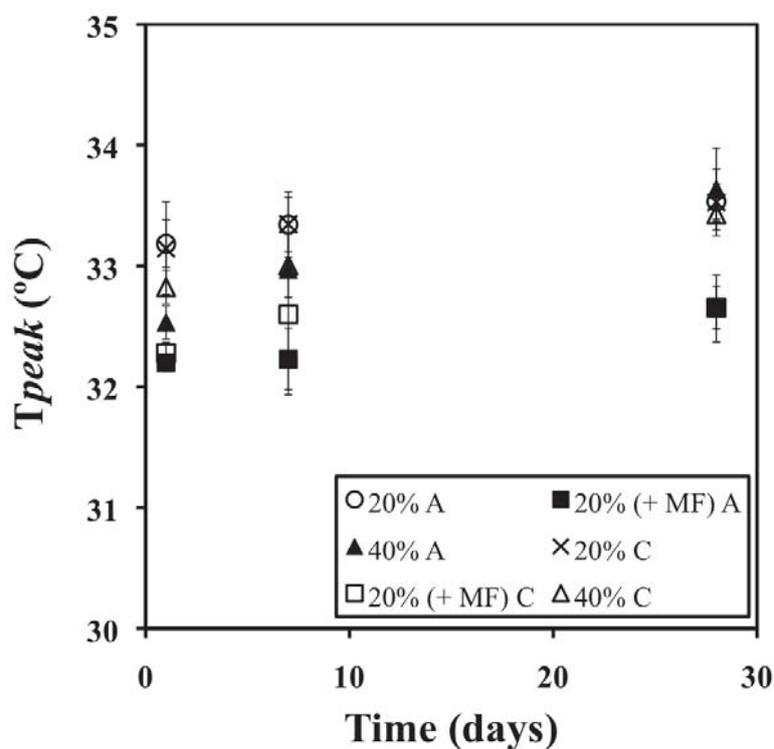


Fig. 9.5 Mean T_{peak} ($^{\circ}\text{C}$) as a function of storage time for emulsions containing 20% aqueous phase (formulation 1), 40% aqueous phase (formulation 2), and 20% aqueous phase with milk fat in addition to cocoa butter (formulation 3), stored in both ambient and chilled conditions. See Table 9.1 for details of the formulations used. Error bars show one standard deviation of the mean.

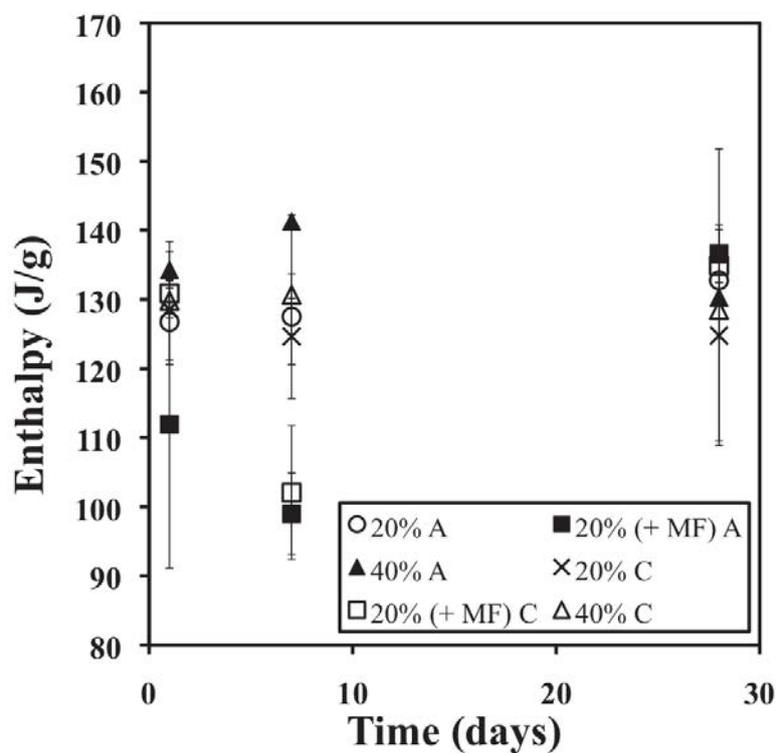


Fig. 9.6 Mean enthalpies (J/g) as a function of storage time for emulsions containing 20% aqueous phase (formulation 1), 40% aqueous phase (formulation 2), and 20% aqueous phase with milk fat in addition to cocoa butter (formulation 3), stored in both ambient and chilled conditions. See Table 9.1 for details of the formulations used. Error bars show one standard deviation of the mean.

As the enthalpies were on average higher than expected for form V, XRD measurements were carried out on a selection of samples. Figure 9.7 shows the data obtained for emulsions containing 20% aqueous phase, 40% aqueous phase, and 20% aqueous phase with milk fat in addition to cocoa butter, stored in both ambient and chilled conditions.

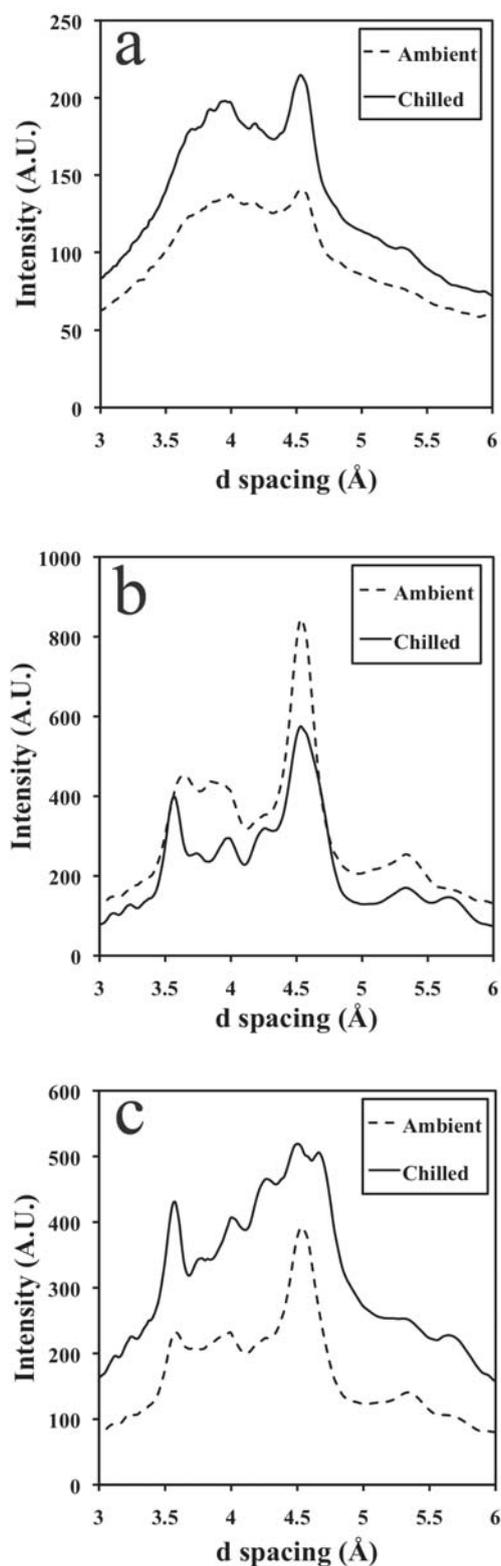


Fig. 9.7 X-Ray Diffraction: a). an emulsion containing 20% aqueous phase (formulation 1), b). an emulsion containing 40% aqueous phase (formulation 2), c). an emulsion containing 20% aqueous phase, with milk fat in addition to cocoa butter (formulation 3). See Table 9.1 for details of the formulations used. The measurements were made nine weeks after processing.

Table 9.6 X-ray diffraction short spacings typical of cocoa butter polymorphisms IV, V and VI (in addition to inconclusive peaks, and peaks atypical for cocoa butter) observed for emulsions, with the strength of peak. Similar classifications to those used by Wille & Lutton (1966) are used, where W=weak, M=medium, S=strong, and V=very. See Table 9.3 for details of the processing parameters investigated. 'A' refers to samples stored in ambient conditions; 'C' refers to samples stored in chilled conditions.

