INVESTIGATION OF FLUID MECHANICAL REMOVAL IN THE CLEANING PROCESS

by

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ABSTRACT

The cleaning of pipework from fluids of high viscosity is a significant problem in many food and personal care industries which can cause considerable economic and environmental impact. Three stages are identified in the cleaning of straight pipes; (i) a short core removal stage of product recovery, before water breaks through the filled pipe, (ii) the 1st cleaning stage (film removal stage) when there is a continuous wavy annular film on the wall, and (iii) the 2nd cleaning stage (patch removal stage) in which the material is present as patches on the wall.

The product recovery stage is found to influence the overall cleaning process. Conducting product recovery at low temperatures and high flow rates cause the formation of a wavy wall layer which leads to more rapid subsequent removal.

A two step CIP protocol is proposed to decrease the environmental impact of cleaning of a viscoelastic material (toothpaste) from pipework. Applying cold water in the 1st cleaning stage and hot water in the 2nd cleaning stage results in 40 % energy saving without affecting the cleaning performance significantly compared to traditional CIP protocols used in plants.

Yield stress of deposits is the key effect on the cleaning of deposits. A new dimensionless number is explored. It is physically a ratio of flow energy to the yield stress of a deposit. It has collapsed cleaning time data onto a one curve for different deposits cleaned at different velocities. It can be used to predict cleaning times of deposits or identify the governing cleaning mechanisms in cleaning.

It is found that the magnitude of pressure loss in flow can be used to quantify the flow and turbulence effect on cleaning and scale up lab scale data. The results show that although fluid mechanical removal is a complex process, cleaning times of deposits can be predicted by knowing its rheology and fluid mechanical parameters of cleaning fluid.

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NOMENCLATURE

Abbreviations

CIP	Cleaning in place
SEM	Scanning Electron Microscopy
UV	Ultraviolet
MHFS	Micro-foil heat flux sensor
CFD	Computational fluid dynamics
FDG	Fluid Dynamic Gauging
AFM	Atomic Force Microscopy
MPV	Mix proof valve
EHEDG	European Hygienic Engineering Design Group
RFC	Radial flow cell

Symbols

v	flow velocity
Re	Reynolds number
τ_w	wall shear stress
ρ	density
μ	viscosity
E_A	activation energy
T_A	absolute temperature of a deposit
R	gas constant
r	cleaning rate
T_p	temperature at the product recovery stage
F_p	flow velocity at the product recovery stage
T_c	temperature at the cleaning stage
F_c	flow velocity at the cleaning stage
h	apparent thickness of the deposit film in pipe
L	length of the pipe
R	radius of the pipe
m	mass of the material left in the pipe
m_0	original mass of the material in the fully filled pipe
E	energy consumed during CIP
Q	volumetric flow rate
t	cleaning time
ΔT	temperature difference
c_p	heat capacity of water
g	acceleration due to gravity
h_f	the friction head loss component of the system
ϵ	pump efficiency
τ	yield stress of a deposit
D	diameter of a pipe
f	friction factor
v_f	the mean flow velocity on a non affected section
K	the resistance coefficient
$h_{norm.}$	normalised head loss
G'	elastic modulus
G''	viscous modulus

Chapter 1: INTRODUCTION

Cleaning is everywhere and can be explained as the removal of foreign bodies to return a system to its original state. In the industry, the impact of cleaning operation is so crucial that it should be done in minimum time and cost. The economic costs associated with cleaning are discussed in section 1.4. Understanding how cleaning happens will help to achieve the industrial goal of minimising its costs. However, cleaning is still poorly understood, and this lack of understanding remains the biggest problem in cleaning research (Fryer and Asteriadou, 2009). One of the fundamental reasons for this problem is that most of the previous experimental studies have investigated complex deposits which need both chemical and physical actions to be removed. A number of processes are involved in deposit removal, making cleaning a difficult operation to understand and optimise; this will be illustrated in section 2.2.2. Therefore, to gain a more fundamental understanding, the cleaning process needs to be studied in the simplest possible way. For example, in daily life, our instinct is to first apply mechanical force to clean. Afterwards, if this fails, hot water or chemical solution is poured onto the soil in order to soften it and then it becomes easier to remove the soil by mechanical force. Therefore, we can anticipate that there are actually two main variables in the cleaning process;

i) Mechanical force
ii) Soil properties (rheology or cohesive bonds between the soil molecules and adhesive bonds between cleaning surface and soil molecules)

Ideally, the cleaning time of any deposit should be predictable by quantifying the effects of these variables on cleaning, but such quantification has not been resolved yet. Resolving some of these issues is the backbone of the aims of this thesis as some of the benefits would be:

- Underlying processes which cause cleaning might be revealed. Therefore, cleaning can be better understood.
- This understanding may lead to optimisation of cleaning by knowing which variables are important during the cleaning process.
- Cleaning time might be predicted by knowing the cleaning time of one soil with known properties.
- If scale up rules for cleaning could be developed, the cleaning time would be estimated at industrial scale from lab scale experiments.

1.1 Fouling and the need of cleaning

Fouling can be described as the unwanted build up of deposits on a surface and this causes regular cleaning of production equipment. Fouling is a significant problem in many different industries such as chemical, oil, food and pharmaceutical industries. The presence of the fouled deposit increases pressure drop (due to both the increase in surface roughness and the decrease in the cross-sectional area of the flow channels) and reduces heat transfer efficiency inside the processing equipment. Therefore, operating costs increase in the plant. Moreover, (especially in the food and personal care industries) fouling may compromise product quality by cross contamination or microbial growth on the deposit. Therefore, it is essential to stop production to clean the process plant. This will decrease the overall productivity of the process plant as cleaning must be done regularly. For instance, cleaning is often done daily in the food industry because cleaning the process plant properly is so vital that failure to do so may cause major brand damage if unacceptable or contaminated product reaches the market.

1.2 Cleanliness criteria and measurement

Depending on the process, a system will require different cleanliness levels. The classifications for the extent of cleaning include (Christian, 2003):

i) atomically: clean on a nano-scale;
ii) biologically (or sterile): free of microorganisms;
iii) chemically: absence of substances that may interfere with product processing;
iv) physically: no physical measurement of the deposit is possible and none can be optically detected;

For example in the pharmaceutical industry, systems must be biologically and chemically clean as a single microbe might be catastrophic. Whereas, in the personal care industry, it is enough to reach a physically clean state as the cleaning is mainly done due to reductions in efficiency (heating and fluid pumping) that result from fouling and risk of cross contamination at change over. Table 1.1 shows some of the methods used to determine the extent of cleaning according to the cleaning requirement. Some of the methods and their advantages and disadvantages will be discussed in Chapter 2.

1.3 Industrial cleaning and Cleaning-in-Place (CIP) systems

In the past, process equipment was dismantled and cleaned individually. Large-scale production results in the need for Cleaning-in-Place (CIP) systems, which involves the circulation of hot cleaning fluids through closed systems of pipes, heat exchangers, etc.

without dismantling any component on the production line. These processes have become highly developed and automated (Tamime, 2008). Cleaning regimes are generally comprised of a number of cycles. Rinsing stages, where only water is used, are often involved. For deposits that can be removed by water (as is often the case for personal products such as toothpaste, where as much material as possible is recycled), the cleaning method is just rinsing with hot water alone throughout the cleaning process. Therefore, for this kind of deposits, cleaning is achieved by a fluid mechanical removal process. However, for deposits that cannot be removed with water alone, a complex sequence of operations are required, such as (Cole, 2011):

- pre-rinse- circulation of water to remove loosely bound bulk product,
- detergent cycle- action of the cleaning chemical (acid or alkali) to break down difficult to remove soils to a form that can be removed with the fluid flow,
- post-detergent rinse- to remove any traces of the detergent or its break-down products,
- sanitization- disinfection and surface conditioning,
- final rinse- circulation of water prior to product processing.

1.4 Cost of cleaning

The fundamental aim of cleaning research is to minimise the significant process costs associated with cleaning. These include (LeBlanc, 2000):

- capital expenditure,
- equipment footprint (space occupied in factories for example for CIP equipment),
- product lost as effluent,
- loss of production (i.e. that could have been made during cleaning runs),

- water, energy and cleaning chemical usage,
- disposal and treatment of effluent.

The total cost of cleaning was found as 0.79 % of the manufacturing costs (excluding the downtime for cleaning) in one of the plants of a major healthcare company (Cole, 2011). Minimisation of environmental impact of cleaning is also becoming more important due to legislative pressures towards zero emission processes. However, currently applied conditions in CIP systems are far from optimal because (i) confirmation of cleanliness is achieved by off-line sample assessment which is an open-loop control of the cleaning process, (ii) there is not any effective measurement method found which exactly detects the end point of cleaning (a surface strictly free from residual film or soil) in the industry (Plett and Grasshoff, 2006). Therefore, forecasting to predict end point of cleaning has a great potential for cleaning optimisation as it can result in achieving the termination of cleaning operations in a timely manner. However, in-depth understanding of the cleaning processes is necessary for the success of this end-point detection philosophy (Yang et al., 2008, Martin et al., 2013).

1.5 Aims of the thesis

The above sections have highlighted the need to understand and optimise cleaning in order to reduce its considerable economic cost and environmental impact. To understand and optimise cleaning, the following steps should be considered;

- choosing an appropriate deposit type and a hydrodynamic system to study cleaning,
- determining cleaning stages thoroughly; how does cleaning happen in a given hydrodynamic system? Are there multiple stages - and if so what are the effect of individual stages on the overall cleaning time?

- in each cleaning stage, which variable is important - deposit properties or fluid mechanical parameters? Therefore, does applying different process conditions at different cleaning stages help to optimise cleaning? For instance, there may be some stages of the cleaning process, which are so dominated by fluid mechanical parameters that increasing temperature to change the deposit properties will be ineffective and cause waste.
- the effect of deposit properties on cleaning; how do deposit properties affect cleaning? Can cleaning time be predicted by knowing deposit properties?
- the effect of fluid mechanical parameters; by studying cleaning at different scales, can the underlying mechanical process which causes cleaning be identified? Can scale up be possible?

This thesis describes work that tries to find answers to the above issues which are essential to understand cleaning process fundamentally.

1.6 Structure of the thesis

Chapter 2 reviews previous work on cleaning, with special consideration of how to investigate cleaning in the most effective way to understand the whole cleaning process. It reports some of the controversial issues and gaps in the literature.

Chapter 3 explains the experimental methods used in this thesis and the procedures followed for conducting cleaning experiments on both laboratory and pilot scale.

Chapter 4 studies the product recovery stage of the pipe cleaning process. It focuses on the effects of this stage on the overall cleaning behaviour and time. It also presents findings about how modifying conditions in this stage help the overall process of fluid mechanical removal in pipes.

Chapter 5 investigates the cleaning stages of a pipe cleaning process (after the product recovery stage) in detail. This chapter focuses on the relative importance of the process variables in each cleaning stage with the aim of reducing energy usage in the cleaning process. Two different scales are investigated and an optimum CIP protocol is proposed for pilot scale cleaning.

Chapter 6 investigates the effect of rheology of a deposit on the cleaning behaviour and time to find a relationship between bulk fluid dynamical forces and internal forces of a deposit in the cleaning process.

Chapter 7 studies the scalability of cleaning and the effect of flow turbulence on the cleaning time of a deposit in aim to identify underlying processes which govern cleaning mechanically.

Chapter 8 gives conclusions drawn from this work. Possible contributions of this work is presented and recommendations for future work made.

Tables Chapter 1

Table 1.1: Methods of determination of soil remaining after cleaning (Plett and Grasshoff, 2006).

Method	**Procedure**
Visual	Presence of soil, Wettability, Adherence of gas bubbles
Optical	Adsorption (transparent surfaces), Reflectance
Microbiological	In situ Sterile cotton-wool swab, Rinsing with nutrient solution
Radiological	Incorporation of labelled tracers to product, microorganisms, or fouled layer
Gravimetric	Weight of remaining soil
Chemical	Dissolution of remaining soil and chemical analysis

Chapter 2: REVIEW OF CLEANING STUDIES

Chapter 1 has highlighted that the lack of understanding in the cleaning process is the most important problem in cleaning research. To understand cleaning and minimise cleaning costs, there are five critical issues which should be analysed thoroughly before beginning any experimental work:

i) choosing the best monitoring and experimental method to study cleaning,
ii) types of deposits and cleaning mechanisms,
iii) choosing proper hydrodynamic system to study cleaning,
iv) the effect of process parameters on cleaning,
v) forces involved in the cleaning process,
vi) the relation between lab scale and pilot scale experiments.

This literature review is conducted according to the above issues. Main conclusions, what kind of experiments should be done, controversies and gaps in the literature are highlighted to help select suitable methods to achieve the aims of this thesis.

2.1 Cleaning monitors

Determining and choosing the most efficient monitoring method is the first stage in the understanding of cleaning process. Identifying and understanding the removal mechanisms of a soil is the key step, hence, visual observation is crucial in cleaning studies. Christian (2003), Aziz (2007), Bird and Fryer (1991), Goode (2011) and Cole (2011) observed cleaning visually and successfully determined cleaning behaviours of the various deposits such as tomato paste, whey protein, egg albumin, yeast and toothpaste. Visualisation was carried out in a variety of ways, including filming of discs coated with deposit being cleaned in a glass channel. Bird and Fryer (1991) studied the cleaning of whey protein deposit and analysed the deposit surface visually by Scanning Electron Microscopy (SEM). This was an ex-situ method, therefore, the deposit was removed during cleaning for analysis. They were able to relate cleaning behaviour with surface morphology of the deposit. However, ex-situ methods are impracticable in the industry.

It is also important to investigate the kinetics of cleaning to understand dependence of process parameters on cleaning. Jennings et al. (1957) used a radioactive labelling method to study the kinetics of cleaning of milk deposit. Again this cannot be used in the industry, but they successfully found first order removal kinetics of milk deposit depending on cleaning chemical concentration. Likewise, Yan et al. (1997) used a radioactive tracer technique to study oil film removal from stainless steel pipe. They found that oil was quickly removed due to applied shear stress by the flushing fluid when the oil film was still thick. However, oil could not be removed totally, and the roughness of the pipe wall had a considerable effect on the amount of residual oil remaining in the tube at the end of the cleaning process. The oil removal was retarded when the rougher tubes were used.

A chemical protein assay was used to measure the cleaning kinetics of a protein containing deposit (Bird and Fryer, 1991, Gillham et al., 1999, Tuladhar et al., 2002). By this method, Gillham et al. (1999) found useful information such that there were three stages in the cleaning of whey protein deposit and process parameters which dominated the cleaning rate changed between each cleaning stage. Initially, temperature of cleaning solution controlled the cleaning whereas in the last stages of cleaning, flow rate became the dominating parameter. Therefore, they concluded that optimisation could be made in the industry if the beginning of each cleaning stages could be detected by proper monitors. Similarly, Xin et al. (2004) used continuous ultraviolet (UV) assay method to establish removal kinetics of whey protein deposit. They were able to find that dissolution of the deposit in the cleaning solution was the controlling process in the removal of this deposit at low velocities. Lelievre et al. (2001) used discontinuous method to measure removal kinetics of Bacillus Cereus spores from stainless steel pipes. They cleaned twenty soiled pipes at the same time and removed four pipes at five minute intervals. This allowed them to explore the effect of soiling conditions on the removal rate of spores. They found that adhesion medium did not affect the removal rate of Bacillus Cereus spores.

Monitors such as micro-foil heat flux sensor (MHFS) and turbidity meter (which were used to determine the extent of cleaning (Christian, 2003, Aziz, 2007, Gallot-Lavallee et al., 1984, Cole, 2011, Goode et al., 2010)) could not provide kinetic information despite their paramount importance for the industry. For instance, Gillham et al. (1999) compared data from a MHFS with removal rate measured by chemical assay method and they found that heat recovery did not happen in the same way as removal rate. Turbidity meter also failed to provide kinetic data since it could not measure lumps of deposit as opposed to dissolved material (Cole, 2011).

Measuring pressure drop and heat recovery has been used widely in plants to monitor the extent of fouling and cleaning. However, these methods can neither be sensitive to small amount of deposit nor provide kinetic data for optimisation purposes (Robbins et al., 1999, Changani, 2000).

Overall, the literature on monitors of cleaning has showed that monitoring methods can be divided into two groups:

- i) *For industrial purposes:* the monitoring method should be;
  - a) in-situ; the process cannot be stopped and the deposits should not be taken out from the cleaning surfaces for measurements,
  - b) continuous; allowing the monitoring of deposit removal on line without stopping the operation.
- ii) *For laboratory purposes:* This category should focus on understanding of the cleaning process thus monitoring method;
  - a) should allow cleaning processes to be visualised to identify critical removal mechanisms,
  - b) should reveal the kinetics of the cleaning process to understand dependence of process parameters on cleaning,
  - c) can be ex-situ if necessary to gain understanding of what is happening to the deposit during cleaning.

It can be concluded that an experimental method should allow cleaning rate data to be monitored to understand the cleaning process in detail.

2.2 Types of deposits and cleaning mechanisms

After selecting a proper monitoring method, choosing an appropriate deposit to study cleaning is the next step in the understanding of the cleaning process.

2.2.1 Types of deposits

It is difficult to compare cleaning of different deposits in a way which generates mechanical understanding. Therefore, the best way of cleaning each type of deposits is developed independently. To permit results from different cleaning problems to be compared across the industry, Fryer and Asteriadou (2009) classified deposits based on the cleaning fluid which they need to be cleaned:

i) Type 1 deposits: Highly viscous or viscoelastic deposits which can be removed by the action of water alone. In the personal care industry, often the deposit (such as cream, shampoo, toothpaste, etc.) is the process fluid itself. Some food deposits such as cooked tomato paste, ketchup, yoghurt, mayonnaise, etc. are also in this group.

ii) Type 2 deposits: Biofilms which need biocide to kill adhered microorganisms.

iii) Type 3 deposits: These are solid deposits which need alkali or acid chemicals to be removed in addition to water flow action. Chemicals either dissolve the deposit or transform the deposits into a removable form. Alkali chemical is used for organic deposits whereas acid is used for mineral scales.

2.2.2 Cleaning mechanisms

A number of studies have been made; most work has been done on types 2 and 3 deposit that require chemical for complete removal.

Type 3 deposits

Cleaning process is very complex for this group of deposits since both chemical and fluid mechanical removal are involved in the cleaning process. Chemical removal process may involve (Plett, 1985);

i) *reaction* between cleaning chemicals and cleaning fluid, therefore, much of the cleaning chemicals may not take part in the removal of deposit,

ii) *mass transport* of the cleaning chemicals to the surface of the deposit. The thickness of any fluid boundary layer may be important here. It is affected by turbulence (flow conditions) or concentration gradients (molecular diffusion) and adsorption on the liquid–solid interface,

iii) *diffusion* of the cleaning chemicals into the deposit. This process depends on the deposit structure and the rate of diffusion will be a function of the structure. Penetration of cleaning chemicals into the deposit may be increased by surface active agents due to their wetting ability. Some deposit may be such that without this action they will resist the cleaning chemical, for instance Biofilms (Bremer et al., 2006),

iv) *reaction* between cleaning chemical and deposit components. It may include melting, mechanical break-up, wetting, swelling, desorption, emulsification, hydrolysation of proteins, saponification of fats and dispersion,

v) *diffusion* of the reaction products back to the deposit surface,

vi) *transport* of the reaction products to the bulk fluid. Again concentration gradient and flow conditions determine the removal rate.

Listing this sequence of six steps does not imply that they always have to occur (Plett and Grasshoff, 2006). The above processes are only one part of the removal process. Fluid mechanical removal may also be involved, if the deposit does not immediately dissolve due to reaction, as one major effect of cleaning chemicals is to weaken the cohesive bonds between elements of deposit to generate a form of deposit that can be removed by fluid flow. Hence, deposit is removed non-uniformly by swelling and subsequent break up of the deposit into discrete islands (Bird and Bartlett, 1995, Grasshoff, 1989, Christian, 2003). This is schematically shown in Figure 2.1.

For the case of whey protein deposit removal, a cleaning rate curve involves three stages which is shown in Figure 2.2 (Gillham et al., 1999, Bird and Fryer, 1991):

i) *Swelling stage:* Upon contact with the chemical, deposit swells and protein matrix of high void fraction is formed. Cleaning rate starts to increase in this stage. Temperature controls the process.

ii) *Platue stage:* Detachment of the swelled deposit by fluid shear or dissolution happens. Cleaning rate is maximum here and stays constant until only patches of deposit are left on the surface. Flow velocity is found to be the governing process parameter in this stage.

iii) *Decay stage*: Removal of remaining patches of the deposit is observed. In this stage, flow condition is also the governing parameter and cleaning rate gradually decreases to zero.

Egg albumin deposit is also observed to swell like whey protein deposit when it contacts with the chemicals. However, it is removed uniformly by dissolution (Aziz, 2007). Cohesive bonds between egg albumin molecules might be strong enough to resist breaking up into smaller parts by fluid flow forces or different reactions might occur in egg albumin. Therefore, forces inside the deposit play the pivotal role in the determination of the removal mechanism.

It can be concluded for type 3 deposits that understanding the overall cleaning process by studying this type of deposit would be very difficult as the removal process involves the very complex removal mechanisms described above. Moreover, different removal modes were observed even though deposits were in the same group. This is because the properties of soil (cohesive and adhesive forces) also controls the cleaning behaviour of a soil.

Type 2 deposits

For the cleaning of type 2 deposits (biofilms), chemicals are also needed both to decrease adhesive bonds between microorganisms and surface for easy removal and to kill the residual microorganisms after removal. Therefore, the cleaning process may include (Plett, 1985);

i) fluid mechanical removal,
ii) transport of chemicals into the surface of the film and diffusion into film,
iii) reaction of chemicals to weaken adhesive bonds between surface material and biofilm,
iv) transport of the spores or film into the bulk cleaning fluid.

Goode (2011) characterised the cleaning mechanism of yeast and found two main removal stage:

i) *Swelling stage:* Deposit hydration and swelling occurred. Very little cleaning was observed here. This stage was mostly temperature dependant.
ii) *Removal stage:* Deposit was removed by fluid action in part by dissolution and in patches. Temperature was again observed to be more dominant parameter in this stage.

Discrete removal of yeast occurred probably due to weakening of the cohesive bonds by deposit hydration and swelling. This behaviour resembles the removal behaviour of whey protein deposit although they are different kinds of deposits.