		1		5		6		8		10		12		14		
	Short Spacing (Å)	A	C	A	C	A	C	A	C	A	C	A	C	A	C	
Form IV	4.15		M	M	W											
	4.35		VW	W												
Form V	3.39									VW		VW			VVW	
	3.67	W				M		W								
	3.75			VW			VW		W	VW		VW	W			
	3.87				M			W								
	3.98	M	VS	S	S	S			M	S						
	4.23							VW								
4.58	VS	S	VS	S	VS	S	VS	VS								
Form VI	3.70				VW											
	3.86			M												
	4.04						W			W	W	M	VVW	VW		
	4.27						M		W		VW	M				
Inconclusive	3.69									M						
	3.84									VW		VW				
Atypical	3.12 / 3.13 / 3.15						VW		VW		W		VW	VW	VW	
	3.24 / 3.25 / 3.26						VW		VW		W		VW	VW	VW	
	3.56 / 3.58 / 3.59						S		S		S	W	S	M	M	
	4.50										S					
	4.66						VS				M		M			
	3.08														VW	
	5.06						VW									
	5.30 / 5.33 / 5.34						W	VW	W	W	W	W	VW	VW	VW	VW
	5.64 / 5.68							W		W		W		VW		

The XRD short spacings (\AA), as presented in Table 9.6 and Figure 9.7, were compared with Wille and Lutton (1966) data on the polymorphisms of cocoa butter (see Table 1.5). All of the samples showed peaks typical of cocoa butter in polymorphic form V, whereas some samples showed signs of crystals in forms III, IV or VI (see Table 9.6), and some peaks were not typical for any of the polymorphs observed by Wille and Lutton (1966).

All the samples had a strong, or a very strong peak around 4.58\AA (ranging from 4.53\AA to 4.58\AA), indicating crystals in form V. Some samples also had peaks at other *d*-spacings typical of form V, including 3.67\AA (samples 1 ambient, 6 ambient and 8 ambient), and 3.98\AA (samples 1 ambient, 5 ambient, 5 chilled, and 8 chilled). A *d*-spacing of 3.75\AA is only observed for cocoa butter crystals in form V, and is also observed for numerous samples. Three of the samples (1 chilled, 5 ambient and 5 chilled) had peaks typical of form IV crystals at approximately 4.15\AA and 4.35\AA , although these peaks were weak or medium, rather than strong or very strong as cited by Wille and Lutton (1966). The majority of the samples also had some *d*-spacings typical of form VI, including sample 5 ambient which had a medium peak at 3.86\AA , sample 5 chilled which had a very weak peak at 3.71\AA (although a strong peak is normally detected in this area), and numerous samples at had peaks around 4.04\AA and / or 4.27\AA .

Three of the samples also had inconclusive peaks i.e. peaks which could indicate crystals in various polymorphic forms. For example, sample 10 ambient had a medium peak at 3.69\AA , possibly indicating crystals in form V (3.67\AA weak) or form VI (3.70\AA strong). Very weak peaks at 3.84\AA were also observed for samples 10

chilled and 12 chilled, which may indicate crystals in form III (3.86Å), form IV (3.81Å), form V (3.87Å) or form VI (3.86Å).

A number of peaks atypical of cocoa butter crystals in any of the polymorphic forms were observed for the majority of the samples. Whilst most were weak or very weak, a number of medium, or strong peaks were seen. Samples 12 and 14 showed many more atypical peaks, possibly as a result of the milk fat in these samples.

The XRD data has suggested that for all of the emulsions the predominant polymorphic form is form V. However, some of the samples show peaks typical of IV crystals, suggesting that full tempering has not occurred in all cases, and some peaks typical of forms VI, which may be expected given the nine week period before emulsification and taking the measurements.

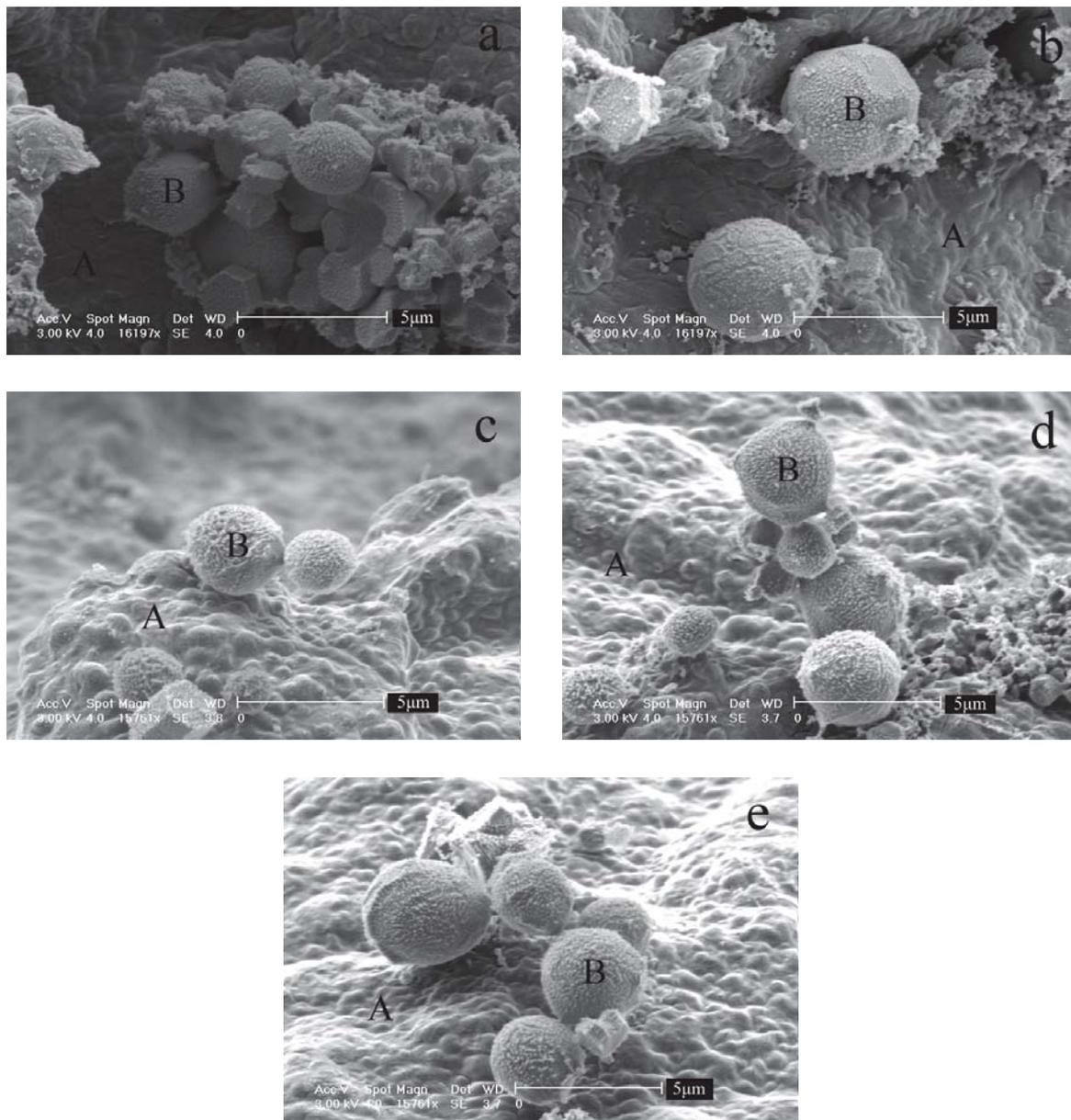


Fig. 9.8 Cryo-SEM images of emulsions produced during scale-up experiments. All the micrographs indicate a continuous fat phase (A), with water droplets, covered by sintered fat crystal shells (B). See Table 9.3 for details of the processing parameters investigated.

The emulsions were visualised using cryo-SEM (see Fig. 9.8) and show fat shells covering the water droplets, which are sintered together into a crystal network, similar to those seen at laboratory scale. Furthermore, these shells are observed not only in the cocoa butter emulsion samples, but also in samples that include the dry mix,

indicating that the mixing of the dry ingredients into the emulsion did not disrupt the crystal shells.

9.3 Chocolate

During the scale up experiments a number of full chocolates were produced. This was carried out by taking the emulsion at the end of the processing line (i.e. from the pin stirrer), and adding the dry ingredients (a mix of sugar, skimmed milk powder, cocoa powder and vanilla; see Table 9.2) in a planetary mixer, until the solid particles were fully mixed into the emulsion, and the mixture looked homogeneous. There was no temperature control at this stage of processing.

There were some limitations when adding the dry ingredients to the emulsions. When making chocolates using the emulsion with 20% water (formulation 1), the entire quantity of dry ingredients could not be added. The dry ingredients were added slowly, until the mix became too viscous, or became crumbly. This occurred at approximately 60% fat, 40% dry mass. When the emulsion contained 40% water (formulation 2) all the dry ingredients could be added if mixed in slowly. Chocolates made during this stage showed some softening, and would not contract enough to demould. The whole percentage of the dry ingredients could be added to the emulsion made with 80% fat emulsion with milk fat (formulation 3); the resulting product was harder than in previous trials, and could be demoulded, but was not shiny or glossy. This was thought to be as a consequence of the lack of temperature control when mixing in the planetary mixer, which prevented crystals in form V from forming.

A limitation of using a planetary mixer to mix in the dry ingredients was the lack of temperature control. Rather than holding the emulsion at the outlet temperature of process, it was allowed to cool in the planetary mixer, causing crystallisation, and not allowing for homogeneous mixing.

The effect that mixing in the planetary mixer had on droplet size could not be accurately measured, as NMR measurements could not be taken as the signal was too weak.

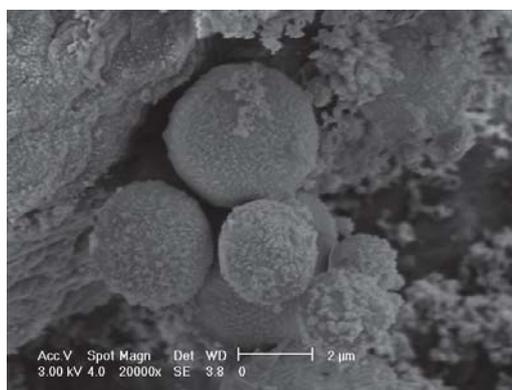


Fig. 9.9 A cryo-SEM image of an emulsion that has been mixed with the dry chocolate ingredients (cocoa solids, sugar, milk solids; see Table 9.2 for the ratio of dry mix to emulsion, and the percentage of ingredients within the dry mix, which was used to produce the chocolate).

9.4 Discussion

The aim of the work presented in this chapter was primarily to scale up the lab emulsification experiments to pilot plant scale. A few changes were made to the formulation (for example, the addition of milk fat), the equipment used was different to the lab equipment (two chilled scraped surface heat exchangers were used, as opposed to the one scraped surface heater exchanger used at lab scale, and the pin stirrer offered no temperature control), and the addition of cocoa solids, milk solids

and sugar was considered. All the emulsions were produced successfully, all being fat continuous with no water loss on storage (i.e. no pooling at the bottom of the sample) or on snap. All the emulsions had small droplets (2 – 6 μ m), which were stable over the course of 12 weeks, although they varied on the amount of free water present, and the amount of water loss on storage. Furthermore, the emulsions produced had fat crystals in polymorphic form V, with melting peaks between 30 and 34°C, and *d*-spacings that suggested that the predominant polymorphic form was form V (although some of the samples had *d*-spacings that are typical for crystals in forms III, IV and VI, suggesting a mixture of crystals in some samples). Furthermore, images of the emulsions showed similar sintered fat crystal shells to those presented in Chapters 7 and 8.

Moulding of the emulsion resulted in a glossy, hard product. However, when the dry ingredients were added, and the resulting chocolate was moulded it was soft, and did not demould. It appears that the method failed to produce a typical chocolate on the introduction of the other ingredients. This is thought to have been as a result of the crude method of adding the dry mass, as the planetary mixer did not result in homogeneous mixing. Furthermore, the lack of temperature control may have resulted in heating of the emulsion during mixing, or conversely too much cooling so the addition of the ingredients became more difficult.

The emulsions produced during scale-up were comparable to those made at lab scale (results presented in Chapters 7 and 8), indicating that similar emulsions can be produced at this scale, with this equipment. However, the production of a full chocolate (the emulsion with the dry mix of sugar, milk solids and cocoa solids) was

not successful. Thus, future work should consider the use of a jacketed mixer that can allow for temperature control during mixing. This should enable the dry ingredients to be mixed in to the emulsion homogeneously, without the mixture becoming too viscous. Furthermore, temperature control should hold the fat at the desirable temperature, so that the 'tempering' achieved during emulsification is not lost (i.e. the emulsion should not be heated above the melting temperature of form V). Care should be taken so that heating or vigorous mixing does not disrupt the emulsion produced (i.e. melting the fat crystal shells at the interface, and causing coalescence). If the emulsion is disrupted it is likely that the water and the dry ingredients will interact, causing aggregation of the particles.

10. Conclusions and Future Work

The consumption of reduced-fat food products offers a way of reducing the fat and energy ingested, with beneficial effects for body weight and health. Currently, reduced-fat chocolates are not readily available on the British market. Thus, research in the area of fat reduction in chocolate is relevant and challenging. However, consumers may not purchase or consume a reduced-fat chocolate, as a reduction in sensory quality is often associated with reduced-fat foods. Furthermore, consumers may not find such a product indulgent or hedonically satisfying. Thus, the aims of the research presented in this thesis were twofold:

- To investigate chocolate consumption, beliefs about health and indulgence and consumer response to reduced-fat chocolate;
- To investigate formulation engineering routes for a reduced-fat chocolate that has a similar flavour, texture and mouthfeel to a full fat equivalent.

This is thought to be the first time that chemical engineering and psychology have been combined in a PhD project. Although in food research the end user is the consumer, often research, especially in academia, neglects psychology when designing food structures. This seems unwise, as the consumer will ultimately determine the success of the product; it is financially risky to engineer a product that may be rejected when is put on the market. Arguably more importantly, regardless of how healthy the product is, if the consumer is not willing to accept it, the product will be a failure, and as such will have no impact on obesity reduction or health

preservation. The results presented in this thesis highlight the need to combine the two disciplines in food research.

Initial focus groups (presented in Chapter 3) indicated that the high calorific content of chocolate often results in regret, guilt, depression or stress following consumption. As a result, ambivalence towards chocolate exists: conflict between the pleasurable sensory appeal of chocolate, and the negative effect on body image and health (due to its high fat, sugar and calorie content). However, when thinking about the concept of healthy indulgence (for example a reduced-fat chocolate) consumers believed this would be a confusing oxymoron i.e. they did not believe that a healthy product could also be indulgent, and thought that the sensory quality would be sacrificed for the health benefits.

Thus, the intention for the work presented in Chapter 4 was to explore expectations towards a reduced-fat chocolate by manipulating label information given during the consumption experience. When presented with two identical standard milk chocolates labelled as 'Milk Chocolate' and 'Reduced-fat Milk Chocolate', ratings of expected liking were significantly different: consumers expected to like the reduced-fat chocolate less than the standard chocolate. This would suggest that prior opinions of the chocolates sensory quality (related to experience of other low-fat foods, and their associated inferior tastes), or its expected hedonic or indulgent properties, might prevent consumers from purchasing or consuming a reduced-fat chocolate. However, following consumption ratings of actual liking did not differ i.e. the chocolate that consumers were led to believe was reduced in fat was liked as much as the standard chocolate. Furthermore, ratings of sensory attributes (melting rate, creaminess,

richness, thickness, milkiness, smoothness and sweetness) were similar in both labelling conditions. This suggests that the belief that the chocolate is reduced in fat does not affect liking or sensory intensity or idealness i.e. personal experience played a greater role in this case than expectations. Thus, if the sensory attributes of a reduced-fat chocolate can be matched to a standard chocolate, and the consumer can be encouraged to consume the chocolate initially, actual liking should not be affected by the knowledge that it is reduced in fat.

A concern for the production of a reduced-fat chocolate has been the knowledge that the product is reduced in fat may result in increased energy intake, both in terms of the product itself, and also during subsequent meals. In this study no significant differences in anticipated normal consumption, or unrestricted consumption were observed. This suggests that energy intake would not differ, and the reduced-fat chocolate would not be eaten to excess. Future work should consider the effect of labelling condition of actual consumption of the chocolates (rather than anticipated, or expected consumption). Furthermore, future work should consider consumption during subsequent meals, to determine whether *ad libitum* consumption amount is a result of expectations about the foods energy density, or of the post-ingestive experience (for example, the energy absorbed by the body following consumption). Once a reduced-fat chocolate has been produced it would be interesting to consider the interaction between labelling condition and energy density or sensory acceptance on liking and consumption.

The aim of the work presented in Chapters 7, 8 and 9 was to identify whether the calorie and fat content of chocolate could be reduced by introducing water in the form

of a fat continuous water in oil (W/O) cocoa butter emulsion. Results showed that fully emulsified W/O emulsions could be produced using either a high shear mixer, or a margarine line, using either lecithin or PGPR (see Chapter 7). Formulation changes were also considered, including PGPR and gelatin concentration, as were processing parameters, including shaft speeds, shaft temperatures, and flow rate (see Chapter 8). All the emulsions had small droplets (typically with an average droplet size of 1-5 μ m), with very little 'free water', although differences were observed with formulation and processing changes (see Chapter 8).

The margarine line allowed for the temperature to be controlled during the process, so that emulsions with fat crystals in form V could be produced i.e. it was possible to mimic the tempering process during emulsification. It was shown that the A and C unit inlet and outlet temperatures had an impact on the crystal form produced during emulsification (see Chapter 8). Smooth crystalline shells were observed at the droplet interface. It is thought that the temperature profile allowed fat crystals to act as Pickering particles and sinter at the interface, which should result in greater physical and microbial stability.