Studying the cleaning of type 2 deposits is also problematic due to hardness of producing consistent fouling deposit and the fouling conditions may dictate cleaning behaviour. It was observed that the removal rate of Bacillus spores was influenced by the flow conditions applied during the soiling (attachment) procedure (Lelièvre et al., 2002a). Guillemot et al. (2006) and Goode et al. (2010) studied the cleaning of yeast and they found different removal behaviours. Guillemot et al. (2006) found only 10 % reduction in the number of yeast cells in the studied range of wall shear stress (0-80 Pa). However, Goode et al. (2010) found up to 50 % removal of the deposit area in the shear stress range of 0-1.24 Pa. This was probably due to the different conditions used to prepare yeast deposits: the yeast was aged at 20 °C and for 1 h in the work by Guillemot et al. (2006) and at 30 °C and for 5 days in the work by Goode et al. (2010). Biofilms are often formed by discrete species which can cause the existence of different adhesive and cohesive forces in the biofilm. Therefore, this may also affect the cleaning behaviour even if the same cleaning conditions are applied and most of the cases prepared deposits on which experiments are conducted are not really representative of industrial systems (Fryer and Asteriadou, 2009). Thus, it would be very hard to understand what are the independent effects of fluid mechanical and chemical removal processes on the deposit removal when type 2 deposits are used to study cleaning.

Type 1 deposits

For the cleaning of type 1 deposits, chemicals are not used. Hence, cleaning may involve three physical processes;

i) fluid mechanical removal (removed by fluid action alone),
ii) diffusion of water into deposit and
iii) dissolution of the deposit into the bulk fluid if deposits can be dissolved in water.

Christian (2003) studied cleaning of baked tomato paste which could be removed by water alone. It was generally removed in discrete pieces directly from the surface as shown in

Figure 2.3. This was because cohesive bonds between tomato paste molecules were stronger than adhesive forces between deposit and cleaning surface (Fryer et al., 2006). Size and the frequency of the detachment of each piece depended on the process parameters. For instance, tomato paste was removed rapidly in smaller pieces from the surface at the highest flow rate and temperature studied; 2.3 l min^{-1} and 70 °C. Notably, at low temperatures (30 °C) and high flow rates (2.3 l min^{-1}), the entire deposit lifts from the surface as a whole.

Cole (2011) studied another type 1 deposit which was toothpaste. In contrast to baked tomato paste which directly removed from the cleaning surface, toothpaste deposit was generally removed from the top of the deposit in the same manner as the removal of whey protein deposit and yeast. Initially, large chunks of toothpaste were removed from the top leaving discrete patches on the surface. Then, patches were eroded away until cleaning was completed. Also, for toothpaste deposit, no preparation is needed and it can be used as supplied. Therefore, the cleaning behaviour of this kind of deposit should be the same in lab and industrial systems.

Research on type 1 product types suggests they are often shear thinning. They have an effective viscosity that is a function of shear rate, and exhibit viscoelastic properties (Goode, 2011). Cleaning of type 1 deposits is probably controlled by the rheological properties of the deposit and the flow properties (such as the shear force applied to the deposit) of the cleaning fluid (Fryer and Asteriadou, 2009).

All of the above studies show that when chemicals are used, the cleaning process becomes very complex. It involves chemical processes (dissolution-reaction) as well as fluid mechanical process. Also, the preparation of deposits is found to be crucial. All of these cause existence of different removal mechanisms in the cleaning of same type of deposits. Most cleaning studies have investigated removal of types 2 and 3 deposits. Consequently, true understanding of cleaning process remains unresolved. Hence, type 1 deposits are used in this thesis to make the cleaning problem simpler. Moreover, removal mechanism is shown to depend on the soil properties. Therefore, the effect of rheology of deposits on cleaning should be studied to better understand cleaning. This issue is investigated in Chapter 6.

2.3 Types of hydrodynamic systems to study cleaning

So far, the cleaning process has been studied in various hydrodynamic systems. In order to understand cleaning, it is essential to produce well-controlled flow conditions. The parallel plate flow cell, the radial flow cell, fluid dynamic gauging, the impinging jet, the rotating disk and pipework are amongst the examples of these systems. Schematics of these systems are shown in Figure 2.4.

The parallel plate flow cell has been generally used to allow in situ monitoring of cleaning process and produces well controlled flow with constant shear stress along the sample surface. It was used in various applications such as cell detachments (Bouafsoun et al., 2006, Goode et al., 2010), oil drop removal from stainless steel surfaces (Boulangé-Petermann et al., 2006), food deposits removal; whey protein (Christian, 2003), egg albumin (Aziz, 2007). However, different experiments should be conducted in the parallel plate flow cell in order to investigate the effect of flow on cleaning.

On the other hand, the impinging jet, the radial flow cell, and the rotating disk have a common advantage that they generate different ranges of flow action on the studied sample in a single experiment. Therefore, critical wall shear stress values can be found by these systems to detach soils from surfaces (Detry et al., 2009b). Various studies have been conducted with these hydrodynamic systems. Bacterial detachment was studied by using the jet impingement (Bayoudh et al., 2005, Bundy et al., 2001), particles removal was investigated by using the radial-flow cell (Detry et al., 2007, Detry et al., 2009b) and cleaning of milk protein deposits was studied by using the rotating disk (Morison and Larsen, 2005, Morison and Thorpe, 2002).

Fluid dynamic gauging has been generally used to measure online thickness of the samples (Chew et al., 2004a, Tuladhar et al., 2000) or the swelling of a deposit during the cleaning process (Chew et al., 2005, Chew et al., 2006).

However, a common disadvantages of the all mentioned hydrodynamic systems are that results obtained from them are hardly applicable to the industry and are difficult to scale up. For instance, Cole et al. (2010) compared the cleaning of fouled coupons in a horizontal parallel plate flow cell with the cleaning of fully filled pipes which are generally encountered in the industry. They identified different removal modes in these flow systems. Therefore, they could not find a simple relation between cleaning of a pipe and a planar surface. Moreover, Jensen and Friis (2004, 2005) tried to predict cleanability of a mixed proof valve by investigating cleanability of a radial-flow cell. They concluded that the cleaning of a complex equipment cannot be predicted by the cleaning of a simple geometrical system because complex phenomena such as fluid exchange at the vicinity of the surface and wall shear stress oscillation take place in a complex equipment, which further enhance cleaning. Therefore, Detry et al. (2009a) concluded that it was important to do experiments in pilot plant scale to assess the cleanability of closed equipments. For this reason, a study of cleaning of pipes at pilot scale would be valuable. In this thesis, circular straight pipes are chosen as a hydrodynamic system to investigate the cleaning process. Studying the pipe cleaning offers several advantages:

- It is easy to generate well-controlled flow in pipes thus the parameters such as wall shear stress, Reynolds number or friction loss can easily be calculated.
- Studying the cleaning of pipes with different diameters may allow scale up.
- The results obtained can be easily implemented in the industry at least in the pipework of a processing line.

2.4 The effect of process parameters in cleaning

To understand and optimise cleaning, the effect of process parameters on cleaning should be analysed. There are mainly three energies which are required to remove a deposit from a surface (Romney, 1990).

(i) mechanical or kinetic energy (depends on equipment design as well as flow rate, turbulence or wall shear stress),

(ii) thermal energy (depends on temperature) and

(iii) chemical energy (depends on the type and concentration of the chemicals).

2.4.1 Flow effect

Mechanical energy can be generated by scrubbing, brushing and scratching actions to remove a soil when an industrial equipment is dismantled. However, mechanical energy is supplied by the fluid flow in cleaning in place systems. It also acts as a carrier for the chemical and the heat energy. The flow effect on cleaning of a deposit is often characterised in terms of flow velocity (v), Reynolds number (Re) and surface shear stress (τ_ω according to Blasius equation).

$$\mathrm{Re} = \frac{Dv\rho}{\mu} \tag{2.1}$$

$$\tau_{\omega} = 0.079\,\mathrm{Re}^{-0.25} \cdot \frac{1}{2}\rho v^{2} \tag{2.2}$$

where D is pipe diameter (m), v is average flow velocity (m s^{-1}), ρ is density (kg m^{-3}) and μ is viscosity (kg $m^{-1}s^{-1}$) of the liquid (Coulson et al., 1999).

However, one of the biggest gaps in the literature is that it is still not clear how to characterise the flow effect in the best way. There are many controversies among the published studies. The guideline of a flow circulation velocity of at least 1 m s^{-1} was reported (Timperley, 1989). Also, as a first recommendation a velocity of 1.5 m s^{-1} is quoted (Tamime, 2008). However, both have little theoretical underpinning. For instance, Bird and Fryer (1991) found short cleaning times even at velocities less than 0.6 m s^{-1}.

Jennings et al. (1957) found a threshold Reynolds number of 25,000, below which the mechanical effect was negligible and concluded that turbulence (in terms of Re) was the most influential factor in the cleaning process. Hankinson and Carver (1968) also related cleaning data with the Reynolds number. However, Bird and Fryer (1991) noted that cleaning rate was not changed significantly when moving from laminar to turbulent flow in geometries they considered. Timperley (1981) explored that cleaning of microorganisms from stainless steel pipes was much better correlated with the wall shear stress, than with Reynolds number.

Hasting (2008) postulated that the cleaning process was controlled by boundary layer thickness rather than shear forces, since if not, there should be a velocity value at which all deposits should be removed instantaneously (total cleaning time is zero). However, this was not the case in cleaning and many studies showed that cleaning times decreased and eventually reached an asymptote value by increasing flow velocities (Timperley and Smeulders, 1988, Cole et al., 2010). Hasting (2008) continued that detergent diffusion across the boundary layer could be controlling, and increasing flow velocity might eventually result in sufficiently thin boundary layer that the diffusion process would be no longer controlling, thus an asymptote was observed. At this point, the reaction rate between detergent and soil might become controlling. This also highlights the complexity of the processes involved in the cleaning of heat-induced soils. Therefore, as stated in section 2.2.2, it is essential to study simple soils to analyse the flow effect on cleaning clearly.

Lelievre et al. (2002b) measured local wall shear stress values by an electrochemical method in different pipe configurations and they confirmed the significant effect of mean wall shear stress on bacterial removal. Nevertheless, they observed some low shear stress zones in pipes which were very cleanable. In these zones, the fluctuation rate, a kind of turbulence level, was found high. Therefore, they concluded that it was essential to consider not only mean wall shear stress, but also the fluctuation rate in order to predict the cleanability of pipes.

Pulsed flow was also considered by many authors to enhance the cleaning rates (Augustin et al., 2010, Bode et al., 2007, Scholer et al., 2011, Blel et al., 2009). They all found that pulsed flows did promote cleaning process. However, due to the complexity in the cleaning process, it remains uncertain, whether the main effect results from the flow effect (enhancement in wall shear stress and potential change in flow direction) or from the chemical effect (mass transfer enhancement) due to pulsation. This also illustrates the importance of studying cleaning of simple deposits which are cleaned by the action of fluid flow alone to characterise the flow effect.

Friis and Jensen (2002) studied the cleanability of closed processing equipments such as a valve house, an up-stand and various expansions in tubes. They predicted the wall shear stress values by computational fluid dynamics (CFD) simulations. They also observed that efficient cleaning could be achieved even if the critical wall shear stress were not exceeded. They concluded that the degree of turbulence, the type of flow pattern and three dimensional flow should be taken into account, and they identified two controlling factors for cleaning; the wall shear stress and the nature and magnitude of recirculation zones. This study was noteworthy since it clearly illustrated the necessity of studying different geometries to study the flow effect on cleaning.

The flow effect in different geometries

Jensen and Friis (2005) investigated the cleanability of a mix proof valve (MPV) fouled with B. Stearothermophilus spores. MPVs are certified by the European Hygienic Engineering Design Group (EHEDG) as a hygienic design through several hygiene tests. In these tests, it was observed that the MPV was easier to clean than a straight pipe. They used the idea of a critical wall shear stress to explain the cleanability of the MPV. A radial flow cell (RFC) was used to predict critical wall shear stress under which cleaning did not happen in the RFC. After cleaning tests, the residual soil was identified on the test surface of RFC at a critical radius of approximately 35 mm, which corresponded to a critical wall shear stress of 3 Pa. Then, they tested if this critical wall shear stress was applicable to the prediction of cleanability of the MPV. They showed that;

- most of the areas of low shear stress well below the critical wall shear stress were clean,

- only areas of extremely low wall shear stress and interestingly a few areas of wall shear stress higher than the critical wall shear stress of 3 Pa were not clean after cleaning.

The results proved that the cleanability of a complex equipment could not be predicted by wall shear stress alone.

Jensen et al. (2007) studied the effect of increasing velocity on the cleaning of a T-piece. They identified some areas which were exposed to very low velocities, hence they were always last to clean. Also, increasing flow velocity did not change their location in the T-piece and only very small increases were seen in the local velocity values of these shadow areas. Therefore, they concluded that the problem areas might be reduced through increasing the flow rate, but the problem areas would always be in the same place where they used to be.

Lelievre et al. (2003) investigated the effect of local wall shear stress variation on the cleanability of a three-way valve. An electrochemical method was used to measure the wall shear stress in selected zones. Their general conclusion was that the geometry of a complex equipment and the flow pattern during the cleaning process have a big influence on the cleanability level. They drew this conclusion by the following findings:

- The valve outlet was found to be highly cleanable despite low values of mean local wall shear stress and fluctuation rates.
- While rubber materials are often believed to be poorly cleanable, they observed that some seat areas which were made of rubber were more cleanable than adjacent stainless steel zones.

Blel et al. (2008) studied the cleaning of a part of a dairy processing line consisting of straight pipes, gradual asymmetric expansion or contraction pipes and bends by using nonintrusive flow characterization. Gradual expansion was found to be poorly cleanable due to the fluid separation, which caused flow disturbance. Wall shear measurement by the electrochemical method showed that gasket and welding zones caused an increase of the mean wall shear stress by promoting flow disturbances with a nonboundary layer detachment from the wall. However, these zones presented low-cleanability level. They predicted that this might be due to the high surface roughness in gasket and welding zones.

Plett and Grasshoff (2006) mentioned that turbulent flow conditions might be more effective than average wall shear stress on cleaning due to time fluctuating characteristics of turbulent flow. Therefore, cleaning effect can be improved by turbulent promoting designs or pipeline configuration. Hence, the effect of turbulence on cleaning is studied and results are presented in Chapter 7 through studying different pipe line configurations.

2.4.2 Temperature effect

Heat is the second form of energy that is used in the cleaning process. Generally, the cleaning rates increase as temperature increases (Hankinson and Carver, 1968, Jennings, 1959, Timperley and Smeulders, 1988). However, too high a temperature may result in complex fouling deposit due to heat alteration reactions. Moreover, detergent solution and the surface of the equipment may react at high temperature and this can cause defects on the equipment surface.

Arrhenius type of equation is usually used to express the temperature effect on cleaning rates in the range of temperatures where heat alterations of soils do not happen:

$$r = Ae^{-E_A/RT_A} \qquad (2.3)$$

where r is the removal rate, A is pre-exponential factor, E_A is activation energy, R is the gas constant and T_A is an absolute temperature of deposit.

Depending on the deposit type, the temperature effect on cleaning rates can be due to various reasons:

- Change in the rheology. If a deposit does not require detergent solution, increasing temperature probably decreases the viscosity or yield stress of the deposit and this will promote cleaning rates (Fryer et al., 2011).
- Change in the strength of internal forces. The strength of cohesive bonds between deposit molecules or adhesive bonds between deposit molecules and material surface was found to decrease by increasing temperature (Akhtar, 2010).
- When the diffusion process controls the removal, diffusive transport or physicochemical transformations (low E_A) limit the overall cleaning rate even if temperature is increased (Hoffmann and Reuter, 1984).
- When chemical reactions control the removal, the chemical reaction between the detergent and the soil (high E_A) determines the cleaning rate (Bird and Fryer, 1991). The cleaning process here is highly depended on temperature.

2.4.3 Chemical effect

The last form of the energy supply is the chemical energy in the cleaning process. Detergents are used to change the form of a fouled deposit into an easily removable form or to dissolve deposit. Increasing the detergent concentrations increases the supplied energy. However, opposite to the flow and temperature effects, increasing the concentration of detergent will not necessarily give better cleaning. It was proven that there were optimum values of chemical concentration which resulted in the best cleaning performance (Jennings, 1965, Jeurnink and Brinkman, 1994, Grasshoff, 1997). For the removal of protein, starch and glucose deposits, optimum cleaning agent concentrations were observed (Bird and Bartlett, 1995). For instance, optimum NaOH concentration of 0.5 wt % was reported for the removal of whey protein deposit. Further increase in the NaOH concentration increased the cleaning time (Bird and Fryer, 1991). Bird and Fryer (1991) attributed this to the structure of the deposit since they found that after exposure to the chemical solution with a concentration of more than 0.5 wt %, the deposit structure was less open, thus it may not be susceptible to the fluid shear. The chemical action may also be inhibited by high-velocity flow, if there is insufficient time for the chemical to act on a deposit.

As can be concluded from the literature, all three forms of energies are interrelated with each other in the cleaning process. The flow of cleaning fluid, giving mechanical energy is the most important form of energy type. Once the effect of flow is characterised and better understood, the effect of temperature and chemical concentration on cleaning would be realized subsequently. Therefore, studying simple deposits at ambient temperatures may help to better understand the effect of flow alone on the cleaning process. Hence, in Chapter 7, characterisation of the flow effect on cleaning is investigated by studying cleaning of simple deposits at ambient temperature to exclude the effects of heat and chemical energies on cleaning.

2.5 Forces involved in the cleaning process

Removal of deposits from surfaces is mainly achieved by two processes:

Dissolution: If the deposit can be dissolved in the cleaning fluid, cleaning may occur even in static conditions (Mercadé-Prieto and Chen, 2006).

Mechanical removal: Fluid mechanical forces should overcome the adhesive and cohesive forces in a deposit. Otherwise, removal of the deposit cannot be achieved completely, if dissolution does not take place.

The physical stress was found as 1250 Pa to remove a milk deposit from a surface (Grasshoff, 1997). It is much greater than the fluid mechanical stress at the surface of a plate heat exchanger (1-20 Pa). This shows the importance of the dissolution process. However, revealing the relationship between fluid mechanical forces and cohesive/adhesive forces is still crucial to understand the cleaning process in detail. There are three steps in achieving this goal:

i) Measuring or estimating fluid flow forces. Inertial forces or surface shear forces can be calculated. Computational Fluid Dynamics (CFD) can be used to estimate shear forces and velocity profiles in a system (Jensen and Friis, 2007).

ii) Measuring the internal properties of deposits. For this purpose rheology, micromanipulation technique, Fluid Dynamic Gauging (FDG) technique or Atomic Force Microscopy (AFM) have been used in the literature.

iii) Changing rheology of the same deposit systematically to study the effect of deposit properties on cleaning. In the literature, there has been no any work which studies this issue despite its valuable importance in the understanding of the cleaning process. Hence, it is investigated in this thesis and results are reported in Chapter 6.

The measurement techniques are discussed in the next sections.

2.5.1 The micromanipulation technique

The method uses T-shape probe made of stainless steel and it measures the force to draw this probe across the deposit surface by a shovelling action. If the deposit is removed right from the surface, measured force can be accepted as a kind of adhesive force. If the cut height (the distance between the probe and the surface) is more than zero, some sort of cohesive forces are measured (Liu et al., 2002). It is an easy technique to use although it is expensive to construct initially. It has the advantage that the probe can be adjusted so that the deposit can be cut at different heights and the balance between cohesive and adhesive effects can be determined directly. For instance, the effect of cut height was studied for egg albumin, whey protein, bread dough and tomato paste deposits to identify adhesive and cohesive effects on cleaning (Liu et al., 2006). For instance, Figure 2.5 showed that the energy for partial removal of egg albumin deposit exceeded that for total removal. They successfully found that tomato paste, bread dough and egg albumin deposits had a higher cohesive than adhesive strength, whilst whey protein had lower cohesive strength than adhesive strength. Yeast also was found to have stronger adhesive forces than cohesive forces (Goode, 2011). Hence, these results can explain the observed cleaning behaviours of these food deposits mentioned in section 2.2.2: When adhesive forces are stronger, deposits are removed from top to bottom in layers. On the other hand, deposits are removed directly from the surface in large chunks, when cohesive forces are stronger. However, this technique is not precise for the following reasons:

- In addition to adhesive forces, it also measures deformation and thus includes some measure of the rheology of the deposits, how they deform.

- Moreover, mass of the deposit drawn by the probe may affect the result, thus different cut height measurements or drawing different amounts of same deposit right from the surface may give different results.

2.5.2 The FDG technique

This is an inexpensive and portable equipment that can be used to measure the thickness of deposit meso- and macro-layers on surfaces, and thus good for studying swelling kinetics of proteins (Gordon et al., 2010). How it measures the deformation strength was explained by Chew et al. (2004b): The fouled surface was immersed in a Newtonian liquid and a nozzle was brought close to the surface. A suction pressure differential was applied so that liquid was drawn into the nozzle. Flow rate measurement gave the location of surface as the position of the nozzle was known to micron accuracy. Deformation or yielding of the surface resulted in changes in deposit thickness, thus flow rate changed. This laminar gauging flow imposed stresses, so deformation strength of deposit was attributed to these stresses. Finally, CFD simulations of the laminar gauging flows were used to calculate these stresses to identify the forces associated with deformation of the deposit.

A comparative study was made between the micromanipulation and the fluid dynamic gauging probe for strength measurements (Hooper et al., 2006a). Removal of baked tomato puree deposits from stainless steel coupons was studied with both techniques and both of them showed quantifiable effects of baking time and hydration time on removal behaviour. Although, they displayed similar trends, the gauging results showed noticeably greater scatter, probably because this was a more localised measurement. Whereas, a superior reproducibility and a consistent trend were observed in the micromanipulation data since this technique was reporting an average of the rupture force over the whole deposit area. Moreover, gauging technique cannot allow adhesive and cohesive interactions in the deposit to be explored separately.

2.5.3 The AFM

In this technique, a cantilever is placed into contact with a deposit and the force of detaching the cantilever is determined. Therefore, it only measures the adhesive forces between a surface and the deposit molecules (Akhtar, 2010). This is the main disadvantage of this technique as the removal of deposits often occurs by the detachment of the top layers of the deposit. Moreover, it is a much more sensitive measurement method than micromanipulation and gauging method, and more parameters are required to set up experiments. However, adhesive strength of a series of deposits (toothpaste, caramel, Turkish delight and sweetened condensed milk) was studied to compare micromanipulation and AFM methods and both of them gave comparable results as shown in Figure 2.6 (Akhtar et al., 2010). AFM works at the nm-scale. Therefore, the main possible advantage of the AFM is that it may be used in the development and characterisation of potentially new antifouling surfaces, and those surfaces may initially only be available in very small quantities. Thus, it may help material designers to test surfaces without having to do experiments at a higher scale.