The aim of the work presented in Chapter 9 was primarily to scale up the lab emulsification experiments to pilot plant scale. The emulsions produced during scale-up were comparable to those made at lab scale (results presented in Chapters 7 and 8), being fat continuous, with small droplets (2-6 μ m), and with crystals in polymorphic form V. Furthermore, in order to produce a full chocolate using the emulsion, the addition of cocoa solids, milk solids and sugar was considered. The method failed to produce a typical chocolate on the introduction of the other ingredients. This is

thought to have been as a result of the crude method of adding the dry mass, as the planetary mixer did not result in homogeneous mixing. Furthermore, the lack of temperature control may have resulted in heating of the emulsion during mixing (resulting in the break up of the structure), or conversely too much cooling so the addition of the ingredients became more difficult. However, the use of the emulsion in a full chocolate showed promise, and by using temperature controlled mixers directly after emulsification, mixing should result in a homogeneous product, without disruption of the emulsion. Future work should consider this in more detail. This could include using a jacketed scraped-surface vessel with temperature control, and relatively low shear in order to prevent disruption of the crystal network.

The aim of the work presented in Chapter 5 was to gain a better understanding of current chocolate packaging, and to explore approaches to packaging a reduced-fat chocolate bar. Focus groups encouraged idea generation surrounding the packaging of a reduced-fat chocolate bar, resulting in four important components being recognised (colour, size / shape, slogan and logo), each with four manipulations (caramel or blue, long or square, pleasure or taste oriented, and heart or tick, respectively).

Overall, consumers exhibit relatively consistent responses to the type of packaging required for a reduced-fat chocolate to be liked. Both men and women believed that a reduced-fat bar would be aimed towards women, who are more weight and health conscious. It was shown that the most liked package was a caramel, square bar with a heart logo, with either of the slogans (there was no apparent preference, although when explaining their preferences, some thought that the taste-oriented slogan was preferable), as displayed in Figure 10.1.



Fig. 10.1 The ideal packaging combination, with the highest score for expected liking for both male and female consumers (results are presented in Chapter 5).

When asked to explain why this package was their favourite, participants thought this bar looked expensive, luxurious and welcoming, indicating that the bar would taste similar to regular chocolate, and that they would love the taste. Overall, the data suggests that specific marketing approaches will aid the successful introduction of a reduced-fat chocolate into the market. Future work could consider presenting the packages with a chocolate sample in a similar way to the labelling study presented in Chapter 4, to consider the effect that the packaging has on liking of the chocolate.

Thus, this study demonstrates that the production of cocoa butter emulsions can be achieved using technology typically used for margarine manufacture, and shows the potential for the use of cocoa butter emulsions in chocolate formulations for fat reduction. Furthermore, although some consumers were critical towards the concept of ‘healthy indulgence’, and ratings of expected liking were lower for a chocolate that consumers believed was reduced in fat, it was shown that the experience of the chocolates taste had a greater impact on liking than expectations did. This offers an excellent opportunity to produce a successful reduced-fat chocolate. Although it is important that future work considers the taste and sensory attributes of reduced-fat chocolates, in order to inform the formulation and processing of the product, this

approach highlights the impact that psychology alone has on acceptance, and indicated the need to integrate the two disciplines, in order to successfully launch reduced-fat products.

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Appendix 1: Letter of Ethical Approval

UNIVERSITY OF
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School of Psychology



Appendix 2: Example Consent Form

Consent Form

This is to certify that I,....., hereby agree to participate as a volunteer in a scientific experiment as an authorised part of the research undertakings within the School of Chemical Engineering at the University of Birmingham conducted by Jennifer Norton, under the supervision of Professor Peter Fryer.

My part in the investigation has been fully explained to me by Jennifer Norton and I understand her explanation. The procedures of this investigation and their risks have been answered to my satisfaction.

I understand that I will be video and audio recorded. I understand that all data will remain confidential with regard to my identity.

I understand that I am free to withdraw my consent and terminate my participation at any time and without penalty. I understand that I can withdraw my data even after the experiment is complete, and up to the point of publication.

My responsibility as a participant is to participate actively and willingly and if I choose not to do so, I will exercise my right to withdraw without penalty. If I choose not to withdraw, I understand that I am expected to participate actively.

I understand that I may request a summary of the results of the study.

Any complaints concerning the conduct of the research should be addressed to Professor Peter Fryer, School of Chemical Engineering, University of Birmingham.

.....

Participant's Signature

Date

I, the undersigned, have fully explained the investigation to the above individual.

.....

Investigator's Signature

Date

Appendix 3: Example Debriefing Form

Debriefing

Thank you for taking part in the discussion group. The purpose of the discussion was to investigate chocolate consumption, and to gain information about what you look for in chocolate, when and how much you eat of it, and why you eat it.

You were given the Dutch Eating Behaviour Questionnaire (DEBQ) which aims to examine your eating behaviour, in terms of emotional eating (eating in response to emotional arousal states such as fear, anger or anxiety), external eating (eating in response to external food cues such as the sight or smell of food) and restraint (overeating after a period of slimming). This information will be used in conjunction with the opinions given during the discussion session. The demographic questions (age, height and weight) will be used to compile average data for the group.

As already mentioned, your data will remain confidential with regards to you identity. If you wish to do so you can withdraw your data even after the experiment is complete, and up to the point of publication.

You may request a summary of the results of the study by contacting Jennifer Norton.

Any complaints concerning the conduct of the research should be addressed to Professor Peter Fryer, School of Chemical Engineering, University of Birmingham.

Contact Details

Jennifer Norton



Appendix 4: Focus Group Questionnaire

SECTION 1:

The following information will be used to compile average data for the group. Your responses will be kept anonymous, and will not be used on an individual basis. For this reason **please be honest**.

Please complete the following section by **ticking** the relevant response.

Age:

16-20	<input type="checkbox"/>
21-30	<input type="checkbox"/>
31-40	<input type="checkbox"/>
41-50	<input type="checkbox"/>
51-60	<input type="checkbox"/>
61+	<input type="checkbox"/>

Gender:

Male	<input type="checkbox"/>
Female	<input type="checkbox"/>

Weight:

Under 7 Stone	<input type="checkbox"/>
7 – 8 Stone	<input type="checkbox"/>
8 - 9 Stone	<input type="checkbox"/>
9 - 10 Stone	<input type="checkbox"/>
10 – 11 Stone	<input type="checkbox"/>
11 – 12 Stone	<input type="checkbox"/>
12 – 13 Stone	<input type="checkbox"/>
13 - 14 Stone	<input type="checkbox"/>
14 – 15 Stone	<input type="checkbox"/>
16 – 17 Stone	<input type="checkbox"/>
17 – 18 Stone	<input type="checkbox"/>
18 – 19 Stone	<input type="checkbox"/>
Above 20 Stone	<input type="checkbox"/>

Height:

Under 5'	<input type="checkbox"/>
5' – 5'1	<input type="checkbox"/>
5'2 – 5'3	<input type="checkbox"/>
5'4 – 5'5	<input type="checkbox"/>
5'6 – 5'7	<input type="checkbox"/>
5'8 – 5'9	<input type="checkbox"/>
5'10 – 5'11	<input type="checkbox"/>
6' – 6'1	<input type="checkbox"/>
6'2 – 6'3	<input type="checkbox"/>
6'4 – 6'5	<input type="checkbox"/>
Above 6'6	<input type="checkbox"/>

Occupation / area of study:
Nationality:

SECTION 2:

Please complete the following section by **ticking** the relevant response.

	never	seldom	sometimes	often	very often	not relevant
1. How often do you refuse food or drink offered because you are concerned with your weight?						
2. Do you watch exactly what you eat?						
3. Do you take into account your weight when choosing what you eat?						
4. Do you deliberately eat foods that are slimming?						
5. Do you deliberately eat less in order not to become heavier?						
6. How often do you try not to eat between meals because you are watching your weight?						
7. When you have eaten too much, do you eat less than usual in the following days?						
8. In the evening how often do you try not to eat because you are watching your weight?						
9. Do you try and eat less at mealtimes than you would like to eat?						
10. If you have put on weight, do you eat less than you usually do?						
11. When preparing a meal are you inclined to eat something?						
12. If food tastes good to you, do you eat more than usual?						
13. If food smells and looks good, do you eat more than usual?						
14. Can you resist eating delicious foods?						
15. If you see or smell something delicious to eat, do you eat it straight away?						
16. Do you eat more than usual when you see others eating?						
17. If you see others eating, do you also have the desire to eat?						
18. If you walk past the bakery do you have the desire to buy something delicious?						
19. If you have something delicious to eat, do						

you eat it straight away?						
20. If you walk past a snackbar or a café, do you have the desire to buy something delicious?						
Do you have the desire to eat when you are:						
21. ...irritated?						
22. ...cross?						
23. ...frightened?						
24. ...emotionally upset?						
25. ...bored or restless?						
26. ...approaching something unpleasant?						
27. ...depressed or discouraged?						
28. ...feeling lonely?						
29. ...nothing to do?						
30. ...anxious, worried or tense?						
31. ... disappointed?						
32. Do you have a desire to eat when things are going against you or when things have gone wrong?						
33. Do you have a desire to eat when somebody lets you down?						

End of questions
Thank you very much

Appendix 5: Screening Questionnaire

The following questionnaire is used to gain information about yourself that will determine your suitability for the study. All the information you give will be kept confidential, and will be used for the purpose of this experiment only. Please answer by typing in answers and emailing it back to jxn734@bham.ac.uk.

Your full name:

Your email address:

Please complete the following section by **placing an 'X'** in the relevant box.

How old are you?

16-20

21-30

31-40

41-50

51-60

61+

Are you male or female?

Male

Female

Do you follow a specific diet that prevents you from eating certain foods?

Low sugar

Low fat

Vegetarian

Vegan

Gluten Free

Other

No

If 'other', please specify:

Do you have a specific food allergy?

Yes

No

If 'yes', please specify:

How often do you eat / drink the following foods?

	More than once a day	Once a day	3 times a week	Once a week	Once a month	Once every few months	Never
Carbonated drinks							
Tea							
Fruit							
Crisps							
Chocolate							
Yogurt							
Coffee							

If given the opportunity, how likely is it that you would try the following foods?

	Very likely	Quite likely	Neither likely nor unlikely	Not likely	Very unlikely
A new flavour of crisps					
A lower fat version of a food you have tasted before					
A luxury version of a food you have tasted before					
A vegetable you have never heard of before					
A dish from another country					
A new health food					

Thank you for completing the questionnaire,

Jennifer Norton

Appendix 6: Script

My name is Jennifer Norton and I am a third year PhD student. I am running this consumer study as part of my research.

All I require of you today will be to taste a milk chocolate sample and answer a number of questions about it.

There are a number of important things that I need to tell you first:

It is really important that you don't have a nut allergy, as the chocolates may contain traces of nuts. By agreeing to take part you confirm that you do not have a nut allergy, or any other allergy that may prevent you from eating chocolate.

You are free to withdraw from the experiment at any point.

If you decide that you want to take part it is important that you participate actively and answer all of the questions to the best of your ability.

You will need to sign the consent form in order to take part. Your name will be on this form, but everything else you fill in today will require your personal participant code. I will be the only person that will be able to connect your name to your code, so your data will be kept completely anonymous.

You have been randomly assigned to one of a few experimental groups, and over the course of the two sessions you will taste 2 of the chocolate products.

If you would like to take part then I need you to complete the consent form.

Whilst filling in the consent form could you eat a cracker and drink some water. This is to cleanse your palate and ensure that there is no contamination from anything you have eaten previously.

Appendix 7: Instructions for Participants

This session will last approximately 20 minutes. There are four parts to the session: Section's A, B, C and D. Sections A, B and D involve filling out a questionnaire and verbally answering two questions.

You will be given a sample of chocolates and the accompanying questionnaire. **Before tasting the chocolate** answer Section A of the questionnaire. After completing Section A, taste the chocolate and answer Section B.

When you have finished Sections A and B you will be instructed on Section C. After completing Section C, please fill in Section D. After completing all Sections of this questionnaire you will be given another short questionnaire.

If this is your first session please ask the experimenter to remind you of the date and time of your second session before leaving. You will receive payment following the completion of both sessions 1 and 2.

If you are willing to take part in this experiment please read and sign the consent form. When you are ready you can start.

REMINDER: YOU MAY NOT TAKE PART IN THIS EXPERIMENT IF YOU HAVE A NUT ALLERGY, OR ANY OTHER ALLERGY THAT PREVENTS YOU FROM EATING CHOCOLATE.

Line-scale Questions

When given a line-scale question please make a vertical mark across the horizontal line at the point that you consider to represent the rating for the product.

For example, if played a sound you would answer a line-scale question in the following way:

1. How loud do you think the following sound is?

Extremely quiet

Extremely loud



Multiple Choice Questions

When given a multiple choice question please place an 'X' in the box that you consider to represent the rating for the product. Remember: **Please tick ONE box only.**

For example, if played a sound you would answer a multiple choice question in the following way:

2. What is your opinion of the volume of the sound?

Far too loud

Slightly too loud

Just about right

Slightly too quiet

Far too quiet

Appendix 8: Labelling Study Questionnaire

Participant Code:

Date:

Sample:

A. Please take a look at the chocolate (DO NOT TASTE IT)

1. How much do you think you'll like this chocolate?

Like not at all

Like extremely



B. Now taste the chocolate: eat ONE SQUARE ONLY.

1. How much do you like the chocolate overall?

Like not at all

Like extremely



Eat ONE MORE SQUARE of chocolate

2. How fast do you think this chocolate melts in the mouth?

Melts very slowly

Melts very quickly

3. What is your opinion of the melting rate of the chocolate?

Melts far too quickly

Melts slightly too quickly

Just about right

Melts slightly too slowly

Melts far too slowly

4. How creamy do you think the chocolate is?

Not at all creamy

Extremely creamy

5. What is your opinion of the creaminess of the chocolate?

Much too creamy

Slightly too creamy

Just about right

Not quite creamy enough

Not at all creamy enough

6. How rich do you think the chocolate is?

Not at all rich

Extremely rich



7. What is your opinion of the richness of the chocolate?

Much too rich

Slightly too rich

Just about right

Not quite rich enough

Not at all rich enough

8. When thinking about mouthfeel (i.e. how the sample feels in the mouth), how thick do you think the chocolate is?

Not at all thick

Extremely thick



9. When thinking about mouthfeel (i.e. how the sample feels in the mouth), what is your opinion of the thickness of the chocolate?

Much too thick

Slightly too thick

Just about right

Not quite thick enough

Not at all thick enough

Now EAT THE LAST SQUARE of chocolate

10. How milky do you think the chocolate is?

Not at all milky

Extremely milky



11. What is your opinion of the milkiness of the chocolate?

Much too milky

Slightly too milky

Just about right

Not quite milky enough

Not at all milky enough

12. How smooth do you think the chocolate is?

Not at all smooth

Extremely smooth

13. What is your opinion of the smoothness of the chocolate?

Much too smooth

Slightly too smooth

Just about right

Not quite smooth enough

Not at all smooth enough

14. How sweet do you think the chocolate is?

Not at all sweet

Extremely sweet

15. What is your opinion of the sweetness of the chocolate?

Much too sweet

Slightly too sweet

Just about right

Not quite sweet enough

Not at all sweet enough

D. Price

a. Thinking about the chocolate that you have just tasted, if it were available in supermarkets would you buy it?

b. For a 50g bar of chocolate you would typically spend:

Budget milk chocolate: 14p

Supermarket own brand milk chocolate: 28p

Branded milk chocolate: 49p

Luxury milk chocolate: 89p

How much would you pay for a 50g bar (8 squares) of the chocolate you have tasted today?

Appendix 9: Post Study Questionnaire A

1. Participant Code:

2. Current Time:

3. Prior to taking part in this study, at what time did you last consume a meal?

4. Please give a detailed description of what you ate:

5. Did you have any snack between your last meal and the start of the session?

6. If so, please give a detailed description of what you ate:

7. If you are a smoker, at what time did you last smoke?

Appendix 10: Post Study Questionnaire B

SECTION 1

1. Participant Code:
2. Current Time:
3. Prior to taking part in this study, at what time did you last consume a meal?
4. Please give a detailed description of what you ate:
5. Did you have any snack between your last meal and the start of the session?
6. If so, please give a detailed description of what you ate:
7. If you are a smoker, at what time did you last smoke?

SECTION 2

What is your....

1. current age?

2. nationality?

3. native language?

4. current marital status?

5. current occupation / area of study?

6. What is the highest level of education you have completed?

SECTION 3

For the following section please **circle** the relevant response. When answering the questions please think about **all chocolate**, not specifically the chocolate you have tasted today, or during your first session.

1. My attitude towards eating chocolate is...

extremely positive 1 2 3 4 5 6 7 extremely negative

extremely favourable 1 2 3 4 5 6 7 extremely unfavourable

2. For me eating chocolate is...

not at all pleasant 1 2 3 4 5 6 7 extremely pleasant

not at all unpleasant 1 2 3 4 5 6 7 extremely unpleasant

not at all wise 1 2 3 4 5 6 7 extremely wise

not at all foolish 1 2 3 4 5 6 7 extremely foolish

not at all enjoyable 1 2 3 4 5 6 7 extremely enjoyable

not at all unenjoyable 1 2 3 4 5 6 7 extremely unenjoyable

not at all good 1 2 3 4 5 6 7 extremely good

not at all bad 1 2 3 4 5 6 7 extremely bad

not at all beneficial 1 2 3 4 5 6 7 extremely beneficial

not at all harmful 1 2 3 4 5 6 7 extremely harmful

3. I feel under social pressure to eat chocolate...

disagree strongly 1 2 3 4 5 6 7 agree strongly

4. Most people who are important to me think that I should eat chocolate...

extremely likely 1 2 3 4 5 6 7 extremely unlikely

5a. Are there other people who are likely to influence you eating chocolate...