2.5.4 Rheology

Rheology is the science of the deformation and flow of matter. Therefore, when the fluid mechanical removal is the governing process in cleaning, rheology will be the best characterisation method of soils to predict the removal modes and cleaning time of a soil. It should be possible to predict the cleaning time of one soil by the knowledge of how other soil with a known rheology cleans (Fryer and Asteriadou, 2009). Therefore, there are two salient aspects of studying rheology in cleaning research:

(i) It may reveal the removal modes of deposits: If any deposit has a non zero yield stress, it resists flow and behaves as a solid, thus it may be removed in chunks and by erosion. This has been observed in the removal of toothpaste which has a high yield stress value (Cole et al., 2010). On the other hand, if any deposit has a zero yield stress, it will be simply removed by flowing. Then the problem becomes analogous to annular flow in gas-liquid systems (in which a core of low-viscosity, generally air, displaces a wall film of water), and the same kind of mathematics can be used in developing computational models (Fryer et al., 2011).

(ii) It can also be used to gain an understanding on the internal microstructure of the material: For instance, dynamic oscillatory stress tests measure the response of a material in terms of its elastic (G') and viscous modulus (G"). Therefore, the linear viscoelastic region can be determined. The importance of this region is that it reveals the maximum stress that can be applied to the material without destroying the internal structure of it (Gunasekaran and Ak, 2000). Dynamic oscillation tests can also be used to find yield stress of a deposit. G' is a measure of stored energy (storage modulus) which describes the molecular events of an elastic nature. Whereas, G" is a measure of the energy dissipated as heat per cycle of deformation per unit volume (loss modulus) and describes the molecular events of viscous nature. Therefore, if G' is higher than G" in low applied stress values, it means material shows a kind of solid behaviour and has a yield stress. If the crossover of G' and G" is observed in higher applied stress values, this corresponding stress value might be regarded as yield stress value of a material.

2.6 Scale up in the cleaning process

Interpretation and the use of laboratory data in cleaning studies are valuable for several reasons:

- It will help decrease the cost of cleaning in the industry.
- It will reduce the cost of experiments.
- It may allow prediction of cleaning time and behaviour of one soil at industrial scale.
- It may improve understanding of the overall cleaning process.

Therefore, exploiting the lab data for industrial benefit is one of the most important issues in cleaning research. But at the same time, it is one of the most challenging issues. Fryer and Asteriadou (2009) stated that deposit type was the key factor and they discussed the problematic areas in the scale up problem:

- If heat induced deposits such as milk deposits were studied, deposits prepared at lab scale were often not representative of deposits found in industrial systems as making reproducible fouling deposits was very difficult and aging was not understood well (Changani et al., 1997).
- Most of the cases, whey protein was studied instead of milk as a model deposit to simulate pasteuriser behaviour (Christian and Fryer, 2006). However, different results were obtained to the real soils (Christian et al., 2002, Hooper et al., 2006b).
- Depending on the deposit type, scale up rules should be different. If fluid mechanical removal was the dominating process, soil rheology and flow rate would be of interest. If dissolution process controlled the cleaning, local reaction rate or solubility rather than flow rate should be considered.

Fryer and Asteriadou (2009) have concluded that in cleaning studies, the scale-up rules are not known and how to relate the cleaning rates and times at different scales is still unclear. Also Akhtar et al. (2010) mentioned that many experiments on cleaning used low flow rates and scale-up calculations were challenging.

Some scale up studies

Akhtar et al. (2010) studied surface-deposit adhesion forces at nano and meso scales. Removal of caramel, toothpaste, Turkish delight and sweetened condensed milk from stainless steel, glass and fluorinated surfaces were investigated. Atomic force microscopy was used to measure adhesive forces at nano-scale, whilst adhesive force between deposit some μm thick and surface was measured by the micromanipulation technique. Similar results were obtained from the two types of probe, albeit at different length scales. This showed that force measurements could be scaled up.

If the mechanism of cleaning is not comprehended then scaling up lab data is basically empirical. Therefore, it is essential to study simple deposits which can be cleaned by fluid flow alone. In this context, Cole et al. (2010) did valuable work to relate millimetre scale data with metre scale data by studying the removal of toothpaste. They compared the cleaning times of fouled coupons in a rectangular channel on a bench-top rig (mm scale) with the cleaning times of fully filled pipes on a pilot scale rig (m scale).

They used Reynolds number and wall shear stress to correlate cleaning time data with the process variables (temperature and flow velocity). For the correlation of Reynolds number, they tried to make the system dimensionless to relate the results from two scales. They plotted the dimensionless cleaning time (formed by multiplying the cleaning time by the ratio of mean velocity to pipe diameter) against Reynolds number as shown in Figure 2.7. Although, the data was scattered for the coupon rig, the data laid on a relatively better straight line for the pilot scale.

Also, they used wall shear stress to relate the lab scale data to the pilot plant data at different temperatures and velocities, which is shown in Figure 2.8. Compared to Reynolds number case, better correlation was observed across the two scales and cleaning time was expressed as being inversely proportional to the clean pipe shear stress.

That work was preliminary and showed that it would be possible to model the behaviour of the system using fluid mechanical parameters. However, used correlations could not successfully predict cleaning times at pilot scale by using the results at lab scale. The below issues should be considered to improve that work:

i) The data at intermediate wall shear stress and Reynolds number did not exist in their system. Overlap between two scales is unlikely since different trends are observed at both scales in Figures 2.7 and 2.8.

ii) They compared the cleaning of a coupon coated with an even layer of toothpaste with a certain thickness (2.5 mm) with the cleaning of a fully filled pipe. These hydrodynamic systems cannot be correlated because they found different removal mechanisms. For instance, for pipe cleaning; first they observed a core removal stage; a core of toothpaste was displaced form the pipe and a thick film of toothpaste was left on the pipe wall. Then the layer of toothpaste was sheared away by fluid flow and chunk removal was observed. Finally, some islands of material were left on the pipe wall and these were gradually eroded away. On the other hand, for the coupon cleaning, only erosion from the leading edge was observed and the thickness of the deposit gradually decreased.

iii) The thickness of deposit is also important for the cleaning of coupons. Most probably, putting thicker deposits on the coupon will result in longer cleaning times. However, it was unclear in their study whether studied thickness of deposit (2.5 mm) could simulate pipe cleaning process or not.

iv) Instead of coupons, it might be better relate cleaning of small diameter pipes with that of pilot scale pipes. Therefore, a study with pipes with different diameters will better reveal whether the data depends on Reynolds number or not.

v) Studying reproducible fouling deposits is crucial in cleaning research. Investigation of deposits at different amounts, temperature, structure and aging time would obviously result in different cleaning times and misleading results. Therefore, the link between fouling and cleaning should be appreciated (Wilson, 2005). For the cleaning of fully filled pipes, conditions in the core removal (product recovery) stage will determine the structure of the thin film which will be cleaned in the next cleaning stage. Therefore, in order to examine the effect of temperature and flow velocity on cleaning of fully filled pipes, the product recovery conditions should be the same in each cleaning experiments to produce consistent deposits. However, product recovery conditions were not same in the study. Hence, they observed unusual result that the cleaning time increased with increasing cleaning fluid temperature above 40 °C as illustrated in Figure 2.9. This was probably because they investigated the temperature effect on cleaning of deposits which were in different states as the product recovery (core removal) conditions were different. The effect of product recovery on cleaning is investigated in Chapter 4.

vi) Probably, cleaning of this kind of deposits depends on the rheology of the deposit and some parameters in fluid dynamics. Therefore, studying cleaning of deposits at different rheology may improve understanding of the fluid mechanical removal on the cleaning process further. Hence, Chapter 6 covers this area.

2.7 Conclusions

In the food and personal care industries, cleaning of process equipment is common because of fouling build up leading to the degradation of plant performance, loss of hygiene and the need to avoid cross contamination at product changeover. Process plant cleaning is costly, time consuming and uses many additional resources to the manufacturing operation, contributing significantly to the environmental footprint of the plant (Eide et al., 2003). Therefore, optimisation of cleaning is becoming more important. However, the main obstacle is that the cleaning process is still poorly understood. Therefore, literature review was conducted to find ways to attack this problem. The review of cleaning studies has highlighted that;

- Studying cleaning of complex deposits which need chemicals to be cleaned could not help detailed understanding of the cleaning process as it involves both chemical and fluid mechanical removal at the same time. Nevertheless, once fluid mechanical removal is comprehended, how cleaning happens with chemicals may be understood in detail. Therefore, this thesis has focused on the cleaning of simple deposits which can be removed by the fluid action alone.
- It is realised that the cleaning results obtained from one hydrodynamic system could hardly be correlated with the cleaning results of an another. Also, the flow effect on

cleaning has not been quantified yet. Therefore, cleaning of pipes is studied here as pipes are simple, the most widely encountered geometry in the plants and a study of cleaning of different diameters pipes may allow quantification of the flow effect and scaling up cleaning. The results are presented in Chapter 7.

- The removal of viscous fluids from pipework has not been studied in any detail. Generally, attention has been given only either to the way the core is removed or how the residue is removed (Gabard and Hulin, 2003, Kuang et al., 2003, Park et al., 2003). Results on the effect of product recovery stage on total cleaning has not been published before (to the best knowledge of the author), although some displacement studies with Herschel-Burkley fluids have revealed the importance of product recovery. For instance, displacement of laponite solutions by water (Gabard and Hulin, 2003) and carbopol solutions by gas (de Souza Mendes et al., 2007) was observed to be a perfect plug-like displacement which did not leave any remaining fluid behind at very low velocities. However, pipe diameters studied have been only in the range of 1 to 24 mm which are far from the industrial scale (Poslinski et al., 1995, Huzyak and Koelling, 1997, Kamisli and Ryan, 2001). Studying product recovery stage may develop further understanding of the cleaning of pipes. This issue is covered in Chapter 4.
- Measuring the removal rate is realised to be one of the most important steps in the understanding of the overall cleaning process. Therefore, a simple experimental method is developed to obtain cleaning rate data which is explained in section 3.5.1. This has resulted in determining different stages of cleaning. Then, optimisation of the CIP process in terms of energy consumption is made in Chapter 5 by the response surface methodology.

- Soil properties are found to affect the cleaning time and behaviour. Therefore, the cleaning of various simple deposits is studied to investigate the effect of deposit properties (rheology) on the cleaning behaviour and cleaning time of deposits. The results are reported in Chapter 6.

Figures Chapter 2

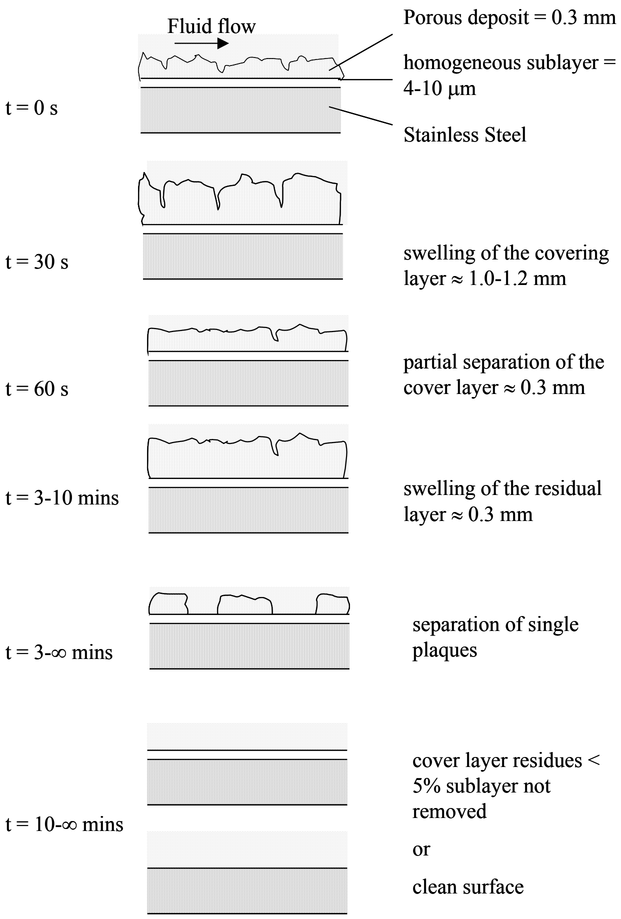


Figure 2.1: Schematic diagram of the removal of milk deposit from a solid surface using NaOH (Grasshoff, 1997).

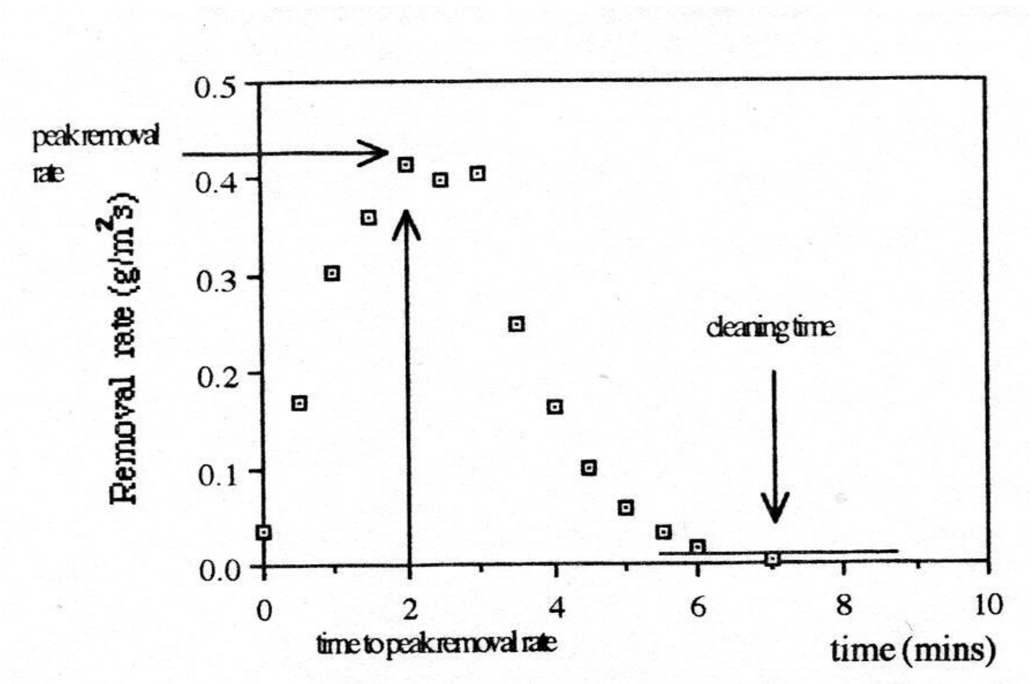
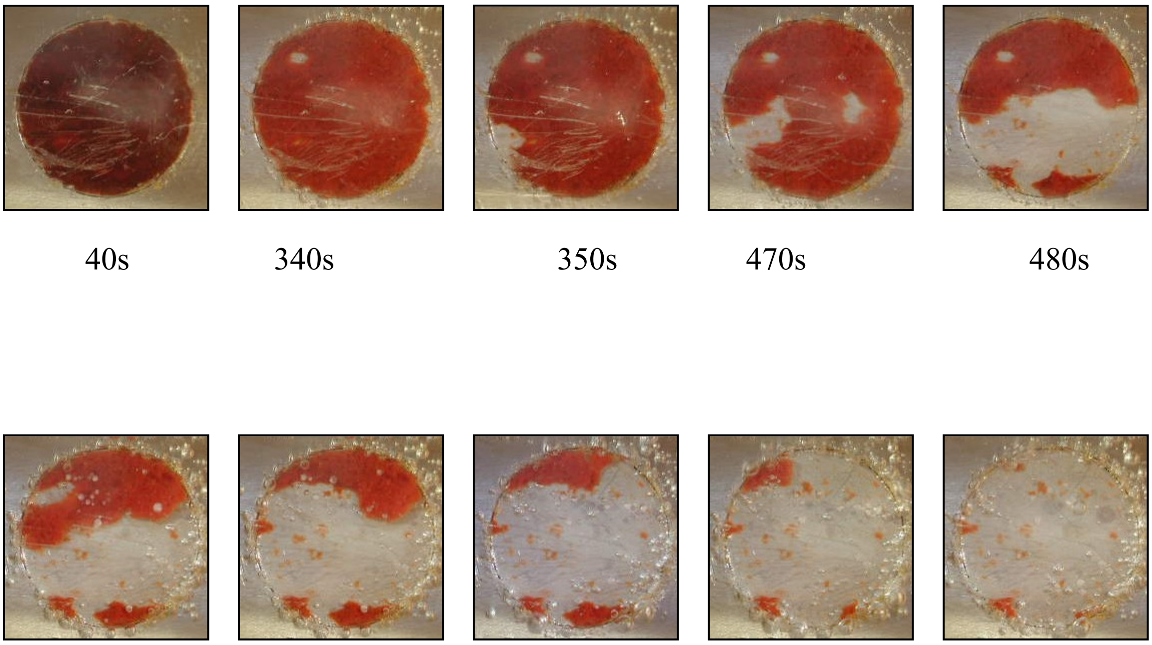


Figure 2.2: Typical cleaning rate curves from whey protein deposit removal (Bird and Fryer, 1991).

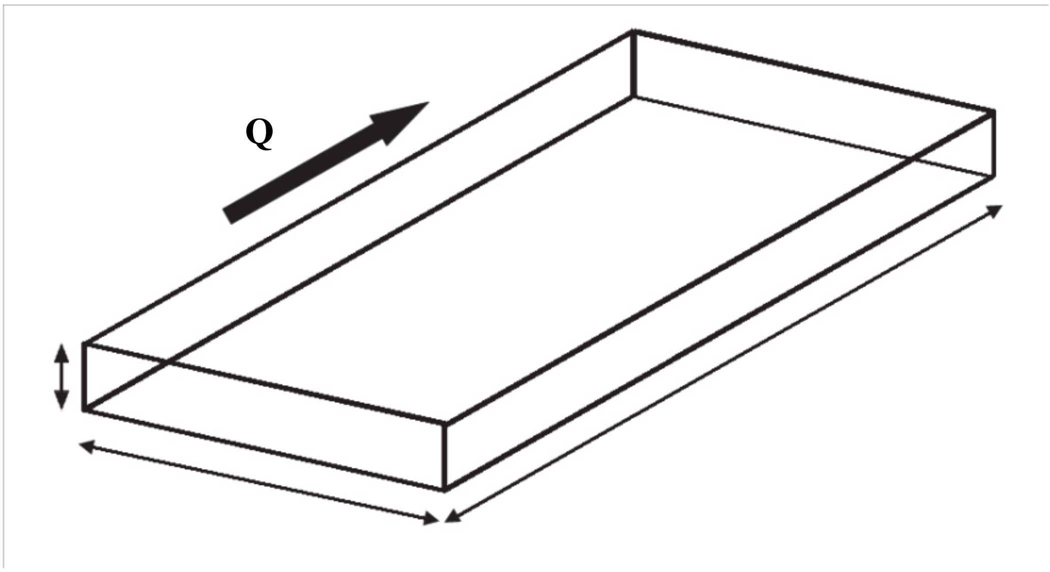


40s 340s 350s 470s 480s

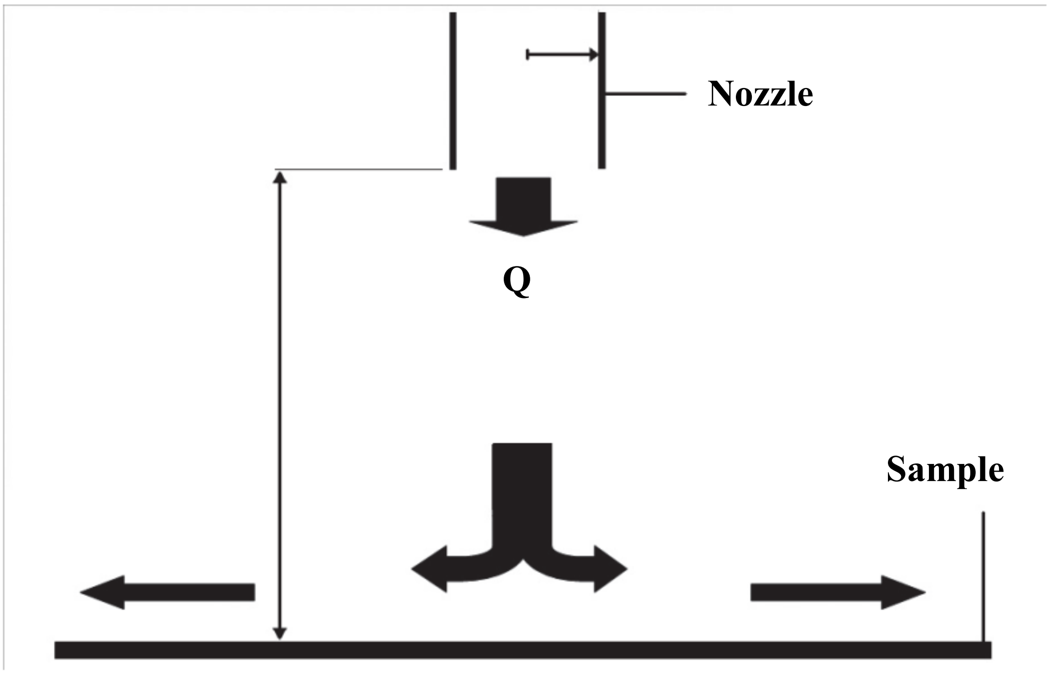
640s 650s 890s 1160s 1270s

Figure 2.3: Step removal of the baked tomato deposit. The images recorded during the experiment at 50 °C, 1.5 l min^{-1} (Christian, 2003).