Yes								No
5b. If you answered 'yes' to question 5a., the views of those people towards you eating chocolate are...								
extremely favourable	1	2	3	4	5	6	7	extremely unfavourable
6. For me, eating chocolate is...								
extremely easy	1	2	3	4	5	6	7	extremely difficult
7. If I want to I can easily eat chocolate...								
extremely likely	1	2	3	4	5	6	7	extremely unlikely
8. How likely is it that, if you try, you are able to eat chocolate...								
extremely likely	1	2	3	4	5	6	7	extremely unlikely
9. I think of myself as a health conscious consumer...								
disagree strongly	1	2	3	4	5	6	7	agree strongly
10. I think of myself as someone who is concerned about the consequences of what I eat...								
disagree strongly	1	2	3	4	5	6	7	agree strongly
11. I intend to eat chocolate tomorrow...								
definitely do not	1	2	3	4	5	6	7	definitely do
12. I will make an effort to eat chocolate tomorrow...								
definitely false	1	2	3	4	5	6	7	definitely true
13. I will try to try to eat chocolate tomorrow...								
definitely will not	1	2	3	4	5	6	7	definitely will
14. For a moment consider only the positive things about eating chocolate and ignore any negative things about it. Please rate how positive those positive things are...								
not at all positive	1	2	3	4	5			extremely positive

15. For a moment consider only the negative things about eating chocolate and ignore any positive things about it. Please rate how negative those negative things are...

not at all negative 1 2 3 4 5 extremely negative

16. To what extent do you have positive thoughts or feelings about eating chocolate?

not at all 1 2 3 4 5 6 7 to an extremely great extent

17. To what extent do you have negative thoughts or feelings about eating chocolate?

not at all 1 2 3 4 5 6 7 to an extremely great extent

18. To what extent are there advantages for you in eating chocolate?

not at all 1 2 3 4 5 6 7 to an extremely great extent

19. To what extent are there disadvantages for you in eating chocolate?

not at all 1 2 3 4 5 6 7 to an extremely great extent

20. Would you say that you are strongly in favour (or strongly against) eating chocolate or would you say that your feelings are mixed?

strongly in favour mixed feelings strongly against

SECTION 4

Please complete the following section by **placing an 'X'** in the relevant box.

	never	seldom	sometimes	often	very often	not relevant
1. How often do you refuse food or drink offered because you are concerned with your weight?						
2. Do you watch exactly what you eat?						
3. Do you take into account your weight when choosing what you eat?						

4. Do you deliberately eat foods that are slimming?						
5. Do you deliberately eat less in order not to become heavier?						
6. How often do you try not to eat between meals because you are watching your weight?						
7. When you have eaten too much, do you eat less than usual in the following days?						
8. In the evening how often do you try not to eat because you are watching your weight?						
9. Do you try and eat less at mealtimes than you would like to eat?						
10. If you have put on weight, do you eat less than you usually do?						
11. When preparing a meal are you inclined to eat something?						
12. If food tastes good to you, do you eat more than usual?						
13. If food smells and looks good, do you eat more than usual?						
14. Can you resist eating delicious foods?						
15. If you see or smell something delicious to eat, do you eat it straight away?						
16. Do you eat more than usual when you see others eating?						
	never	seldom	sometimes	often	very often	not relevant
17. If you see others eating, do you also have the desire to eat?						
18. If you walk past the bakery do you have the desire to buy something delicious?						
19. If you have something delicious to eat, do you eat it straight away?						
20. If you walk past a snackbar or a café, do you have the desire to buy something delicious?						
Do you have the desire to eat when you are:						
21. ...irritated?						
22. ...cross?						
23. ...frightened?						
24. ...emotionally upset?						

25. ...bored or restless?						
26. ...approaching something unpleasant?						
27. ...depressed or discouraged?						
28. ...feeling lonely?						
29. ...nothing to do?						
30. ...anxious, worried or tense?						
31. ... disappointed?						
32. Do you have a desire to eat when things are going against you or when things have gone wrong?						
33. Do you have a desire to eat when somebody lets you down?						

SECTION 5

1. To ensure the validity of this research, please briefly describe what the main aim of the study was?

2. Can you please indicate which brands of chocolate you think you tasted during sessions 1 and 2:

Session 1:

Session 2:

End of questions
Thank you very much

Appendix 11: Packaging Study Screening Questionnaire**Screening Questionnaire – Electronic Version**

The following questionnaire is used to gain information about yourself that will determine your suitability for the study. All the information you give will be kept confidential, and will be used for the purpose of this experiment only. Please answer by typing in answers and emailing it back to jxn734@bham.ac.uk.

Your full name:

Your email address:

Please complete the following section by **placing an 'X'** in the relevant box.

How old are you?	16-20	—
		—
	21-30	—
		—
	31-40	—
		—
	41-50	—
		—
		—
	51-60	—
		—
	61+	—
		—

Do you have a specific food allergy?

Yes

—

—

No

—

—

If 'yes', please specify:

How often do you eat / drink the following foods?

	More than once a day	Once a day	3 times a week	Once a week	Once a month	Once every few months	Never
Carbonated drinks							
Tea							
Fruit							
Crisps							
Chocolate							
Yogurt							
Coffee							

You will be required to attend the discussion session on Wednesday 24th March, during the afternoon. If you are suitable for this experiment you will be contacted via email within a week of returning this questionnaire.

If you cannot be contacted via email please provide a phone number from which you can be contacted:

Thank you for completing the questionnaire,

Jennifer Norton

Jxn734@bham.ac.uk

Appendix 12: Discussion Guide

DISCUSSION GUIDE (90 MINUTES)

INTRODUCTION (UP TO 5 MINS)

Could you find the sticker with your name on it. Could you also read and sign a consent form.

Ask participants if they want drinks. Tell them where the toilets are, and to leave if they need to use them.

There are no right or wrong answers, it is your view and opinions that are important, so I am not going to judge you on the answers you give. The aim of the session is to generate discussion by involving everyone in the group.

You should be aware that your responses will be audio / video recorded, but all data gathered will be anonymous and confidential (your name will not be with the data).

Your involvement is optional, and you can withdraw at any time.

INTRODUCTION OF PARTICIPANTS (UP TO 5 MINS)

The first thing I would like you to do before we get started on the discussion is to spend a few minutes doing the...

1. Thought bubble completion task: you each have two photographs of people choosing bars of chocolate. As you can see there are empty thought bubbles on photos. What I would like you to do is write in the bubbles what you think the person might be thinking or feeling. It's important that you write down anything and everything that comes to mind. **10 minutes**

I am completing this research as part of my PhD. The purpose of the focus group is gather your thoughts and opinions on chocolate packaging.

Please introduce yourself to the group, giving your first name and your favourite chocolate bar.

MAIN BODY

2. In front of you are a group of photographs of chocolate bars. Some of them you will have seen before, and some of them you may not have done. What I would like

you to do is to group the photographs according to healthiness. I want you to focus on the packaging, and any information given on the packaging, and less so on anything you know about the bars already. So if on this side you put bars that you think are healthier, and the other side bars that you think are less healthy. **25 minutes**

Prompts:

- so why have you put these bars in this pile?
- can anyone see anything else that is different?
 - o does that impact on healthiness?

3. I am currently working on a reduced-fat chocolate, and am interested in how it might be marketed. So I would like you to think about a reduced-fat chocolate, and work as a group to complete some of the sentences which I have written on the board.

30 minutes

- ‘The best thing about this product is...’
- ‘The most annoying thing about this product is...’
- ‘The kind of person who would buy this product would be...’
- ‘I’d convince people to buy this product by saying...’
- ‘The wrapper of a reduced-fat chocolate bar should...’

4. I have photographs of the same people buying chocolate bars, but I want you to imagine them buying a reduced-fat chocolate bar packaged in the way we have discussed today. Again I would like to fill in the thought bubbles, writing down anything you think of. **10 minutes**

CLOSURE (5 MINS)

Before closing the session, does anyone have anything else they would like to say or contribute to the discussion?

Thank you for your involvement and contributions today.

Give vouchers and chocolate bars.

Appendix 13: Thought Bubble Completion Task 1



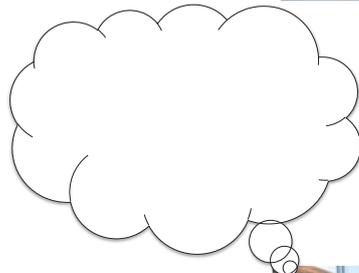
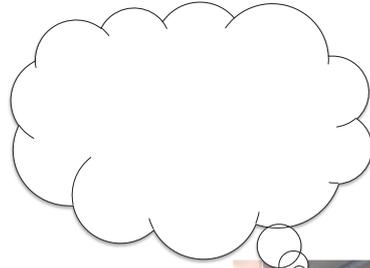
Appendix 14: Photographs of chocolates







Appendix 16: Thought Bubble Completion Task 2



Appendix 16: Rank-rating Questionnaire

1. In front of you are 16 photographs of chocolate bars.

Please rank the photographs, from least to most, according to how much you think you would like the chocolate bars. The bar that you expect to like the least should go in position 1, and the bar you expect to like the most should go in position 16, and all other bars should be in order in-between.