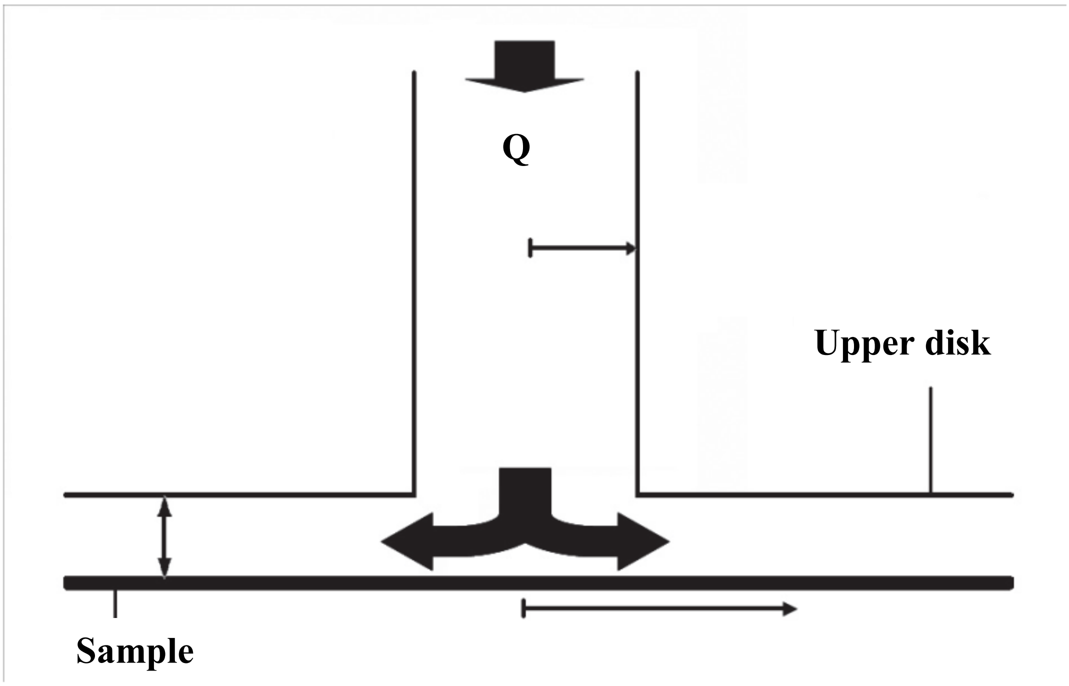
a)



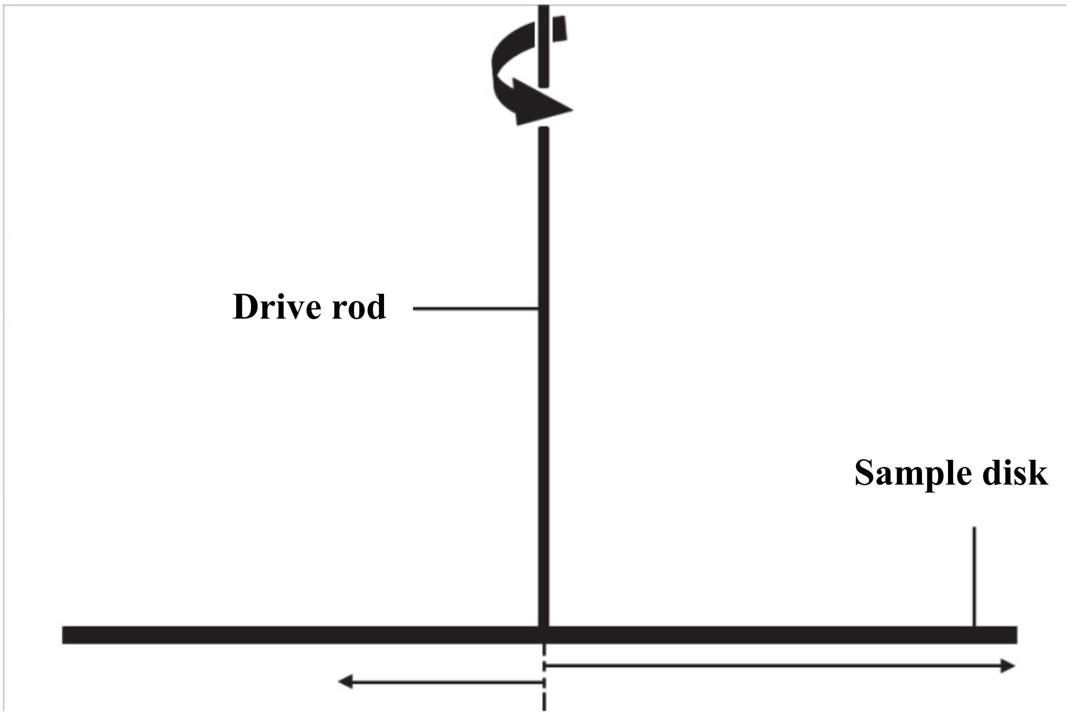
b)



c)



d)



e)

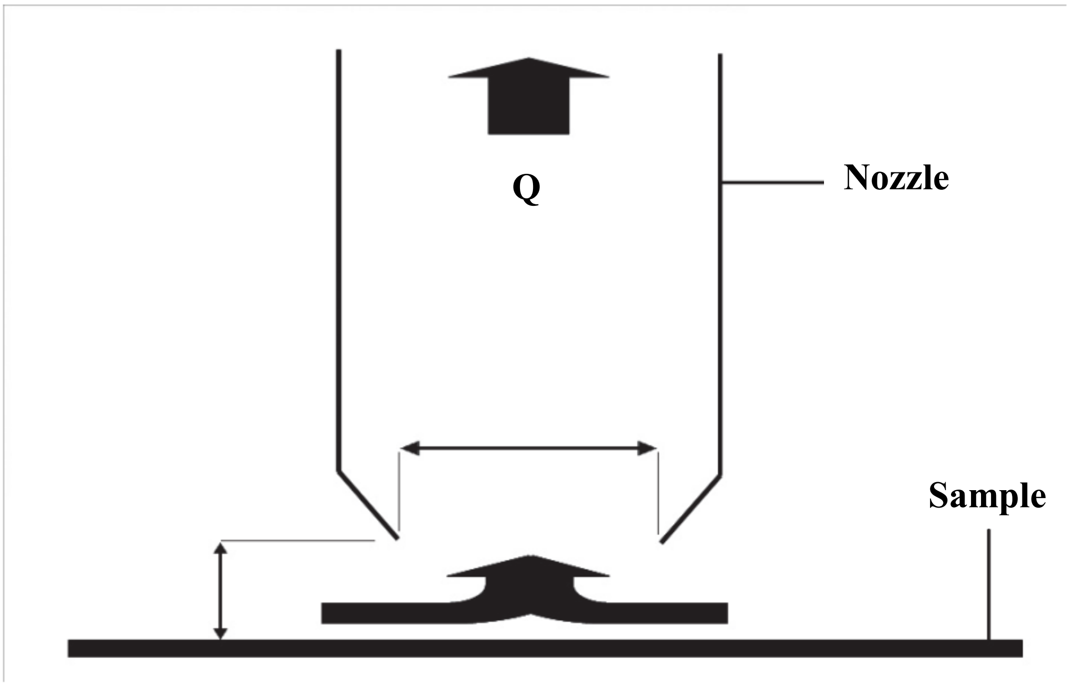


Figure 2.4: Scheme of some hydrodynamic systems. a) The flow channel in the parallel plate flow cell, b) the impinging jet, c) the radial-flow cell, d) a rotating disk apparatus, e) the fluid dynamic gauging. Q represents the flow direction of the cleaning fluid.

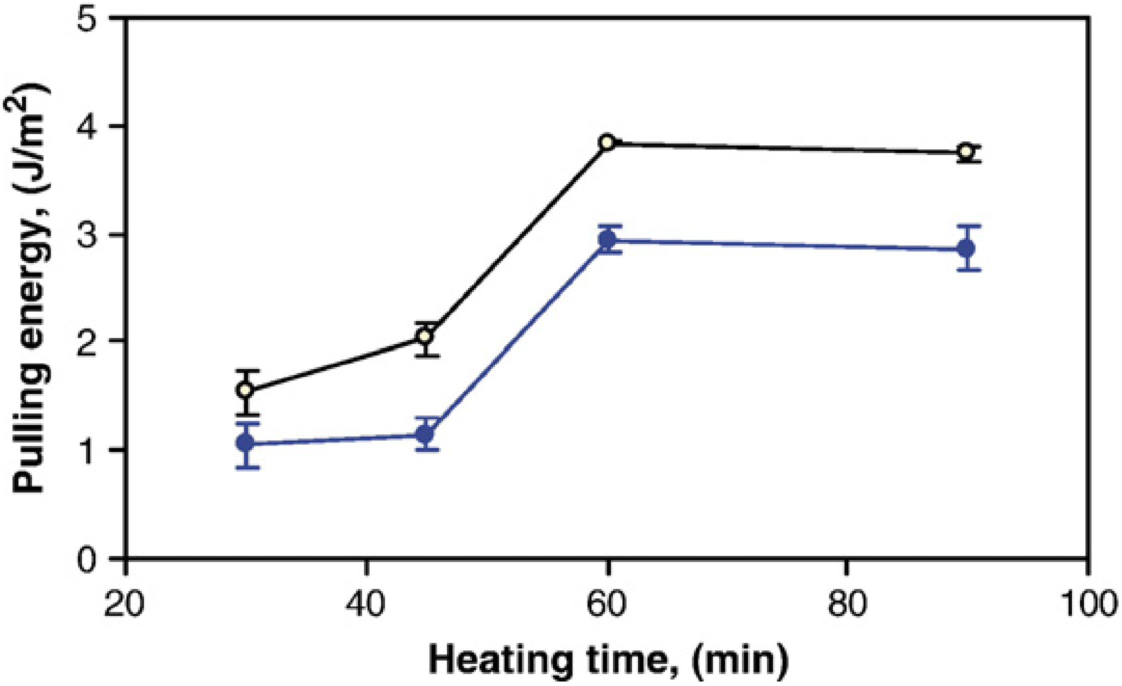


Figure 2.5 Pulling energy required for partial removal (open circles) and total removal (dark circles) of deposits of egg albumin, showing that the energy for partial removal exceeds that for total removal. The initial thickness of the deposit layer was approximately 2.2 mm (Liu et al., 2006).

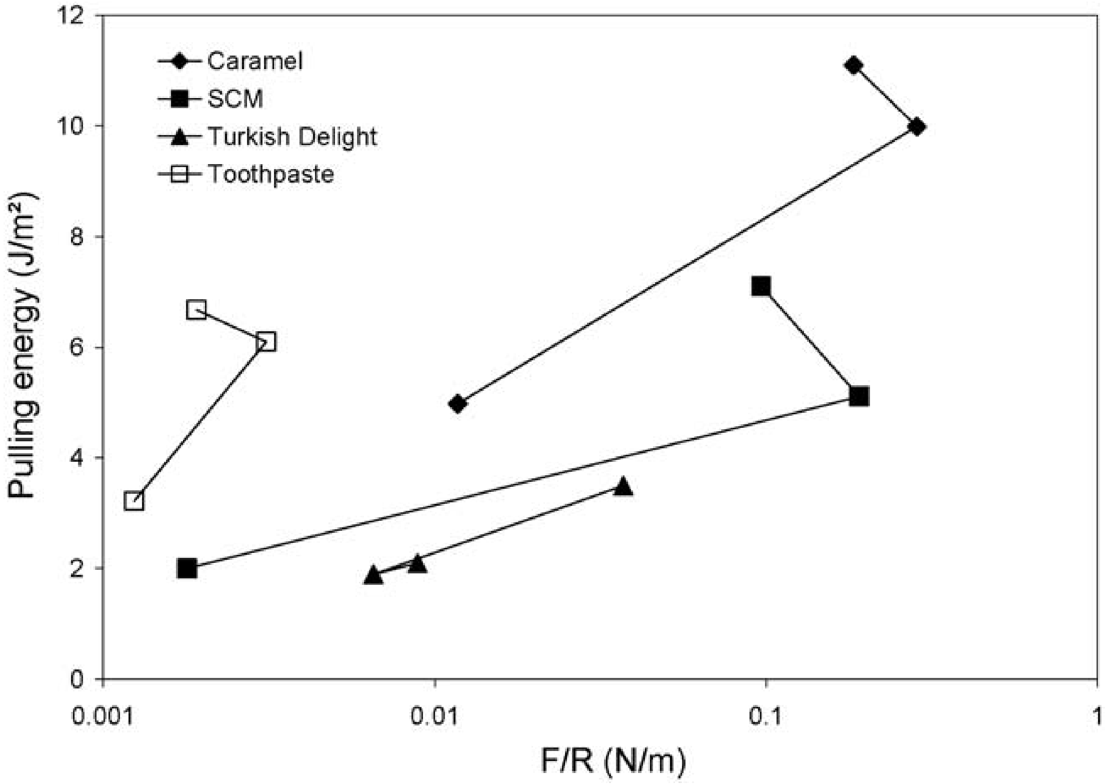


Figure 2.6 Comparison of data from micromanipulation and AFM, plotted for each deposit type (Akhtar et al., 2010).

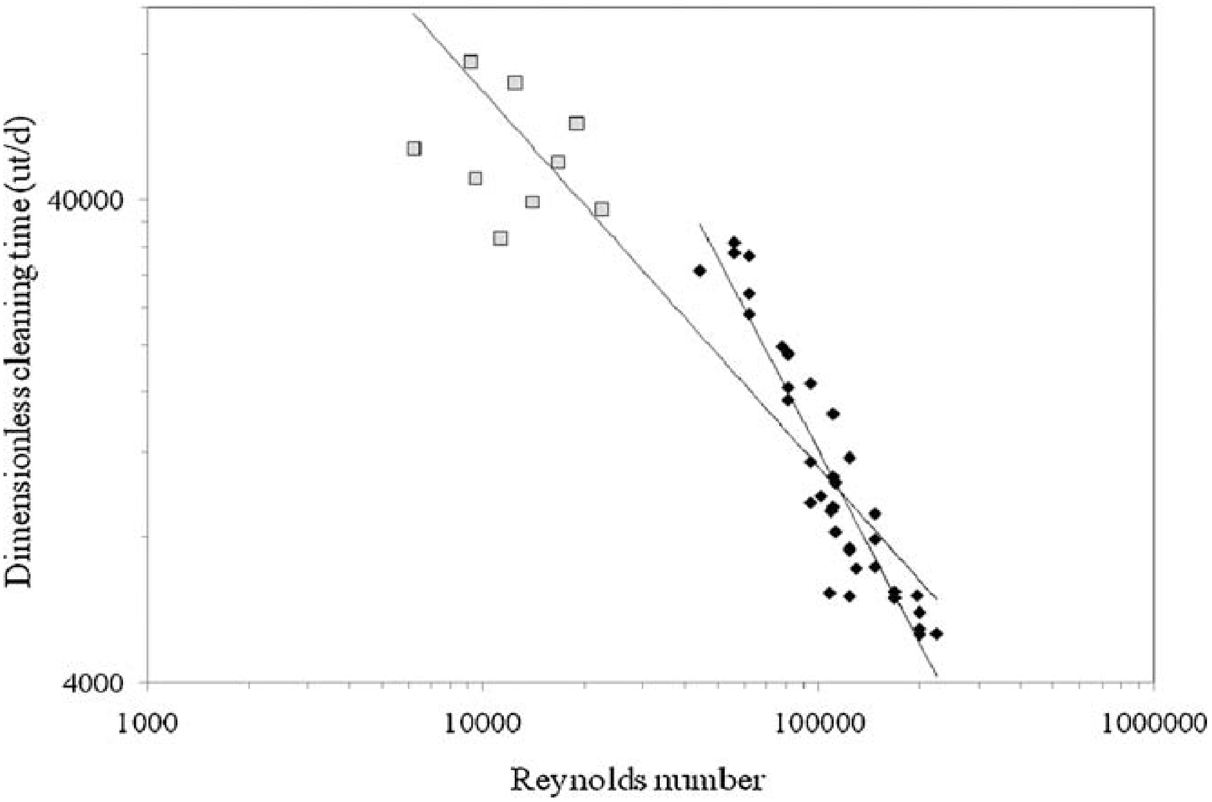


Figure 2.7: Dimensionless cleaning time as a function of Re for pilot plant (filled symbols) and coupon rig (open symbols) data. A power law fit of all data gives a function of $Re^{-0.778}$, $R^2 = 0.8426$, pilot plant data alone gives a function of $Re^{-1.31}$, $R^2 = 0.8715$ (Cole et al., 2010).

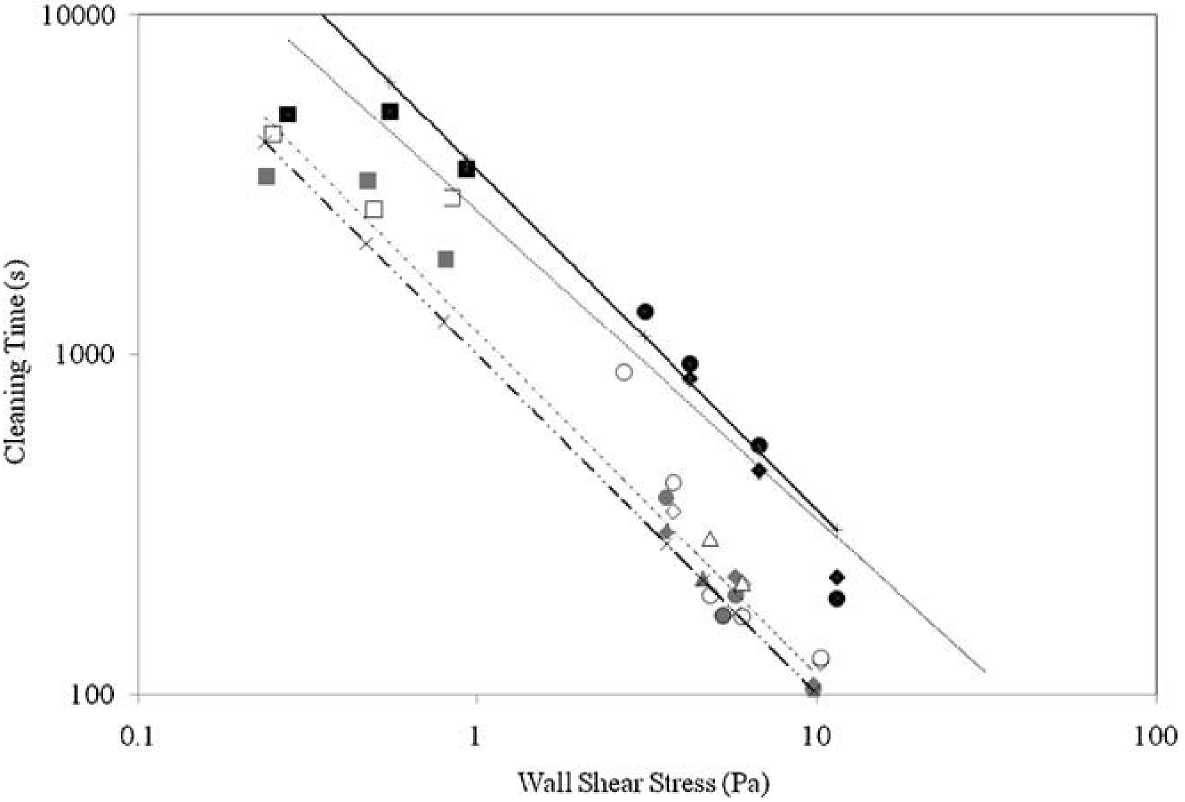


Figure 2.8: Cleaning time against wall shear stress for the coupon rig (squares) and the pilot plant (0.3 m test section = diamonds, 1 m= circles, 2 m = triangles) at different temperatures (20 °C; black, 40 °C; white, 50 °C; gray). Lines are: at 20 °C = grey line is the best fit, black line is fit to inverse wall shear (τ^{-1}), at 50 °C = grey dashed line is the best fit, black dashed line is fit to inverse wall shear (τ^{-1}) (Cole et al., 2010).

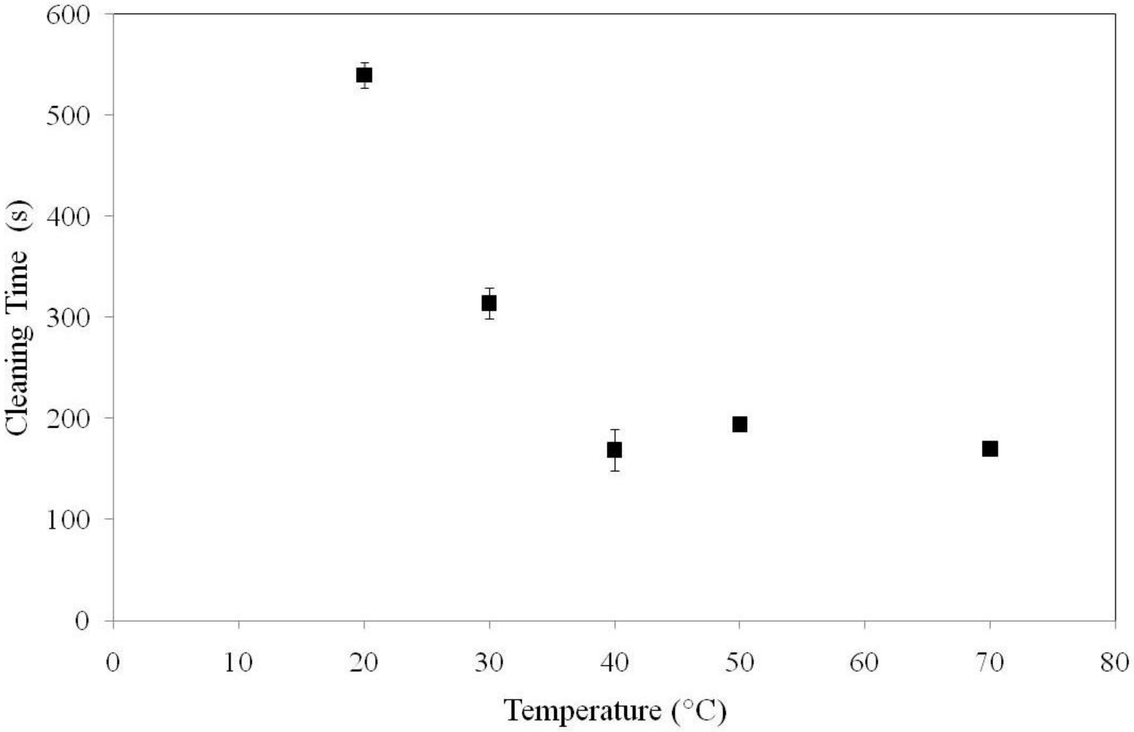


Figure 2.9: Cleaning time as a function of temperature. The test section was 1 m pipe with 47.7 mm ID. The cleaning fluid velocity was 1.7 ms^{-1} based on a clean tube (Cole et al., 2010).

Chapter 3: MATERIAL AND METHODS

3.1 Introduction

The aim of this chapter is to give details of the apparatus and measurement techniques used to gain data presented throughout the remainder of this thesis. Chapter 1 has highlighted the importance of understanding of the overall cleaning process to minimise environmental and economic impact of cleaning. However, the lack of understanding of the cleaning process is the major problem in cleaning research (Fryer and Asteriadou, 2009).

Chapter 2 has suggested that studying cleaning of simple deposits which can be removed by fluid action alone would help better understanding of the cleaning process as it only involves fluid mechanical removal. Using complex deposits will be problematic because production of consistent fouled deposits is generally difficult (Changani et al., 1997). Rheology of simple deposits can be used to indicate cleaning behavior (Akhtar, 2010). Moreover, the review of the literature has highlighted that choosing an appropriate hydrodynamic system and a monitoring method are both important to clarify the cleaning process in detail. Measuring the removal rate during the cleaning process is crucial to identify the controlling parameters.

Therefore, cleaning of simple deposits (which can be removed by water alone) from circular straight pipes is studied throughout the thesis and the cleaning process is monitored by the weight analysis method and the Optek turbidity meter. As mentioned in Chapter 2, if deposits can be removed by fluid action alone, this is defined as the fluid mechanical removal.

In Chapter 4 and 5, the cleaning of toothpaste from pipes is studied in detail to identify cleaning stages of simple deposits thoroughly to find out the possibility of any cleaning optimisation. In Chapter 6, the cleaning of various simple deposits from pipes is studied to study the effect of deposit rheology on the cleaning of simple deposits. In Chapter 7, the cleaning of toothpaste and hand cream from pipes at different scales is studied to quantify the flow effect on cleaning and to study scale up in the cleaning process.

This chapter contains the following sections to give background understanding of the experimental setups and methods used to obtain the results presented in Chapter 4, 5, 6 and 7:

i) the lab scale rig and operation
ii) the pilot scale rig and operation
iii) rheology of materials
iv) the monitoring methods, the weight analysis method, the Optek turbidity meter, the comparison between the turbidity and the weight measurement and the experimental procedures
v) the conclusions

3.2 Lab scale cleaning rig

The lab scale experimental rig was previously developed to monitor the cleaning process by visual analysis and thermal measurement at the University of Birmingham (Cole, 2011, Goode, 2011). However, they used a horizontal duct in this rig to study cleaning on planar surfaces. As mentioned in section 2.4, a circular pipe geometry was chosen in this thesis to study cleaning, hence, the horizontal duct test section was substituted by a 1 m section of 0.0239 m diameter pipe in this rig. This pipe was connected by triclamps into the place where the duct section was fitted in these previous studies.

A schematic of the lab scale experimental rig is shown in Figure 3.1. The details and the operation of the rig were given in these previous studies. The rig comprises a water tank, a flow transmitter PD 340, a conductivity and temperature meter at the return (LMIT08), a heating tank and a centrifugal pump (Alfa Laval, Denmark) is used to supply water of desired velocity to the system. All parts are connected with plastic hoses with the same diameter as the test section.

This rig was used in two configurations; with the heater (in Chapters 4 and 5) and without the heater (in Chapters 6 and 7). When the heater is connected to the rig (as shown in Fig. 3.1 (a)), the achievable velocity range is much smaller than those generated in pilot scale experiments due to high pressure drop in the heater. To achieve a similar velocity ranges to the pilot scale experiments, the heater could be disconnected from the rig and the flow meter directly connected to the valve at the inlet of the test section using plastic hose (as shown in Fig. 3.1 (b)). Reynolds Number (Re) and surface shear stresses (τ_w) in the system (Blasius equation) were estimated by the following expressions (Coulson et al., 1999):

$$\mathrm{Re} = \frac{Dv\rho}{\mu} \tag{3.1}$$

$$\tau_{\omega} = 0.079\,\mathrm{Re}^{-0.25} \cdot \frac{1}{2}\rho v^2 \tag{3.2}$$

where D is pipe diameter (m), v is average flow velocity (m s^{-1}), ρ is density (kg m^{-3}) and these terms are defined using the physical properties of cleaning water and the clean pipe diameter – these stresses are the lower bound of those in a partly-filled pipe. Table 3.1 shows the achievable velocity, Reynolds number and wall shear stress ranges in the pilot and lab scale (with heater and without heater) rigs over the temperature range of 20 – 70 °C.

As can be seen from Figure 3.1, there is a valve at the beginning of the test section, therefore, steady flow could not be achieved in the lab scale experiments due to space limitations. Nevertheless, a set of experiments were done to study the effect of inlet length on cleaning. Figure 3.2 shows the three different inlet length studied. Cleaning is not stopped until the pipe is completely clean, which is monitored visually by using the discontinuous method used in the weight analysis method (procedures will be explained in section 3.5.1). Similar cleaning times were found in all cases (760 ± 40 s). This result suggests that the test section is long enough that the cleaning of full length of the test section is not affected by the flow disturbances caused by the fittings in the inlet. Therefore, in all the experiments conducted in this thesis, cleaning time of the full length of the pipe is described.

3.3 Pilot scale cleaning rig

A schematic representation and pictures of the pilot plant are shown in Figure 3.3. It has been used in the previous work and is described in detail by Cole et al. (2011). The plant comprises water tanks, a centrifugal pump (3 bar, 5.5 kW) capable of transferring up to 20 m^3 h (Alfa Laval, Denmark), a valve assembly, a steam heated plate heat exchanger (GEA, Germany), and various measurement probes including flow (Promag 51P, Endress-Hauser), conductivity and temperature (LMIT 08, Ecolab) and turbidity (Kemtrak TC007, Kemtrak ab; Optek TF16, Optek-DanulatGmbH). The test sections used were 0.3 and 0.5 m stainless steel pipe with an internal diameter (ID) of 0.0477 m (1.6 mm wall thickness). Flow velocities and temperatures used were in the range of 1 – 2.5 m s^{-1} and 20 to 70 °C respectively. Only the Optek turbidity meter was used to monitor the extent of cleaning and to compare cleaning times of deposits. Cole (2011) did extensive work to identify which measurement(s) give the most accurate and repeatable evaluation of the cleaning time and found that the Optek turbidity meter was the most sensitive online measurement probe on the ZEAL Pilot Plant.

The plant was designed to be flexible and allow the study of different geometry plant items, which could be arranged as required into different test sections (for example in Fig. 3.3 (b and c)).

The pilot plant was operated using Matlab R2007b by selecting the directory: C:\Matlab Files\zeal_pilot and selecting the required route by typing run_zeal. Data from the pilot plant is collected by a laptop (Toshiba Satellite Pro L40, model: PSL4BE – 00S00WEN) connected to the signalling panel (GEA Tuchenhagen). Data is collected from the on-line measurements at frequent intervals during the experimental run (typically every second).

Before any cleaning experiment started, the test pipe had been filled with the study material. The filling procedure of pipes was all the same for the lab and pilot scale experiments. The pipe was manually filled with the study material by capping the base of the pipe and transferring the material to a funnel positioned above the pipe. Then, the pipe was tapped against a piece of cloth to allow any air gaps to be filled. The filled pipe was then tri-clamped into the test section area in a horizontal position.

Recirculation of rinsing water was not used in the experiments in this thesis, meaning that continually refreshed water was used throughout the experiment. Within one experiment there might be multiple runs, as there was a limited amount of water in the tank (0.5 m^3). Each run used the same quantity of water. At the end of each run, the tank was refilled and a further run could then take place if the system was not clean. Unless stated, flow was allowed to develop for at least 30 pipe diameters before reaching the test section. The velocity was accurate to ± 0.06 m s^{-1}, in the standard 0.0477 m ID diameter system. The cleaning water was heated to the designated value prior to the water being run through the system, with the exception of the ambient water which ranged from ~8 °C in the winter to ~19 °C in the summer.

3.4 Materials and rheology studies

To investigate the effect of deposit rheology on cleaning in Chapter 6, various types of deposits that could be removed by water were chosen with different rheologies: Newtonian, Herschel-Bulkley and shear thinning behaviour. Eight materials were investigated.

i) Toothpaste was supplied by GSK (UK). Toothpaste is a formulated product, which contains several types of key ingredients to give it function and form. Toothpaste must be able to retain its structure and not separate or lose efficacy over time. Toothpastes as described by Joiner (2007) are commonly a mixture of: *Gums (60 wt %)* which are used as gelling agents to give thickness to the structure, and humectants to provide wetting in the mouth and sweetness. These gums include sorbitol, glycerine and xanthan gum. *Surfactants/ Detergent systems (2 wt %)* to provide a foam structure and promote saliva, for instance, sodium lauryl sulphate. *Silicas (25 wt %)* which provide body to the paste, and abrasivity used to polish the teeth *Flavour (2 wt %)* for taste. *Dyes (2 wt %)* for colour. *Flouride source (1 wt %)* to allow the teeth to regain enamel strength and *water (8 wt %).*

ii) Toothpaste was also diluted with water to generate different soils with different yield stress values. This was done by mixing a prescribed amount of water and toothpaste manually for at least 5 minutes. During mixing the diluted toothpaste, foaming was found, usually when more than 20 wt % of water was used in the mixture. Foamed samples were left for at least 1 day before being used in any experiment to decrease any error caused by the air bubbles. Hand cream was also tested at 70 °C to give a material with a reduced yield stress.

iii) Handcream was supplied by GSK (UK). It is an emulsion of water (70 wt %) and certain fats (20 wt %). The emulsion is of a "water in oil" type unlike the "oil in water" type emulsion of vanishing cream, so-called because it seems to disappear when applied on skin. It is composed of small droplets of oil dispersed in a continuous phase so it is less greasy and more easily washed off using water.

iv) Shampoo and shower gel were supplied from Unilever (UK).

v) Glucose syrup was supplied by Cadbury (UK). It was a type of confectioner's syrup that contains 19% glucose, 14% maltose, 11% maltotriose and 56% higher molecular mass carbohydrates.

vi) Apple sauce (Bramwells) and yoghurt (Broklea, low fat Greek Style) were purchased from the local market, ALDI.

The rheological behaviour of each material was obtained using a TA AR1000 rheometer (TA Instruments, UK). A shear rate ramp was done to find the viscosity and rheological behaviour of the materials.

Experimental procedure for rheology tests: 40 mm diameter, stainless steel parallel plate geometry was used in the rheology studies as investigated materials generally contain 0.5 μm diameter particles, which could have got caught under the tip of the cone in a cone–and-plate system. Toothpaste was placed on the static plate after mapping the geometry and setting the zero gap. Stainless steel plates were used as they are robust and compatible with the samples at all the temperatures of interest. The geometry was lowered to 250 μm gap distance between the parallel plates using the computer controls. The materials generally squeezed out slightly but not so much that it rose up onto the upper plate. Then, the moisture trap was placed over the geometry and sample and left to equilibrate for up to 3 minutes before testing.

In the literature, different methods have been used to find yield stress of a material which may produce conflicting results depending on the techniques employed and on the criteria on which the yield stress was based (Astarita, 1990, Hartnett and Hu, 1989). Here, oscillatory stress sweeps were used to determine the visco-elastic nature and the yield stress of each deposit. If the elastic modulus is higher than the viscous modulus (G'>G") over a particular range of shear stress, the material shows elastic behaviour and it does not flow in this shear stress range. If the elastic modulus is lower than the viscous modulus (G'<G"), the material shows viscous behaviour and it flows in the increased shear stress range. Therefore, crossover of these parameters (G'=G") was used as a yield stress value. Oscillatory stress sweep tests were done at an angular frequency of 6.283 rad s^{-1} (1 Hz) and at 20 °C which was generally the rinsing water temperature used in experiments. This measurement was convenient, easy to detect and gave repeatable yield stress results allowing easy comparison between different deposits.

Figure 3.4 (a and b) shows the rheological behaviour of each material: Hand cream, toothpaste as supplied (100 %), yoghurt, apple sauce, shower gel, glucose and shampoo at 20 °C. Hand cream tested at 70 °C is also shown. Figure 3.4 (c) shows typical yield stress determination for 80 % diluted toothpaste at 20 °C and hand cream at 70 °C. All materials were not shown because the graph becomes unclear with all points plotted. The yield stress determination of the rest of the materials can be found in Appendix A. Table 6.1 in Chapter 6 shows the rheological type of materials, the yield stress values and viscosity (at 15 s^{-1}) of all materials studied.

Figure 3.4 (a) illustrates that most of the studied materials show an inherent shear stress at near zero shear rate and their viscosity decreases with the applied shear rate in Fig. 3.4 (b), demonstrating that they are Herschel-Bulkley type of materials. Only Glucose sample shows Newtonian behaviour as its viscosity stays constant with the increasing shear rate in Fig. 3.4 (b). Shampoo shear thins with the applied shear rate and it has no yield stress value. Rheological and cleaning behaviours of those Newtonian, shear thinning and Herschel-Bulkley type of fluids and diluted toothpaste at different temperatures will be further studied in Chapter 6.

3.5 The monitoring methods

The weight analysis method and the Optek turbidity meter were used to monitor the cleaning process in pipes.

3.5.1 The weight analysis method

MHFS and turbidity probe were used in the previous studies (Christian, 2003, Cole, 2011) to monitor the cleaning process. However, as discussed in section 2.1, these cleaning monitors can only detect the extent of cleaning and could not give any detail about how much deposit is removed at a particular time interval. Therefore, any limiting cleaning stage or underlying processes could not be identified in detail by these probes. Hence, a simple experimental method is developed to monitor the cleaning process inside the pipe. This method is comprised of a sequence of dismantling-draining-weighing-placing procedures of the pipe until the end of the cleaning process. Therefore, weight data of the study material in the pipe can be found as a function of time during the cleaning process.

This method will allow the determination of how much material is removed when, hence, limiting stages of cleaning can be identified by the cleaning rate data (discussed in Chapter 4 and 5). Moreover, it will be easier to compare cleaning behaviours of different deposits whose cleaning rate data is already available in the literature (discussed in Chapter 5). Also, underlying processes can be identified in the cleaning by comparing the cleaning rate data for the experiments at different length scales (will be discussed in Chapter 5 and 7). These are some of the advantageous of the method, however, there are some drawbacks as well.

The main drawback of this method is that it requires tough work; the experiment must be stopped, as short a possible time taken to dismantle, drain, weigh and place the pipe to the rig, then start the experiment. The series of actions must be repeated generally 10 times until the end of cleaning to get analysable weight vs time data. Also, the ability of the method is compounded by the weight of the test sections which varies between 1000 and 5000 ± 0.5 gr.

Another disadvantage of this method is that the experimental error will increase as the number of weighing events increases. Errors may result from various factors; the change in the state of deposit during draining and weighing the pipe, weighing inaccuracies, soaking of water into deposits, a kind of pulsing effect by stopping and initiating the flow after placing the test section. However, the aim of using this weight analysis method is to monitor the cleaning rate (not to find an absolutely accurate cleaning time) in this thesis except in Chapter 7. In Chapter

7, in order to decrease arising errors, weighing frequency is decreased as much as possible to detect cleaning end point as accurately as possible which will be discussed in section 3.5.4.

Repeatability of the weight analysis method

Figure 3.5 shows the repeatability and validity of the experimental method by considering possible factors that may affect measurements. Data is plotted on both linear (Fig. 3.5 (a)) and log scale (Fig. 3.5 (b)) to show the shapes of the cleaning curves.

Figure 3.5 (a) and (b) shows 4 runs on the lab scale cleaning rig to assess the effect of flow disturbances resulting from starting and stopping the flow to take a weight measurement, weighing frequencies and soaking on the repeatability of the experiments:

- In runs 1 and 2, the effect of the frequency of sampling was studied by weighing the pipes at different time intervals until 10 g of toothpaste was left on the wall. The figure shows good agreement between the two runs and data points formed a single line at log scale to an R^2 of 0.97.
- In the 3rd run, the experiment was not stopped until after 600 s to evaluate the effect of sampling frequency on the cleaning; the experiment gives a similar result to the initial 2 runs, with the point at 600 s within ± 2 g of the first two runs, which suggests that the pulsing effect by stopping and starting the flow is small.
- In run 4, sampling was carried out every 5 minutes; there is some difference in the first 300 s, but the last cleaning stages are basically identical to run 3. The repeatability of the experiment is presumably affected by the thin film structure which was obtained after the product recovery stage. This will be discussed in more detail in section 4.2.3.

Figure 3.5 (c) shows the comparison of two experiments at the same weighing frequency to examine the effect of cleaning conditions on the repeatability of the experimental method. At higher cleaning temperatures and velocities (here 50 °C and 0.55 m s^{-1}) there is good agreement between three experiments – however at lower velocity and temperature (30 °C and 0.38 m s^{-1}) there is some scatter, although the trends in the data are the same. This is expected when the simplicity of the experimental method is considered. Nevertheless, similar removal trends were observed in all different experiments which showed that cleaning rate of the material was not affected by the experimental method and weighing frequencies. Thus, by this method, removal rate can be monitored from the beginning which can allow to compare the cleaning of different materials and different scale pipes directly. This is critical to understand cleaning process in great detail.

Experimental procedures for the weight analysis method

The weight analysis method is mainly developed to identify cleaning behaviours and stages to understand the cleaning process as it is not possible to identify the cleaning time with complete accuracy with this method due to low accuracy of the weight measurements (± 0.5 g). The error in the measurement is thus negligible when the weight of material in the pipe is on the order of 100 g but increases as the amount of toothpaste decreases. The 'end-point' of cleaning is defined as when the mass fraction remaining is 0.002 as shown in Fig. 3.6 (a); this results in an error of ca. 60 s in the determination of total cleaning time due to low accuracy of the weight measurements. The errors in time are on the order ± 1 s, resulting from the lag time between seeing the stopwatch and closing the valve or stopping the Matlab program.

The experimental procedure was as follows at lab scale:

- the pipe was first filled manually with the material and placed into the rig;
- water was pumped from the water tank through the bypass loop to ensure the flow (and temperature) stability;
- water flow was then diverted to the test section; the valve was closed manually; immediately after seeing water at the outlet of the test section, i.e. at the end of the product recovery (core removal) phase;
- the test section was then removed from the rig and water drained carefully;
- after weighing and recording the mass, pictures of the outlet end of the test section were taken where needed, the test section was placed in the rig and cleaning performed using water potentially at a different temperature and flow velocity. The same dismantling and weighing procedure was applied at intervals until a negligible amount of toothpaste (≤ 0.5 g) was left on the pipe wall;
- experiments were repeated at least in duplicate.

For pilot scale experiments, the same procedure is applied. However, the by-pass loop was not used as it was dangerous to close and open manual valves while the pump was working in this scale. Only for the product recovery stage, the pump was operated for only 3 s to do product recovery (core removal) and then the pump was stopped to remove and weigh the test section. Only two different flow rates (6.5 m^3/h and 17 m^3/h) were used in this stage due to pump limitations.

3.5.2 The turbidity probe (Optek)

Turbidity is a measure of the light absorbed or scattered by a liquid from a known light source. The Optek turbidity meter detects light scattered from particles (trace suspended solids, undissolved liquids or gas bubbles) in the liquid at an angle of 11°. It gives ppm response as cleaning continues. A typical cleaning graph is shown in Fig. 3.6 (b). It shows that the measurement is insensitive at the initial stages of cleaning, when the turbidity due to the toothpaste is significant. In this cleaning stage, it is saturated and gives a flat-line reading at turbidities higher than 50 ppm as the Optek turbidity (ppm) meter can only measure between 0-50 ppm. However, it provides greater detail at the end of the cleaning experiment. Also, turbidity meter allows determination of the extent of cleaning without disturbing the experiment, thus cleaning can be monitored more accurately than the weight analysis method. Moreover, the accuracy of measurement is given as ± 0.3 % by the company. The Optek turbidity probe was used in Chapters 5 and 6 at pilot scale to find cleaning times.

The cleaning procedure was simple when turbidity meter was used; after placing the filled test section into the rig, water was passed at the desired temperature and flow rate through the test piece for a set time (dependant on flow rate). The water passes were repeated until the turbidity meters read > 2 ppm.

As can be seen in Fig. 3.6 (b), unless stated a numerical reading of 4 ppm from the Optek turbidity meter was chosen to compare cleaning effectiveness of the experiments in this thesis. It is at the low end of the scale for this measurement and outside the area where electrical noise affects the signal (-2 to +2 ppm). The pipe was found having a few islands left, with 0.2 % of the starting weight remaining.

3.5.3 The comparison of turbidity measurement with the mass left in pipe

During some experiments in the pilot plant, ppm response of Optek turbidity probe was read and recorded while performing the weight analysis method to identify which ppm value should be chosen to serve as a comparative indicator of cleaning effectiveness. The experimental procedure was as follows:

- the pipe was first filled manually with toothpaste and placed into the rig;
- water at particular temperature and flow rate was pumped to the test section; after 60 s pumped was stopped for the 1st time; ppm response of the turbidity probe was recorded just before the pump was stopped;
- the test section was then removed from the rig and water drained carefully;
- after weighing and recording the mass inside the pipe, the test section was again placed in the rig and cleaning continued using water at the same temperature and flow velocity. The same ppm reading, dismantling and weighing procedures were applied at particular time intervals until a negligible amount of toothpaste (≤ 0.5 g) was left on the pipe wall;

Figure 3.7 graphically shows that;

- The turbidity meter response did not happen in the same way as the weight measurement. Depending on the experimental conditions, saturation of the turbidity meter was observed when different amounts of toothpaste was present in the pipe.
- Generally, the higher the temperature or flow rate, the less amount of toothpaste was left in the pipe when turbidity probe came off saturation.
- For instance, when the probe came of saturation, 150, 71 and 36 g toothpaste left in the pipe at 15 °C, 20 °C, 40 °C, respectively at 12 $m^3 h^{-1}$, and 190, 150, 114 g toothpaste left in the pipe at 10, 12 and 15 $m^3 h^{-1}$, respectively at 15 °C.

- This indicates that turbidity meter measures a kind of cleaning rate. At high temperatures and flow rates, removal of toothpaste from the pipe is rapid, therefore, the concentration of toothpaste is high in the effluent water. It causes the saturation of the turbidity probe although a little amount of toothpaste is left in the pipe.
- If any ppm value above 10 is chosen on the turbidity probe for comparison of cleaning effectiveness, it may produce deceptive results.
- A ppm value below 6 should be chosen to compare cleaning effectiveness safely as the similar amounts of toothpaste are left in the pipe (± 1 g) for all experiments when ppm response decreases below 6.