When you are happy with the order you have chosen, please write the number of each of the photographs (found in the top right hand corner of each of the photos) into the boxes below:

1	<input type="text"/>	2	<input type="text"/>	3	<input type="text"/>	4	<input type="text"/>	5	<input type="text"/>	6	<input type="text"/>	7	<input type="text"/>	8	<input type="text"/>
9	<input type="text"/>	10	<input type="text"/>	11	<input type="text"/>	12	<input type="text"/>	13	<input type="text"/>	14	<input type="text"/>	15	<input type="text"/>	16	<input type="text"/>

When you have completed this, please turn over.

2. Please imagine that the photo you have put in position 1 has a score of 1 out of 100, and the photo you have put in position 16 has a score of 100 out of 100.

Please give all the other photos a score out of 100 according to how much you think you would like the chocolate bar. Write your answers in the boxes below:

1	<input type="text"/>	2	<input type="text"/>	3	<input type="text"/>	4	<input type="text"/>	5	<input type="text"/>	6	<input type="text"/>	7	<input type="text"/>	8	<input type="text"/>
9	<input type="text"/>	10	<input type="text"/>	11	<input type="text"/>	12	<input type="text"/>	13	<input type="text"/>	14	<input type="text"/>	15	<input type="text"/>	16	<input type="text"/>

When you have completed this, please turn over.

3. Consider the bar that you put in position 1. Please briefly explain why you expect to like this bar the least:

4. Consider the bar that you put in position 16. Please briefly explain why you expect to like this bar the most:

5. What is your age?

6. What is your gender?

Appendix 17: Summary of the explanations for ratings of expected liking

	Milk Chocolate		Reduced-fat Milk Chocolate	
	Positive I like chocolate	Negative I prefer dark chocolate	Positive I like chocolate	Negative I prefer dark chocolate
Expected Liking	Looks normal / typical / familiar	Unattractive coding / no branding	Looks normal / pleasant / tasty /	No branding
	Good shape (size and thickness of squares)	Squares are too big	Nice shape	Simple formation of squares
	Full blocks (not broken)	Not fresh	Looks fresh	
		Too simple / bland / plain (no filling e.g. caramel or nuts)		Looks plain / bland
	Good / rich colour	Too light in colour	Nice colour	
	Doesn't look too milky	Looks too milky		Looks too milky
	Doesn't look too dark	Looks too dark		
	Quite shiny	Not shiny enough		
	No white dust on surface	White dust on surface		White dust on surface
		Looks dull / dry		Looks dull / dry (no glaze on surface)
			Looks like it has a nice texture	
	Looks soft / smooth		Looks soft / smooth	
	Crumbly texture	Looks chalky		Looks chalky
	Looks creamy		Looks creamy	Doesn't look creamy
	Doesn't look greasy	Looks plasticity	Doesn't look plasticity	
				Might be less rich
				Might be less sweet
				Not as flavoursome / won't have a full taste / strange taste / will it taste like the real thing / less tasty
		Less fattening	A diet food	

Appendix 18: Summary of the explanations for ratings of actual liking

	Milk Chocolate		Reduced-fat Milk Chocolate	
	Positive	Negative	Positive	Negative
Actual Liking	Preference for dark		Preference for dark chocolate	
	Tastes normal / good quality	Nothing special / not good quality	Tastes nice / pleasant / normal / good quality	Doesn't taste expensive / not great quality
	Good flavour / Chocolatey	Not very chocolatey flavour / bland / tastes synthetic	Nice flavour / full flavoured / distinctive chocolate flavour / doesn't taste artificial	Not very chocolatey flavour / tastes light (reduced-fat)
	Not milky	Too milky / not milky enough	Milky	Not milky enough / too milky
	Not too bitter	Not dark enough (cocoa content) / too bitter		Too bitter / too dark
	Stale tasting			
	Chunks too thick		Too solid	
	Good texture	Too thick		
	Rich / not too rich	Not very rich	Rich texture / satisfying / isn't sickly	Not very rich
	Soft but crunchy	Not soft	Soft / not too hard	Chewy
	Quite sweet / not too sweet	Too sweet / not sweet enough	Sweetness ok	Not sweet / too sweet
	Melts well	Doesn't melt well	Melts in mouth nicely	Melts too quickly / doesn't melt easily
	Smooth		Smooth texture	Not smooth enough
	Not dry	Chalky texture / sticks to teeth		Waxy
			Not bitty	Dry / grainy/ stuck around teeth
	Really creamy / not too creamy	Not creamy enough / too creamy	Creamy / not too creamy	Not creamy
	Nutty	Nutty		
	Crumbly / not too crumbly		Crumbly	
	Not too heavy	Isn't moreish		
	Nice aftertaste	Strange aftertaste / aftertaste too sweet	Strange aftertaste	

Appendix 19: Packaging concepts



BSPH



BLPH



BSTH



BLTH



BSPT



BLPT



BSTT



BLTT



CSPH



CLPH



CSTH



CLTH



CSPT



CLPT



CSTT



CLTT

Appendix 20: NMR and DSC data for emulsions produced with A & C units (varying rotation speeds and unit temperatures)

Table 1 NMR and DSC results for emulsions when the rotation speeds of the two units were varied.

A Unit (rpm)	C Unit (rpm)	Inlet Temp (°C)	A Unit Outlet Temp (°C)	C Unit Outlet Temp (°C)	DSC		NMR		
					T _{peak} (°C)	Melting Enthalpy (J/g ⁻¹)	d _{3,2} (μm)	Sigma	Free Water (%)
170	170	41.1	25.5	29.2	27.8 (0.2)	120 (3.3)	13.3	0.7	11
170	480	40.1	25.6	29.2	27.4 (0.3)	93 (6.8)	11.4	0.7	14
170	900	39.8	25.4	29.3	27.6 (0.7)	108.4 (3)	8.3	0.7	10.4
170	1300	42.5	25.8	29.6	27.4 (0.7)	101.6 (4.2)	5.7	0.8	11.1
470	170	40.5	23.5	29.3	28 (0.3)	106.1 (0.8)	9.9	0.8	8
470	480	41.1	24.1	29.3	28.8 (0.8)	120.9 (1.5)	12.6	0.9	2
470	900	42.8	24.5	29.4	27.9 (1.3)	98.2 (2.1)	8.9	0.8	3
470	1300	45.3	24.1	29.7	28.7 (0.5)	123.9 (3.4)	4.8	0.8	0
920	170	46.3	23.2	29.2	27.6 (0.5)	93.9 (0.1)	6.5	0.9	0
920	480	44.4	22.7	29.6	26.3 (0.2)	88.9 (1.2)	5.1	0.8	0
920	900	45	23.8	29.5	28.3 (0.4)	116.1 (6.3)	4.3	0.8	0
920	1300	41.6	23.3	26.6	26.6 (1.8)	127.6 (6.5)	4.3	0.7	0
1350	170	44.9	23.2	29.1	26.4 (0.2)	89.9 (0.6)	3.6	0.8	0
1350	480	45.1	25.5	29.2	25.9 (1.2)	92.7 (11.5)	3.1	0.7	3.2
1350	900	45.9	24.1	29.4	27.8 (1.1)	103.3 (7.7)	2.9	0.6	2.4
1350	1300	46.8	24.2	29.6	26.6 (0.5)	100.6 (2.5)	3.4	0.5	0

Table 2 NMR and DSC results for emulsions when processed at different temperatures.