3.5.4 Cleaning time determination in Chapter 7

In Chapter 7, scale up was studied, therefore, the cleaning of lab and pilot scale pipes were investigated and compared. The turbidity meter (Optek) could not be used to determine the cleaning time at lab scale as this device could not fit to the lab scale rig. Therefore, the weight analysis method was used for both scales to determine the cleaning times. In order to minimise the experimental errors, dismantling and viewing the pipe were done at a low frequency until very tiny islands about half cm thick were left on the pipe. The turbidity meter in the pilot scale and inspection of effluent water in the lab scale allowed the estimation of the amount of material left in the test section without dismantling it from the rigs. The end-point of cleaning in chapter 7 was defined visually as the point when no material was left on the pipe wall (test section) to minimise the experimental errors at lab scale due to low accuracy of the weight measurements. In order to catch the end point as accurately as possible, the pipes were viewed at 30 s intervals at the later stages of cleaning. Therefore, the maximum experimental error of cleaning time was ± 30 s. Figure 3.8 illustrates this procedure to determine the exact cleaning time. For instance, the test section was not dismantled in the initial 510 s of the experiment and then it was viewed at 30 s intervals until nothing was seen in the pipe.

3.6 Conclusions

This chapter has introduced the monitoring methods and experimental procedures followed to obtain the results given in Chapters 4, 5, 6 and 7. As discussed in Chapter 2, the cleaning rate data is valuable to identify cleaning stages in detail. Therefore, the weight analysis method was developed to investigate the cleaning process thoroughly. It allowed to monitor how much material removed when from the pipe, therefore, the effect of process parameters during cleaning can be identified. It was shown that scale up could be studied by the available rigs at different length scales.

In Chapter 4, experiments are done to study the effects of process parameters at the core removal (product recovery) stage on the overall cleaning process. This might be useful in optimising cleaning by separating process conditions at the product recovery and cleaning stages.

Chapter 5 investigates the cleaning stages in detail by the weight analysis method to clarify how simple deposits are removed from pipes. This information might both help better understanding of the cleaning process in pipes and result in optimising the CIP process for the cleaning of simple deposits.

The experiments in Chapter 6 are conducted to study the effect of soil rheology on cleaning time and behaviour. This might be useful in categorising the simple soils according to their ease of cleanability by using rheology data. Moreover, the relation between cleaning time and rheology of a soil could be found.

In Chapter 7, cleaning experiments are done at the lab and pilot scale pipes to quantify the flow effect on the cleaning process and to study scale up. The aim is to develop further understanding of fluid mechanical removal in the cleaning process.

Tables Chapter 3

Table 3.1: Achievable ranges of fluid mechanical parameters in the used rigs.

Experimental Rigs	**Velocity (m s^{-1})**	**Reynolds number**	**Wall shear stress (Pa)**
The lab scale rig with the heater	0.2 - 0.6	4300 - 15160	0.18 – 1.68
The lab scale rig without the heater	0.46 - 2.65	10360 - 58720	0.86 – 17.9
The pilot scale rig	1 - 2.65	44970 - 102800	2.8 – 12

Figures Chapter 3

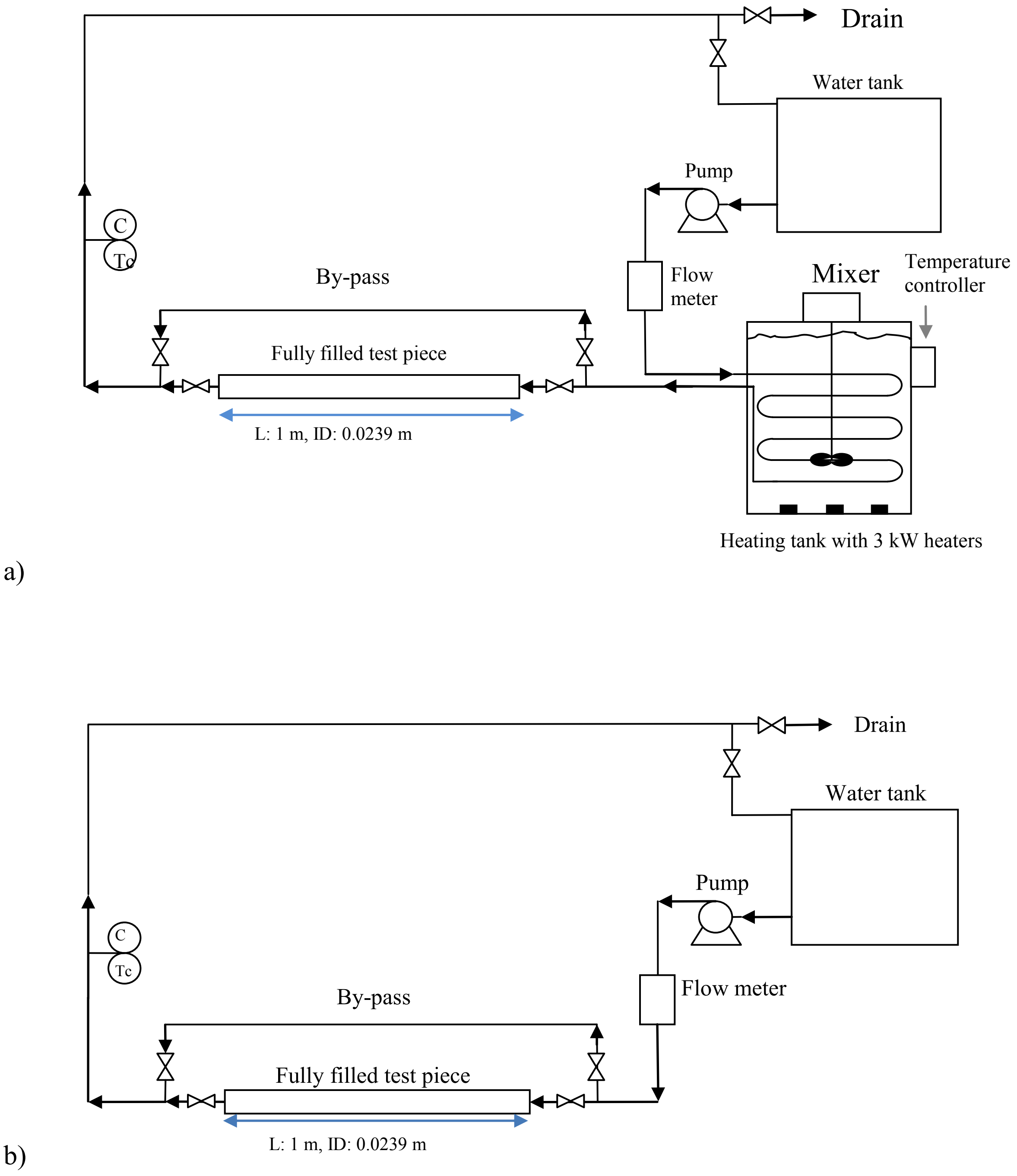


Figure 3.1: Schematic representation of the laboratory scale cleaning rig (a) with the heater and (b) without the heater.

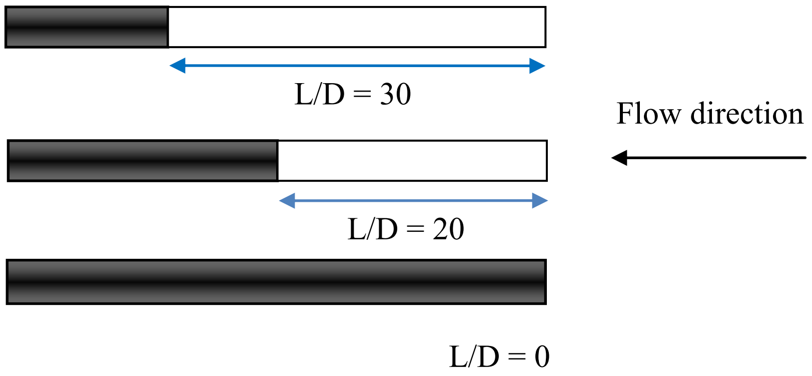
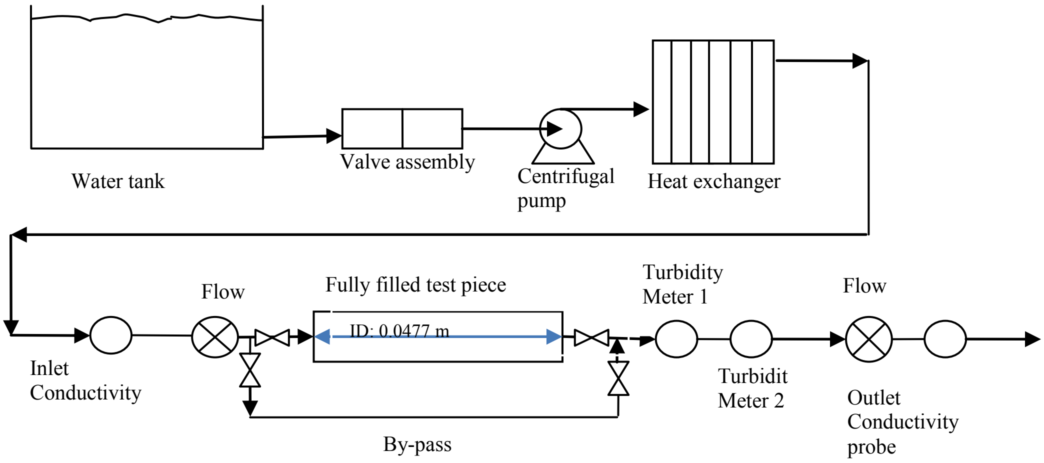


Figure 3.2: The studied inlet lengths. Black area represents the area filled with toothpaste. Cleaning water was used at 50 °C, 0.55 m s^{-1}. L is the inlet length and D is the diameter of the test section (0.0239 m). The full pipe length is 1 m.


Water tank
Valve assembly
Centrifugal pump
Heat exchanger
Flow
Fully filled test piece
ID: 0.0477 m
Turbidity Meter 1
Flow
Inlet Conductivity
Turbidit Meter 2
Outlet Conductivity probe
By-pass

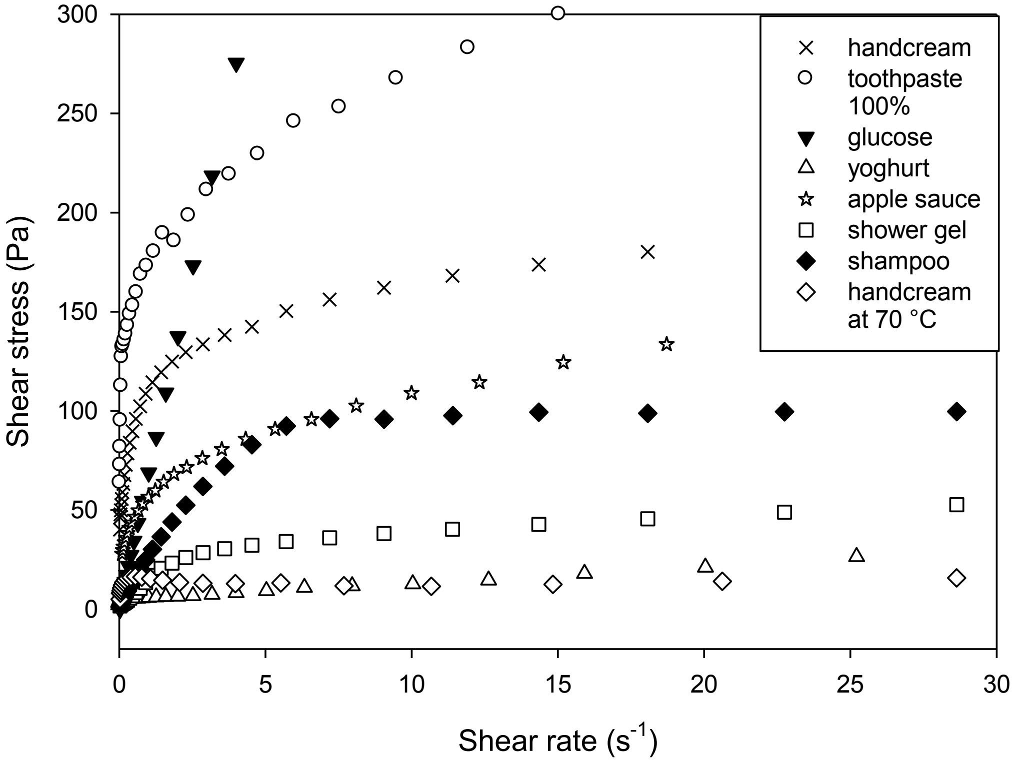
a)


Flow direction
Test section, ID:47.7 mm, L:0.5 m
TK 23
Caution Hot
TK 22
OPTEK turbidity meter

b)

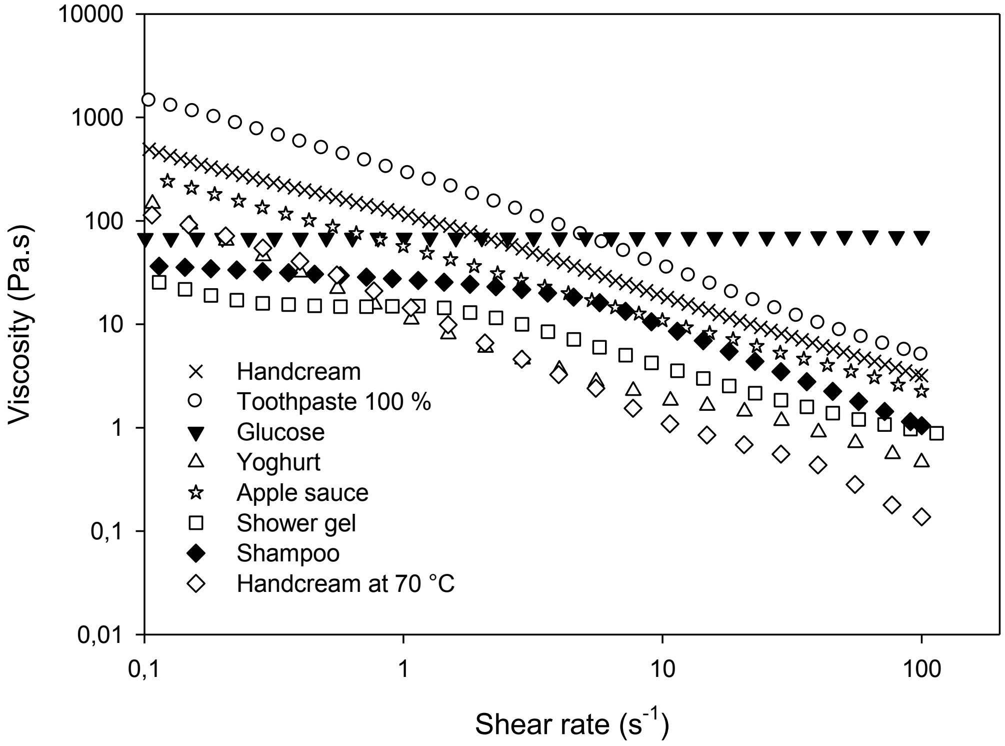
c) 

Figure 3.3: Schematic representation of the pilot plant (a). Typical picture of the pilot plant at different configurations (b and c). Turbidity meter 1: Optek (TF16, Optek-DanulatGmbH), Turbidity meter 2: Kemtrak (TC007, Kemtrak ab).



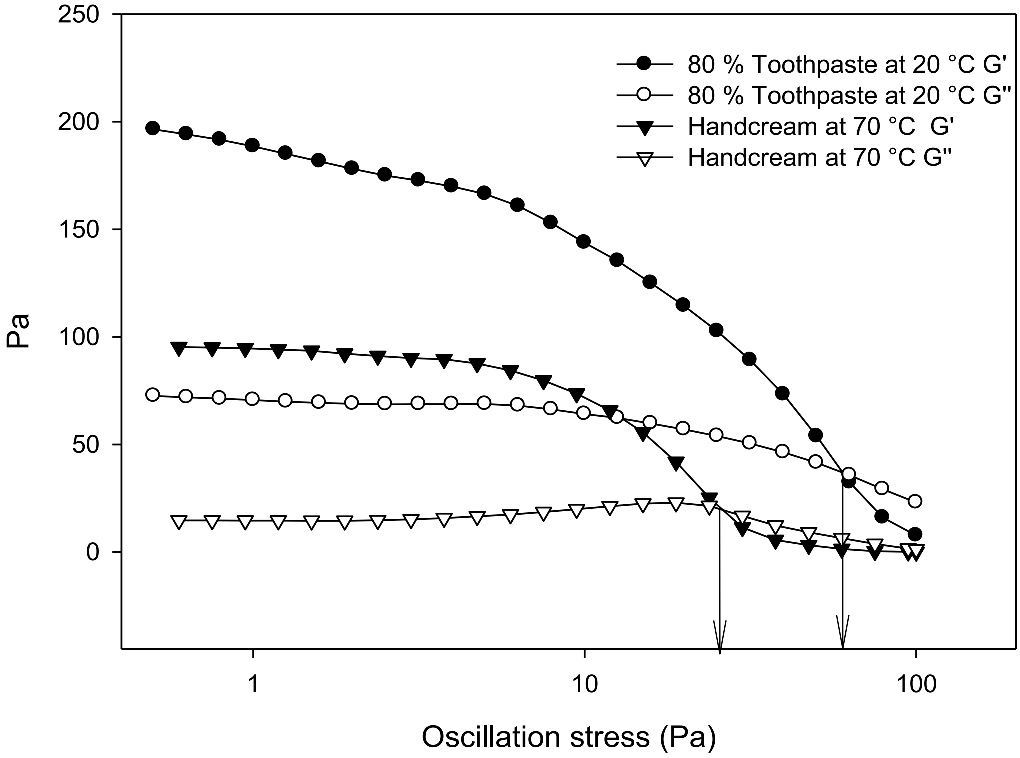
300
250
200
150
100
50
0
Shear stress (Pa)
0
5
10
15
20
25
30
Shear rate (s^{-1})
handcream
toothpaste 100%
glucose
yoghurt
apple sauce
shower gel
shampoo
handcream at 70 °C

a)



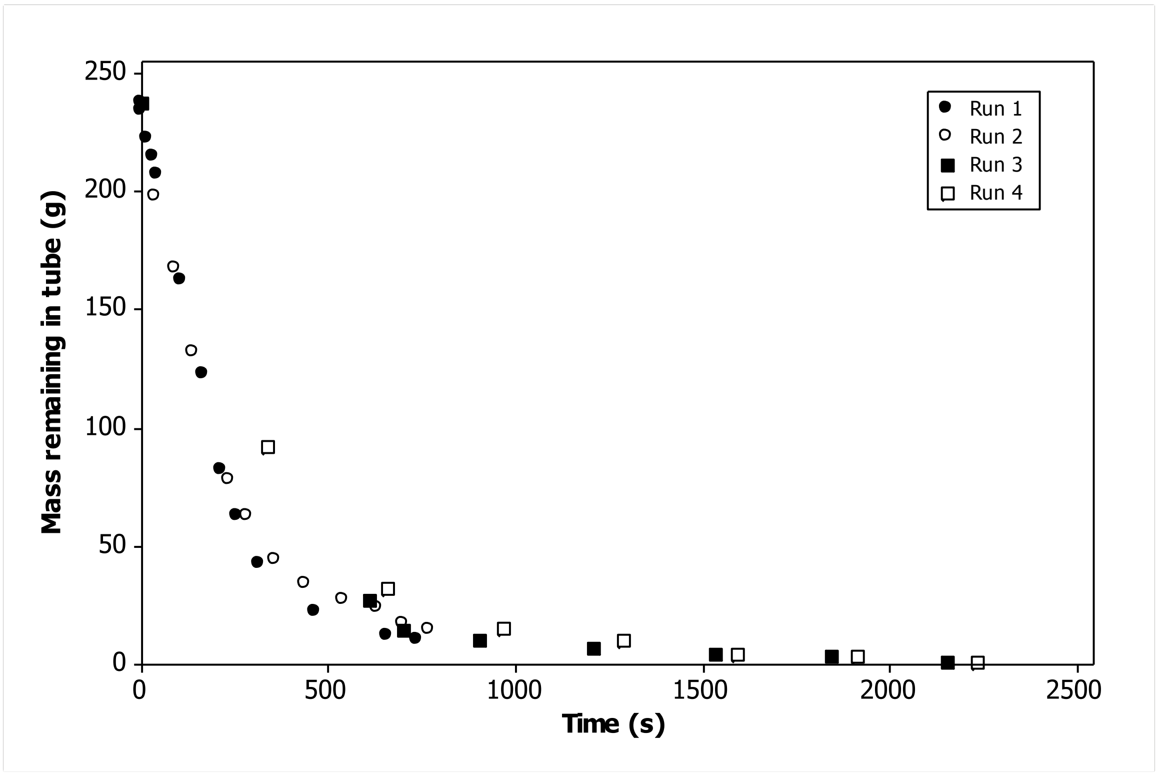
10000
1000
100
10
1
0,1
0,01
Viscosity (Pa.s)
0,1
1
10
100
Shear rate (s^{-1})
Handcream
Toothpaste 100 %
Glucose
Yoghurt
Apple sauce
Shower gel
Shampoo
Handcream at 70 °C

b)

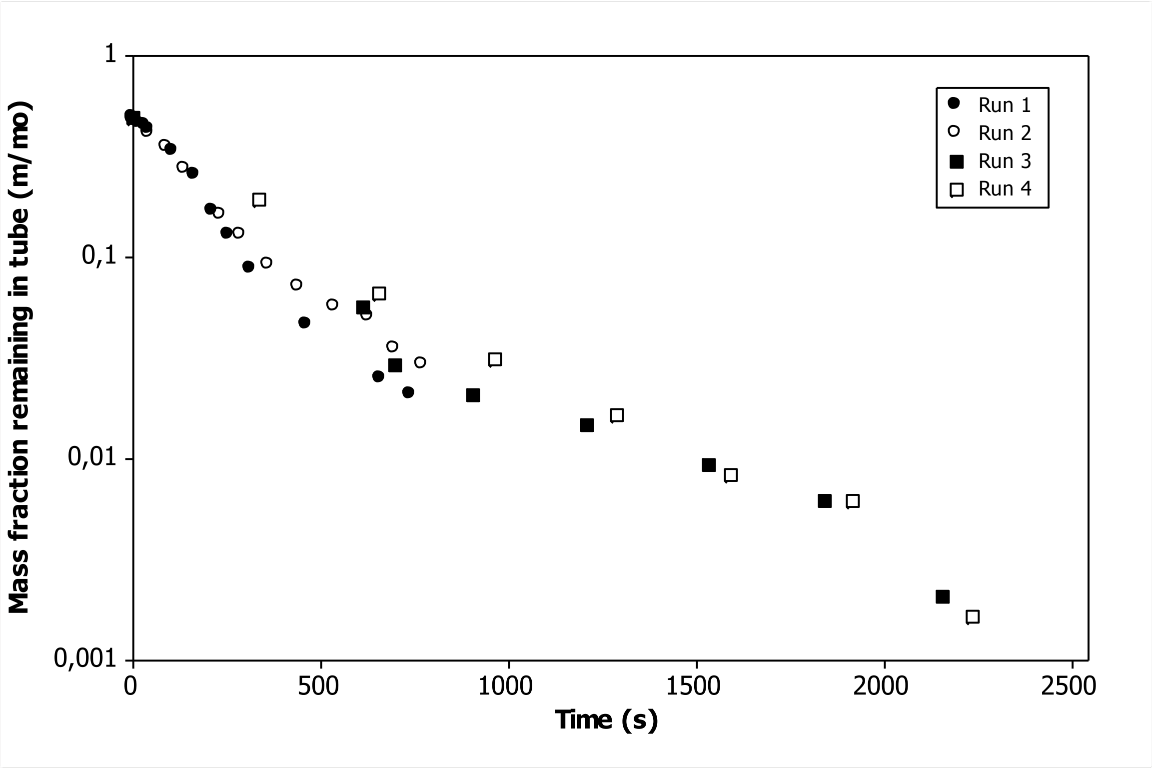


c)

Figure 3.4 (a) Shear stress vs. Shear rate of materials at 20 °C. Hand cream at 70 °C is also shown; (b) Viscosity vs. Shear rate of materials at 20 °C. Hand cream at 70 °C is also shown; (c) Oscillatory stress sweep to determine yield stress values of materials. I.e. the crossover of G’ and G’’ indicated by the arrows.


Mass remaining in tube (g)
250
200
150
100
50
0
0
500
1000
1500
2000
2500
Time (s)
Run 1
Run 2
Run 3
Run 4

a)


Mass fraction remaining in tube (m/mo)
1
0,1
0,01
0,001
0
500
1000
1500
2000
2500
Time (s)
Run 1
Run 2
Run 3
Run 4

b)

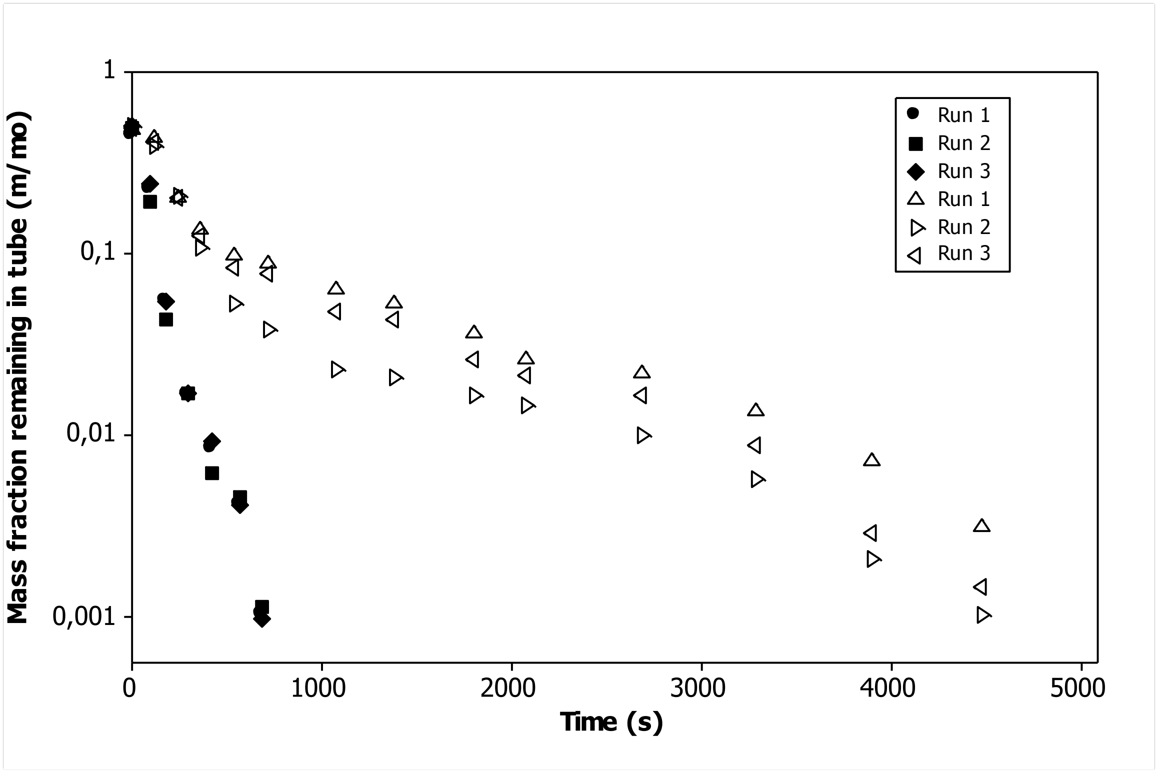
c) 

Figure 3.5 Repeatability of the experimental method by studying the cleaning of toothpaste at lab scale. m is mass of the toothpaste left in the pipe after the product recovery and m_0 is the initial mass in the fully filled pipe. Cleaning conditions are at (a) and (b) 15 °C and 0.55 m s^{-1} and (c) 50 °C and 0.55 m s^{-1} (filled symbols), 30 °C and 0.38 m s^{-1} (open symbols).

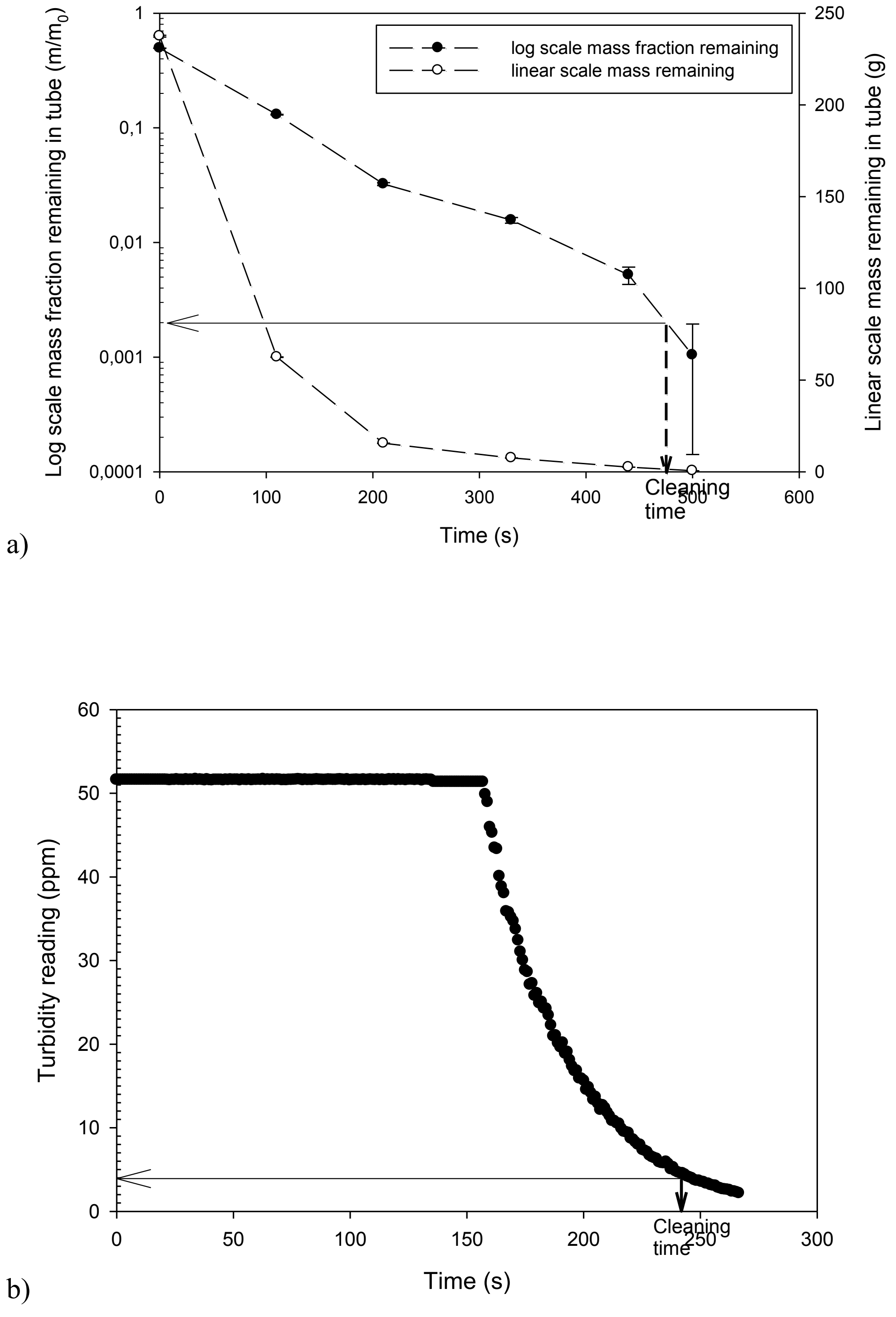


Figure 3.6: (a) Typical determination of the cleaning time during the cleaning of pipe by the weight analysis method. Cleaning was performed by water at 50 °C and 0.55 m s^{-1} in lab scale. 0.002 weight fraction is chosen as cleaning end point.(b) Typical determination of the cleaning time during the cleaning of pipe by turbidity meter reading. Cleaning was performed by water at 20 °C and 2.48 m s^{-1} in pilot scale. 4 ppm is chosen as the cleaning end point.

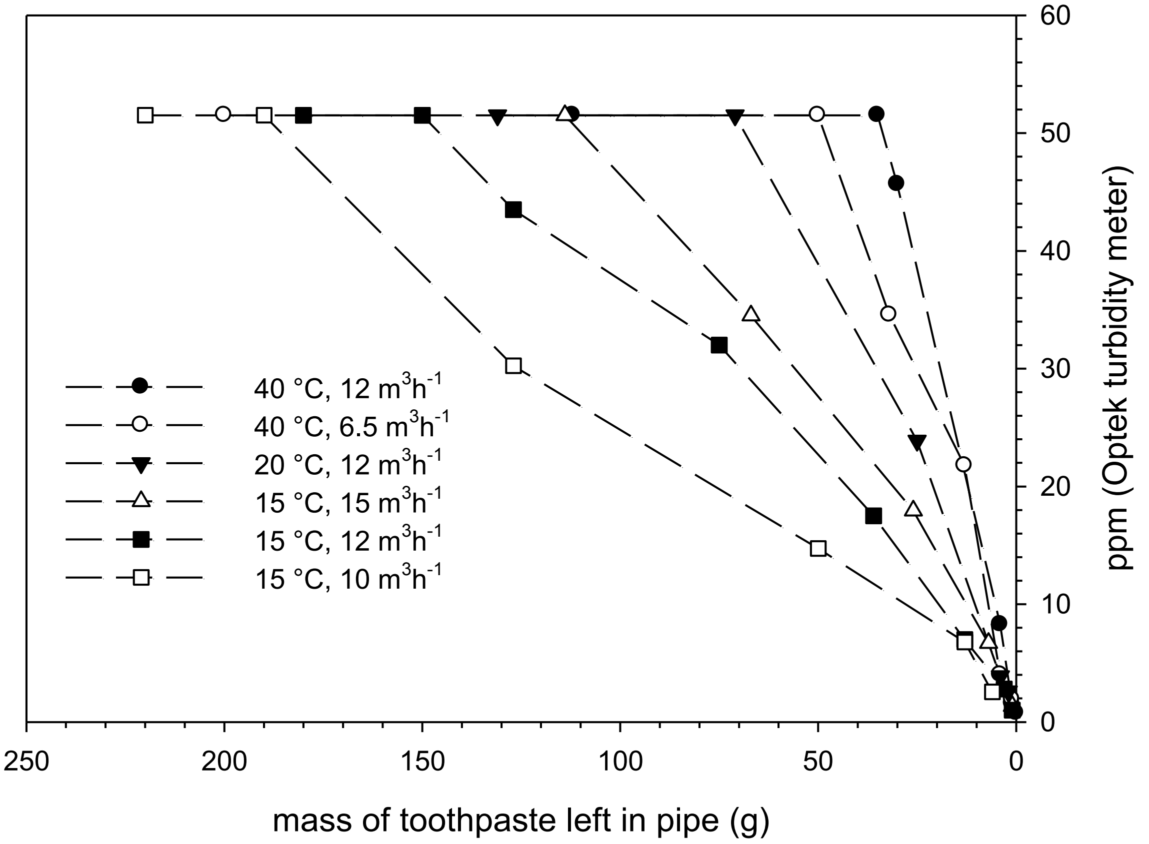


Figure 3.7: A graph of ppm response of Optek turbidity meter against mass of toothpaste left in pipe. Data points are obtained by reading corresponding ppm value on Optek turbidity meter while performing weight analysis method during experiments.

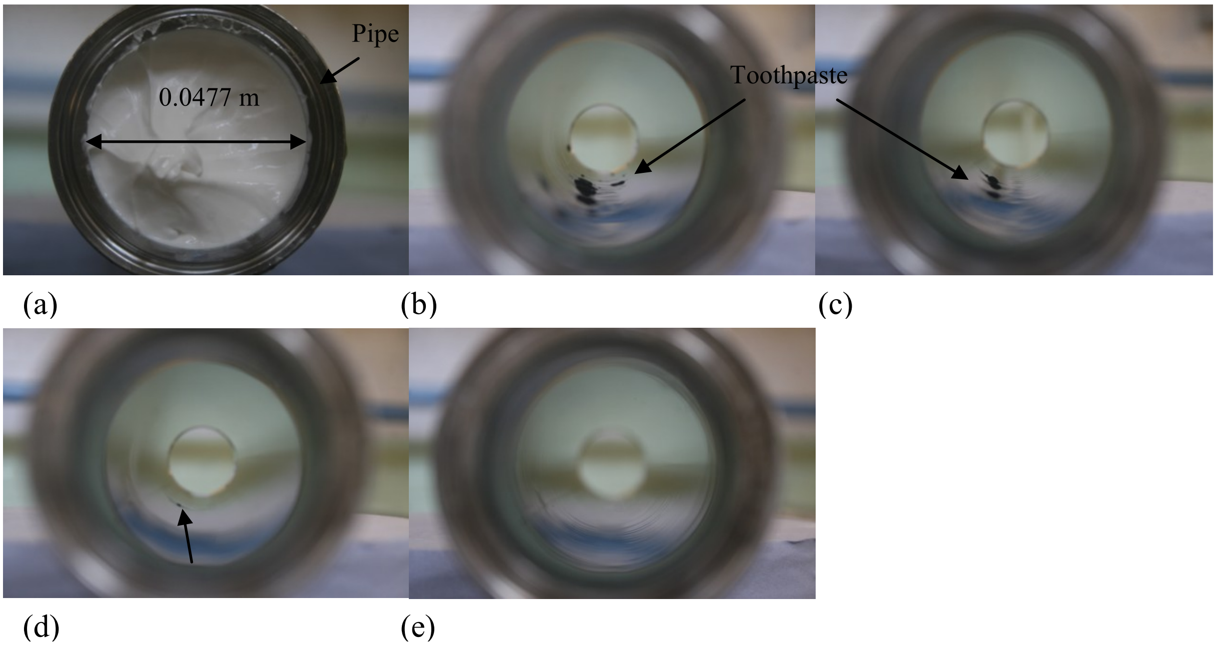


Figure 3.8: Removal of toothpaste at 20 °C, 12 m^3h^{-1} from 0.5 m long stainless steel pipe with an ID of 0.0477 m. Pictures were taken after (a) 0 s, (b) 510 s, (c) 540 s, (d) 600 s, (e) 630 s.

Chapter 4: THE EFFECT OF PRODUCT RECOVERY STAGE ON THE CLEANING OF PIPES

Chapter 1 has highlighted that cleaning process can have large environmental and economic impact. To minimise this, the cleaning process should be understood in detail. However, it is still poorly understood because complex cleaning problems have been studied so far. Chapter 2 has indicated that studying the cleaning of simple deposits (which can be removed by fluid action alone) from a straight pipe geometry might help understanding of fluid mechanical removal in the cleaning process.

To understand the fluid mechanical removal in the cleaning process thoroughly, three steps are followed in this thesis;

i) How a simple deposit is removed from a pipe geometry is analysed. This chapter and chapter 5 cover this area in detail.
ii) The effect of deposit rheology on cleaning is studied in chapter 6.
iii) The effect of flow on cleaning is investigated by comparing the cleaning of lab and pilot scale pipes in chapter 7.

Cleaning of pipes typically happens in two main stages, which is depicted in Figure 4.1. The first part of the pipe cleaning process is the “Product Recovery (core removal)” stage where water is used to push the product out from the fully filled pipe. It results in the removal of a core of material from the pipe. This stage is quick (of duration comparable to the residence time of fluid in the pipe) and leaves a thin layer of product on the pipe wall (middle picture in Fig. 4.1). As the core removal time is so short, the cleaning time of the fully filled pipe is essentially the same as the cleaning time of the thin film generated after product recovery stage.

The process variables are temperature (T_p) and flow velocity (F_p) of cleaning water used at the product recovery stage.

In the "Cleaning Stage", the thin film of the product is removed completely to provide a safe product changeover within the manufacturing plant. Here, a different temperature (T_c) and flow velocity (F_c) of cleaning water can be used, to that in the product recovery stage.

This chapter focuses on the effect of "product recovery stage" on the cleaning and Chapter 5 investigates "the cleaning stage" in detail. These chapters aim to illuminate how cleaning happens in pipes and to find out any optimisation possibilities in the cleaning of pipes.

Since the product recovery stage forms the thin film which is cleaned subsequently, two important questions arise:

i) How does product recovery stage impact on the cleaning of the generated thin film?
ii) Can we minimise the cleaning time of fully filled pipe by the product recovery stage?

At first glance, the product recovery stage is not anticipated to affect total cleaning time as it is just an initial few seconds of the pipe cleaning process. However, literature review (in section 2.6) has shown that it may affect cleaning. Also, in the industry, it is common to attempt to maximise the amount of product recovered.

This study is important to show the relation between the state of a fouling deposit and its cleaning as Wilson (2005) highlighted that understanding the nature of the fouling deposit was the first step in improving a cleaning strategy. The product recovery stage in the pipe cleaning process determines the structure of the formed thin film and this structure may affect the cleaning behaviour of a deposit. A number of numerical and theoretical modelling studies, especially from Imperial College (Sahu et al., 2007, Sahu et al., 2009, Ó Náraigh and Spelt, 2010) have been conducted to examine the stability of thin films.

The aims of this chapter are:

i) to study the effect of process variables at the product recovery stage on the amount of product recovered,
ii) to investigate the effect of the product recovery stage on the cleaning time of pipes.

To do this, temperature and velocity parameters at the product recovery stage (T_p and F_p) are changed to generate different films, then these films are cleaned at the same temperature and velocity (T_c and F_c) conditions at the cleaning stage. The conducted experiments are shown in Table 4.1 and Figure 4.2. The weight analysis method is used to monitor the cleaning process in pipes and to determine cleaning times. The experimental procedures are given in section 3.5.1.

4.1 The effect of process variables on the amount of product recovered

A series of experiments are first carried out to study the effect of product recovery stage on the amount of product recovered. The effect of temperature and flow velocity of cleaning water are studied in the lab and pilot scale during the product recovery stage.

4.1.1 The effect of temperature

Figure 4.3 gives mass ratio data plotted as a function of T_p and shows that;

- the amount of product left in the pipe after the product recovery stage increases with increasing T_p from 15 to 50 °C which means that the product recovery at high temperature decreases the amount of product recovered,
- the observed trend is clearer in the pilot scale experiments which might be by reason of higher velocity value (1.7 m s^{-1} rather than 0.55 m s^{-1}) used in the product recovery stage at this scale.

4.1.2 The effect of flow velocity

Fig. 4.4 plots the relationship between the removal of the material in the product recovery stage and the flow velocity in the product recovery stage. It illustrates that;

- although some scatter is observed in the results, general trends imply that it may be possible to relate the work at the different length scales used here. This is an area which shows both interesting experimental results and is of practical industrial value.
- increasing flow velocity decreases the amount of material left or in other words increases the amount of material recovered at the product recovery stage. One of the reasons for this increase might be that toothpaste is a shear thinning fluid thus higher

flow rates cause reduction in the viscosity of toothpaste and consequently more toothpaste can be pushed out from the pipe with increasing flow velocities. This is in good agreement with the work of Gabard and Hulin that (2003) they observed the amount of shear thinning fluid left on the tube wall decreased with increasing the flow velocity of displacing liquid.

Therefore, applying colder and higher velocity water at the product recovery stage will help increase the amount of product recovered. In the next section, the effect of product recovery stage on the cleaning time of the residual material left on the pipe wall is illustrated.

4.2 Lab Scale Experiments

4.2.1 The effect of flow velocity at product recovery on cleaning

Structure of the residual film is observed to change with the application of different product recovery conditions. First, how the flow velocity of water affects the structure of residual film is investigated. Then, the effect of film structure on cleaning is presented. Fig. 4.5 shows typical thin film structures obtained by changing F_p at the product recovery stage. F_p values below 0.55 m s^{-1} result in similar and nearly smooth thin films. The structure of toothpaste film in the middle of the pipe is generally observed to be similar to that of toothpaste film at the outlet of the pipe. However, the inside of the pipe cannot be pictured due to small pipe diameter.

Afterwards, all these residual films obtained by applying different flow conditions in the product recovery stage are cleaned at the same cleaning condition (0.55 m s^{-1} and 50 °C) to solely analyse the effect of product recovery stage on cleaning. Mass fractions remaining in the pipe are shown as a function of time in Fig. 4.6. It clearly shows that;

- cleaning behaviours of the thin films seem to be affected by the applied different velocity values in the product recovery stage,
- under the same cleaning condition, increasing F_p results in rapid cleaning of the residual film.

It should be noted that comparable cleaning behaviours are observed in Fig. 4.6 for the experiments at an F_p of 0.28, 0.33 and 0.44 m s^{-1}. This is most probably due to similar film structures obtained after the product recovery at these velocity values (see Fig. 4.5). On the other hand, the wavy film formed after the product recovery at 0.55 m s^{-1} causes completely different cleaning behaviour and relatively a short cleaning time.