A Unit Temp (°C)	C Unit Temp (°C)				DSC		NMR		
		Inlet Temp (°C)	A Unit Outlet Temp (°C)	C Unit Outlet Temp (°C)	Tpeak (°C)	Melting Enthalpy (J/g ⁻¹)	d _{3.2} (µm)	Sigma	Free Water (%)
15	25	37.1	21.5	26.2	31.7 (0.9)	134.2 (3.4)	3.7	0.7	2
15	30	42.6	22.6	29.8	30.6 (0.7)	116.2 (4.2)	6.0	0.8	0.9
20	20	40.1	25.9	20.6	28.3 (0.1)	90.2 (3.3)	5.9	0.7	0
20	30	40	25.8	29.3	31.5 (0.4)	116.2 (3.2)	7.6	1	11
25	25	-	27.7	25.5	30.7 (0.04)	140.8 (1.9)	5.5	0.9	1
25	30	41.9	27.5	29.3	28.9 (0.2)	100.9 (9.6)	6.6	0.7	0
25	35	40.3	27.2	33	26.7 (1.3)	108.3 (0.3)	6.7	0.7	0
25	45	-	27.9	41.4	29.6 (0.4)	110.3 (0.6)	6.0	1.1	0
30	25	41.8	31	25	27.9 (0.1)	138.1 (5.5)	5.2	0.7	1.2
30	30	-	29.9	29.8	30.5 (0.2)	128.3 (2)	6.0	0.9	0
30	35	41.7	31.3	33.2	27.7 (0.9)	108.8 (5.7)	6.0	0.8	0.4
30	45	-	30.1	41.4	29.1 (0.3)	110.9 (1.5)	6.4	1.1	0
35	25	42.2	35.4	26	26.1 (0.4)	102.8 (5)	6.1	0.7	0
35	30	41.4	35.3	29.4	27.6 (0.9)	106.1 (1.5)	5.8	0.7	1.2
35	35	-	33	32.9	30.5 (0.5)	120.6 (2.3)	7.3	1.0	0
35	45	-	33.5	40.9	29.7 (0.5)	110.8 (0.7)	5.8	1.4	0
45	25	-	38.7	26.7	30.1 (0.2)	120.7 (3.6)	7.7	0.9	0
45	30	42.8	44.2	29.6	27.5 (0.5)	109.5 (11.1)	5.4	0.9	0
45	35	40.9	43.6	33.9	27.2 (1.1)	112.7 (0.7)	6.7	0.8	0.2
45	45	-	38.2	42.3	29.3 (0.8)	105.4 (2.2)	6	1.3	0
50	50	41.9	48	44.7	26.5 (0.2)	108.2 (1.8)	7.3	0.8	0
60	60	43.6	57.4	56.5	25.4 (0.7)	96 (4)	7.6	0.8	0

Appendix 21: NMR droplet sizing and DSC data of emulsions produced during scale-up

Table 1 NMR data of emulsions, conducted at 1 week

Sample	Ambient			Chilled			
	$d_{3.2}$ (μm)	Sigma (σ)	Free Water (%)	$d_{3.2}$ (μm)	Sigma (σ)	Free Water (%)	
20% aqueous phase	a	3.18	0.37	1.7	3.02	0.46	3.9
	b	2.93	0.4	0	-	-	-
	c	3.06	0.41	0.1	-	-	-
	2	2.67	0.38	1.9	-	-	-
	3	2.87	0.38	0.9	-	-	-
	4	2.62	0.41	0.7	-	-	-
	5	2.98	0.24	1.5	-	-	-
40% aqueous phase	6	2.84	0.27	1.1	-	-	-
	7	3.05	0.14	1.1	-	-	-
	8	3.82	0.53	7.2	-	-	-
	9	5.81	0.97	4	6.74	1.12	0
20% aqueous phase (+ milk fat)	10	4.99	1.57	0	-	-	-
	11	3.10	0.32	8.5	-	-	-
	12	2.56	0.38	0	-	-	-
	13	2.75	0.19	0	-	-	-
	14	2.79	0	0	-	-	-
15	-	-	-	-	-	-	

Table 2 NMR data of emulsions, conducted at 2 weeks

Sample	Ambient			Chilled			
	$d_{3,2}$ (μm)	Sigma (σ)	Free Water (%)	$d_{3,2}$ (μm)	Sigma (σ)	Free Water (%)	
20% aqueous phase	1	3.20	0.34	0	-	-	-
	2	2.87	0.33	0.3	-	-	-
	3	2.84	0.34	0	-	-	-
	4	2.97	0.24	0	-	-	-
	5	2.87	0.29	0	-	-	-
	6	2.98	0.17	0	-	-	-
	7	2.90	0.26	0	-	-	-
40% aqueous phase	8	4.26	0.57	5	-	-	-
	9	5.55	0.91	2	-	-	-
	10	4.96	1.44	0	-	-	-
	11	3.24	0.32	4	-	-	-
20% aqueous phase (+ milk fat)	12	2.76	0.29	0	-	-	-
	13	2.64	0.23	0	-	-	-
	14	2.84	0	0	-	-	-
	15	-	-	-	-	-	-

Table 3 NMR data of emulsions, conducted at 6 weeks

Sample	Ambient			Chilled			
	$d_{3,2}$ (μm)	Sigma (σ)	Free Water (%)	$d_{3,2}$ (μm)	Sigma (σ)	Free Water (%)	
20% aqueous phase	1	3.19	0.34	0.7	3.00	0.42	6
	2	3.13	0.28	1.4	2.99	0.26	6
	3	3.07	0.34	0	-	-	-
	4	2.94	0.29	2	2.53	0.49	6.6
	5	3.12	0.3	1.4	2.77	0.34	7
	6	3.12	0.29	0	2.60	0.42	7.7
	7	3.06	0.3	0	2.70	0.35	4.6
40% aqueous phase	8	4.15	0.52	9	3.55	0.72	9
	9	5.25	0.86	10	5.50	1.19	0
	10	5.24	1.37	0	4.39	1.28	0
	11	3.40	0.21	7.4	3.30	0.45	12.4
20% aqueous phase (+milk fat)	12	2.69	0.37	0	2.42	0.45	2.6
	13	2.99	0	0	2.72	0.11	3
	14	2.97	0	0	2.77	0	1.2
	15	-	-	-	1.74	0.57	1.6

Table 4 NMR data of emulsions, conducted at 12 weeks

Sample	Ambient			Chilled			
	$d_{3,2}$ (μm)	Sigma (σ)	Free Water (%)	$d_{3,2}$ (μm)	Sigma (σ)	Free Water (%)	
20% aqueous phase	1	3.60	0.338	0	-	-	-
	2	3.42	0.3	0.2	-	-	-
	3	3.30	0.33	0.1	-	-	-
	4	3.41	0.281	0	-	-	-
	5	3.59	0.32	0.1	-	-	-
	6	3.37	0.31	0	-	-	-
	7	3.24	0.24	0.2	-	-	-
40% aqueous phase	8	4.39	0.51	8	-	-	-
	9	6.24	0.94	1	-	-	-
	10	4.92	1.48	0	-	-	-
	11	3.50	0.27	6.7	-	-	-
20% aqueous phase (+ milk fat)	12	2.68	0.43	0.3	-	-	-
	13	2.91	0.2	0.2	-	-	-
	14	2.94	0.15	0.1	-	-	-
	15				-	-	-

Table 5 DSC data of emulsions, conducted at 1 day

Sample	Chilled		Ambient	
	<i>T</i> _{peak} (°C)	Melting enthalpy (J g ⁻¹)	<i>T</i> _{peak} (°C)	Melting enthalpy (J g ⁻¹)
1	32.99	130	33.19	115.18
2	33.01	126.63	33.41	128.63
4	33.02	128	33.08	125.13
5	33.04	129.13	33.17	128.63
6	32.9	128.38	33.38	130.88
7	32.92	129.25	32.88	132
8	32.64	118.1	32.44	134.15
9	32.83	138.68	32.47	136.99
10	32.97	131.48	32.69	131.68
11	32.86	130.93	-	-
12	32.34	134	-	-
13	32.25	130.5	-	-
14	32.36	129	32.15	97.21
15	32.17	130	32.25	126.63

Table 6 DSC data of emulsions, conducted at 1 week

Sample	Chilled		Ambient	
	<i>T</i> _{peak} (°C)	Melting enthalpy (J g ⁻¹)	<i>T</i> _{peak} (°C)	Melting enthalpy (J g ⁻¹)
1	33.67	127.75	33.34	129.13
2	33.28	127.63	33.88	123.95
4	33.13	128.63	33.16	124.53
5	33.54	106.35	33.27	128.13
6	33.10	127.63	33.26	130
7	33.36	130	33.15	129.38
8	32.8	137.67	32.89	141.6
9	32.94	131	33.01	140.15
10	33.39	116.32	33.01	142.05
11	32.89	137.57	-	-
12	32.56	95.2	-	-
13	32.08	100.51	-	-
14	32.21	96.31	32.41	94.81
15	33.55	116.18	32.05	103.16

Table 7 DSC data of emulsions, conducted at 1 month

Sample	Chilled		Ambient	
	<i>T</i> _{peak} (°C)	Melting enthalpy (J g ⁻¹)	<i>T</i> _{peak} (°C)	Melting enthalpy (J g ⁻¹)
1	33.35	134.63	33.32	133.5
2	33.45	130	33.67	128.5
4	34	129.13	33.42	131.75
5	33.21	130.88	33.71	137
6	33.67	93.89	33.56	132.25
7	33.48	130.25	33.52	133.5
8	33.46	128.68	33.35	138.72
9	33.49	125.32	33.55	146.3
10	33.41	134.07	34.01	105.93
11	33.35	125.73	-	-
12	32.56	133	33.04	133
13	-	-	32.41	141.16
14	32.55	137.63	32.51	139
15	32.86	134	32.63	133.25