4.2.2 The effect of temperature at product recovery on cleaning

First, the effect of temperature at product recovery stage on the residual film structure is studied and then the cleaning behaviour of these films are compared. Figure 4.7 shows that disturbance of the thin film increases with decreasing the temperature (T_p) of displacing water at the product recovery stage. Afterwards, these films shown in Fig. 4.7 are cleaned under the same cleaning condition (50 °C and 0.55 m s^{-1}). Different thin film structures formed by varied T_p induce different cleaning profiles in Fig. 4.8. It can be observed from Fig. 4.8 that;

- applied temperature values in the product recovery stage have an impact on the cleaning behaviours of the formed thin films,
- decreasing T_p values leads to faster cleaning of the residual films under the same cleaning condition,

Figure 4.9 summarises the effect of T_p and F_p on the cleaning times of the thin films formed at these conditions. It should be noted that the cleaning conditions, T_c and F_c (50 °C and 0.55 m s^{-1}), are the same for the all experiments in Fig. 4.6, 4.8 and 4.9. The only difference is the product recovery conditions (different T_p and F_p at the initial few seconds of the core removal process in fully filled pipes). Fig. 4.9 clearly shows that generally the lower the temperature and/or the higher the velocity in the product recovery stage, the shorter the subsequent cleaning time. The total cleaning time can be reduced up to 45 %. Therefore, the best product recovery condition is found as the highest flow velocity and the coldest temperature of water at the product recovery stage in order to decrease the total cleaning time of pipes and recover more product in the studied range of process parameters.

Notably, in some experiments at the lab scale, the same product recovery conditions does not always result in the same film structures. These experiments provide clear clues about the effect of film structure on cleaning since all experimental variables (T_p, F_p, T_c, F_c) are the same for those experiments except the film structure. Fig. 4.10 shows the result of this kind of two experiments. Mass remaining in pipe is plotted on log (Fig 4.10 (a)) and linear scale (Fig. 4.10 (b)) to analyse the results in detail. The figures graphically show that;

- mainly the thin film structure affects the cleaning time - the wavy film causes faster cleaning process. Therefore, it can be deduced that the process variables T_p and F_p at the product recovery stage have not a direct effect on the cleaning time,
- after 195 s from the product recovery stage, 57 g (20 wt % of the total material in the full pipe) less amount of toothpaste is left in the pipe for the wavy film compared to smooth film,

- the cleaning time of the smooth film is decreased by ca. 25 %, when it is made wavy initially.

4.2.3 The effect of wavy films

From the previous experiments, it is interesting to look further into the "wavy" films, in particular:

- why does wavy films cause faster cleaning?

Many experiments are done to understand the reason and capture relevant clues by taking pictures. Fig. 4.11 shows a typical time course of cleaning for different conditions, images of the film in the tube after the product recovery stage and during cleaning stage show significant differences between different product recovery conditions, and allow a hypothesis about the differences in the overall cleaning time to be developed:

- Fig. 4.11 (a) shows images after the product recovery at 15 °C and 0.55 m s^{-1}: immediately after the product recovery stage the annular film was very wavy, with three-dimensional scalloped waves with a periodicity of ca. 1.6 cm. This 3D waviness persisted in the cleaning stage, leaving a large number of small islets in Fig. 4.11 (a) (iii) after 400 s.
- In contrast, Fig. 4.11 (b) shows that after product recovery at 40 °C the film is much smoother which results in more coherent 2-dimensional waves of amplitude similar to the wavelength after 100 s. The resulting thin layer is much more uniform, and erodes to form much larger patches than in (a) after 300 s. These patches are clearly difficult

to remove by fluid shear – although the conditions at cleaning stage are the same as in 4.11 (a) much less material is removed.

The reason for the rapid cleaning of wavy films might be the higher amount of turbulence caused by the flow over the wavy film since it is well known that energy loss in pipe flow increases with increasing the roughness value of the pipe surface. Therefore, higher disturbances in the film structure may cause increased turbulence thus higher energy losses in the flow. If fluid mechanical removal of deposits happens by the hydrodynamical actions which cause energy losses in the flow, this may also explain why wavy and smooth film induce islands at different sizes in the later cleaning stages as shown in Fig. 4.11. Chapter 7 will investigate this argument in detail.

4.2.4 The effect of process variables at product recovery under variable cleaning conditions

In the above sections (4.2.1 and 4.2.2), the core of fully filled pipe are removed at different T_p and F_p. Afterwards, the generated films are cleaned at the same T_c and F_c (50 °C, 0.55 m s^{-1}) to investigate solely the effect of product recovery conditions on cleaning time. As a result, it is found that higher velocity and colder (at ambient temperature) water at the product recovery stage leads to shorter cleaning times under "one cleaning condition" due to formation of the wavier film.

In this section, different cleaning conditions (T_c and F_c) are also studied to investigate whether the explored relation between the product recovery conditions and cleaning time holds for the different cleaning conditions as well or not.

Fig. 4.12 shows the effect of velocity at the product recovery on cleaning at different cleaning temperatures. It is observed that;

- in all different cleaning conditions, the effect of velocity at product recovery stage on cleaning time is the same; high F_p induces shorter cleaning times regardless of cleaning temperatures. For instance, cleaning time decreases at least 37 % when F_p is increased from 0.33 m s^{-1} to 0.55 m s^{-1} under all cleaning conditions.
- increasing T_c always dramatically decreases the cleaning times.

Therefore, the results illustrate that the effect velocity at the product recovery stage on cleaning time is the same under the all different cleaning conditions studied.

Fig. 4.13 illustrates the effect of temperature at the product recovery on the cleaning time of pipes under different cleaning conditions. It shows that;

- reverse temperature effect is observed here; increasing T_p from 15 to 40 °C results in at least 25 % increase in the cleaning times at all cleaning conditions considered, however, increasing T_c significantly decreases the cleaning times under all product recovery conditions.

As a result, the effect of the product recovery stage on the cleaning time is found consistent under the different conditions applied in the subsequent cleaning stage.

4.3 Pilot Scale experiments

Laboratory scale experiments have illustrated the possible benefits of applying specific conditions in the product recovery stage to optimise cleaning. Cold and high velocity product recovery has resulted in reductions in the cleaning time of the formed films. However, lab scale are limited to the highest flow velocity of 0.55 m s^{-1}, far from the industrial scale, in which flow velocities of 1.5 or 2 m s^{-1} are used. Therefore, it is crucial to conduct experiments at pilot scale before identifying possible industrial benefits of the results.

4.3.1 The effect of flow velocity at product recovery

Two flow velocities are studied in the product recovery stage at pilot scale; at 0.89 m s^{-1} and 1.7 m s^{-1}. Figure 4.14 shows images of thin film structures after the product recovery at these F_p values. Similar to the lab scale experiments, slow product recovery results in the formation of a smoother film. Here, mass difference of the generated films is easily observed and slow recovery causes much thicker and heavier film. Typical cleaning profiles after product recovery at pilot scale are shown in Fig. 4.15. The data shows:

- there is an effect of the flow velocity during the product recovery stage on the amount of toothpaste present in the tube; at 0.89 m s^{-1}, ca. 500 g (45 %) is left on the tube surface (as thicker film is left), whilst ca. 340 g (30 %) is left after the product recovery at 1.7 m s^{-1}. Therefore, amount of product recovered increases by increasing flow velocity at the product recovery as is illustrated in Fig. 4.4;
- subsequently the effect of velocity during the cleaning stage is also clear, with cleaning times changing between 800 and 2300 s. The effect of the product recovery stage is clear; carrying out the product recovery at 1.7 m s^{-1}, followed by cleaning at 0.89 m s^{-1}, reduces the cleaning time from 2300 to 1400 s.

Fig. 4.16 shows the effect of different product recovery conditions on the cleaning times at the pilot scale. In Fig. 4.16 and 4.17, cleaning times are determined by the Optek turbidity probe using the value of 4 ppm. Different film structures are formed after the product recovery at 0.89 and 1.7 m s^{-1} and then these films are cleaned at flow velocities between 0.89 and 2.05 m s^{-1}. The figure shows that;

- carrying out the product recovery at 1.7 m s^{-1} decreases the cleaning time by 10 % to 45 % over the range of flow velocities studied at the cleaning stage,
- the effect of product recovery stage decreases with increasing cleaning velocity.

4.3.2 The effect of temperature at the product recovery

The effect of temperature in the product recovery stage is identified in Fig. 4.17 at the pilot scale. Experiments are done at 15 °C in the product recovery stage, then cleaning of the residual films is performed at the cleaning temperatures shown in the figure. The figure also includes the results of the previous work (Cole et al., 2010) and two repeat experiments of that work to illustrate the benefit of carrying out the product recovery stage at low temperatures. In the Cole's work, the same conditions were used in the product recovery and cleaning stages, that is, when cleaning was performed at 70 °C, the product recovery was also carried out at 70 °C. The repeats are conducted at 40 °C and 50 °C to check the validity of using the previous results for comparison. For repeat experiments, similar cleaning time is observed at 50 °C when the same cleaning procedures are performed as in the Cole's work. While, different cleaning time is obtained at 40 °C (280 s in this work and 170 s in the Cole's work). The earlier cleaning time obtained in the Cole's work is presumably caused by the flow disturbances in that experiment. This might induce wavy structure of the thin film thus cause earlier cleaning time. It can also be the explanation of the unusual result obtained in that work; cleaning time at 40 °C was shorter than the cleaning times at 50 °C and 70 °C. Fig. 4.17 suggests that;

- the cleaning time can be reduced by at least 25 % by performing the product recovery at 15 °C under different cleaning conditions. The effect is most pronounced at the cleaning temperature of 20 °C, where the cleaning time can be decreased by 50 %,
- it means that it could be possible to reduce the amount of water used in the cleaning. At the flow rates used in the experiments of Fig. 4.17, for example, water savings in cleaning are between 900 litres (50 %) and 180 (12 %) litres over the cleaning temperature range of 20 and 70 °C, i.e. the use of a short pulse of high-velocity cold fluid in the product recovery stage will reduce the total water requirement of cleaning. Water is often reused in the cleaning of process plant – however, reduction in the amount of water used in cleaning will obviously bring additional savings in pumping and heating cost.

Therefore, time, the use of energy and water can be reduced in the cleaning process by applying water at ambient temperature at the product recovery stage before the cleaning process.

4.4 Conclusions

In the cleaning of fully filled pipes, the product recovery stage is just initial few seconds of the overall cleaning process. Thus, the product recovery stage is often disregarded in the cleaning of pipes.

Therefore, the aim of this chapter is to identify whether it is possible to optimise cleaning and maximise the amount of product recovered by the product recovery stage. The effect of process variables at the product recovery stage on the amount of product recovered and on the cleaning time of the generated residual films is studied at laboratory and pilot plant scale.

The amount of product recovered increases by applying colder (at ambient temperature) and higher velocity water at the product recovery stage at both scales. These conditions are also found beneficial for decreasing the cleaning time of fully filled pipes at both scales; core removal with a short pulse of high velocity cold water results in faster cleaning process. Up to 50 % reduction is observed in the cleaning time at pilot scale. The reason of this phenomena is investigated and it is observed that the product recovery with high velocity cold (at ambient temperature) water generally induces the generation of wavier films. Our conclusion is that it is actually the structure of the thin film rather than process variables at the product recovery stage that determines the cleaning time and cleaning behaviour of the thin film. Therefore, if the product recovery stage leads to a very wavy film then subsequent removal is more rapid; this suggests that the aim of the product removal stage should be to produce a maximally disrupted form.

The results obtained here may contribute substantial savings to the industries during the cleaning and product recovery operation as same conditions (hot and high velocity water) throughout the pipe cleaning process are used in the traditional CIP applications in the plants. This chapter clearly illustrates the benefit of applying specific conditions in the product recovery stage to optimise the overall cleaning process.

Tables Chapter 4

Table 4.1: The studied temperature and flow velocity conditions at product recovery and cleaning stage and the obtained cleaning time results.

Effect of product recovery on cleaning time											
Exp.	**Product recovery conditions**				**Cleaning stage conditions**				**μ_{water}**	**$\mu_{tootpaste}$ at 100 s^{-1}**	**Cleaning time**
No	°C	m s^{-1}	Re	τ(Pa)	°C	m s^{-1}	Re	τ(Pa)	mPa.s	Pa.s	Sec.
1	15	0.28	6178	0.35	50	0.55	25229	0.94	0.55	2.55	1090
2	15	0.28	6178	0.35	50	0.55	25229	0.94	0.55	2.55	800
3	15	0.33	7281	0.46	50	0.55	25229	0.94	0.55	2.55	950
4	15	0.33	7281	0.46	50	0.55	25229	0.94	0.55	2.55	820
5	15	0.33	7281	0.46	50	0.55	25229	0.94	0.55	2.55	910
6	15	0.44	9708	0.77	50	0.55	25229	0.94	0.55	2.55	880
7	15	0.44	9708	0.77	50	0.55	25229	0.94	0.55	2.55	750
8	50	0.55	25229	0.94	50	0.55	25229	0.94	0.55	2.55	780
9	50	0.55	25229	0.94	50	0.55	25229	0.94	0.55	2.55	840
10	40	0.55	21223	0.98	50	0.55	25229	0.94	0.55	2.55	800
11	40	0.55	21223	0.98	50	0.55	25229	0.94	0.55	2.55	790
12	30	0.55	17434	1.03	50	0.55	25229	0.94	0.55	2.55	690
13	30	0.55	17434	1.03	50	0.55	25229	0.94	0.55	2.55	620
14	15	0.55	12135	1.14	50	0.55	25229	0.94	0.55	2.55	450
15	15	0.55	12135	1.14	50	0.55	25229	0.94	0.55	2.55	520
16	15	0.55	12135	1.14	50	0.55	25229	0.94	0.55	2.55	530
17	15	0.33	7281	0.46	30	0.55	17434	1.03	0.8	4.28	1900
18	15	0.33	7281	0.46	30	0.55	17434	1.03	0.8	4.28	2000
19	50	0.55	25229	0.94	30	0.55	17434	1.03	0.8	4.28	1600
20	50	0.55	25229	0.94	30	0.55	17434	1.03	0.8	4.28	1750
21	40	0.55	21223	0.98	30	0.55	17434	1.03	0.8	4.28	1680
22	40	0.55	21223	0.98	30	0.55	17434	1.03	0.8	4.28	1560
23	30	0.55	17434	1.03	30	0.55	17434	1.03	0.8	4.28	1275
24	30	0.55	17434	1.03	30	0.55	17434	1.03	0.8	4.28	1420
25	30	0.55	17434	1.03	30	0.55	17434	1.03	0.8	4.28	1800
26	15	0.55	12135	1.14	30	0.55	17434	1.03	0.8	4.28	1224
27	15	0.55	12135	1.14	30	0.55	17434	1.03	0.8	4.28	1250
28	15	0.33	7281	0.46	40	0.55	21223	0.98	0.65	3.06	1320
29	15	0.33	7281	0.46	40	0.55	21223	0.98	0.65	3.06	1550
30	15	0.33	7281	0.46	40	0.55	21223	0.98	0.65	3.06	1600
31	40	0.55	21223	0.98	40	0.55	21223	0.98	0.65	3.06	1070
32	40	0.55	21223	0.98	40	0.55	21223	0.98	0.65	3.06	1023
33	15	0.55	12135	1.14	40	0.55	21223	0.98	0.65	3.06	670

Table 4.1: continued

Effect of product recovery on cleaning time											
Exp.	**Product recovery conditions**				**Cleaning stage conditions**				**μ_{water}**	**$\mu_{tootpaste}$ at 100 s^{-1}**	**Cleaning time**
No	°C	m s^{-1}	Re	τ(Pa)	°C	m s^{-1}	Re	τ(Pa)	mPa.s	Pa.s	Sec.
34	15	0.55	12135	1.14	40	0.55	21223	0.98	0.65	3.06	700
35	15	0.33	7281	0.46	15	0.55	12135	1.14	1.15	5	3140
36	15	0.33	7281	0.46	15	0.55	12135	1.14	1.15	5	3300
37	40	0.55	21223	0.98	15	0.55	12135	1.14	1.15	5	2630
38	40	0.55	21223	0.98	15	0.55	12135	1.14	1.15	5	3100
39	15	0.55	12135	1.14	15	0.55	12135	1.14	1.15	5	2000
40	15	0.55	12135	1.14	15	0.55	12135	1.14	1.15	5	1910
41	15	0.89	38186	1.92	15	0.89	39275	2.22	1.15	5	2160
42	15	0.89	38186	1.92	15	0.89	39275	2.22	1.15	5	2210
43	15	1.7	78550	7.46	15	0.89	39275	2.22	1.15	5	1650
44	15	1.7	78550	7.46	15	0.89	39275	2.22	1.15	5	1550
45	15	0.89	38186	1.92	15	1.37	60457	4.72	1.15	5	800
46	15	0.89	38186	1.92	15	1.37	60457	4.72	1.15	5	970
47	15	1.7	78550	7.46	15	1.37	60457	4.72	1.15	5	1024
48	15	1.7	78550	7.46	15	1.37	60457	4.72	1.15	5	896
49	15	0.89	38186	1.92	15	1.64	72372	6.47	1.15	5	650
50	15	0.89	38186	1.92	15	1.64	72372	6.47	1.15	5	630
51	15	1.7	78550	7.46	15	1.64	72372	6.47	1.15	5	470
52	15	1.7	78550	7.46	15	1.64	72372	6.47	1.15	5	495
53	15	0.89	38186	1.92	15	2.05	90465	9.56	1.15	5	310
54	15	0.89	38186	1.92	15	2.05	90465	9.56	1.15	5	320
55	15	1.7	78550	7.46	15	2.05	90465	9.56	1.15	5	255
56	15	1.7	78550	7.46	15	2.05	90465	9.56	1.15	5	220

Figures Chapter 4

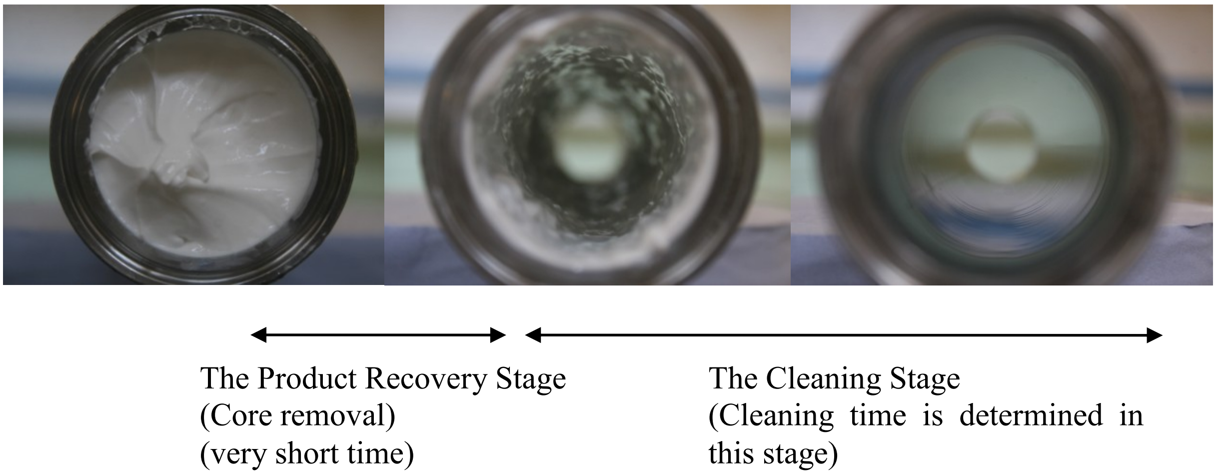


Figure 4.1: Pictures taken during the cleaning of pipes. The picture in the middle is the state of material after product recovery. The duration of this is of a similar magnitude to the residence time of fluid in the pipe.

a)

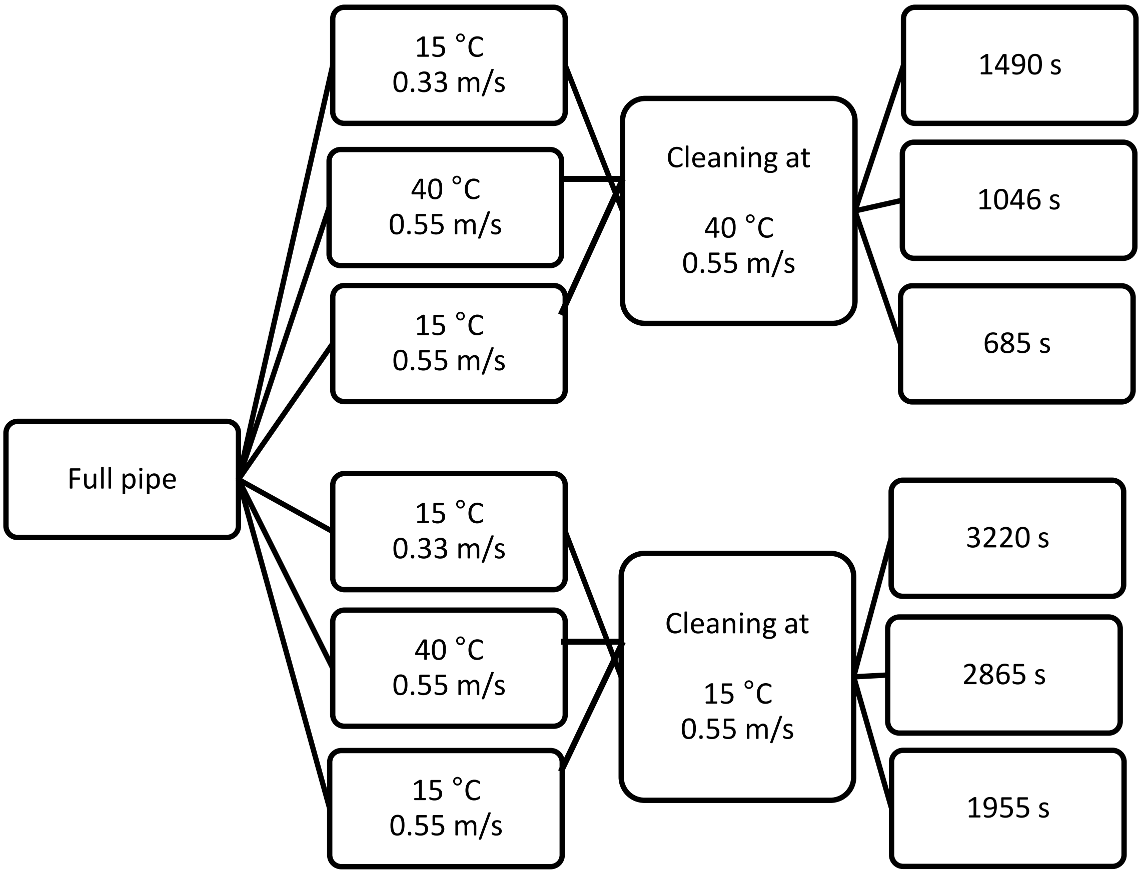
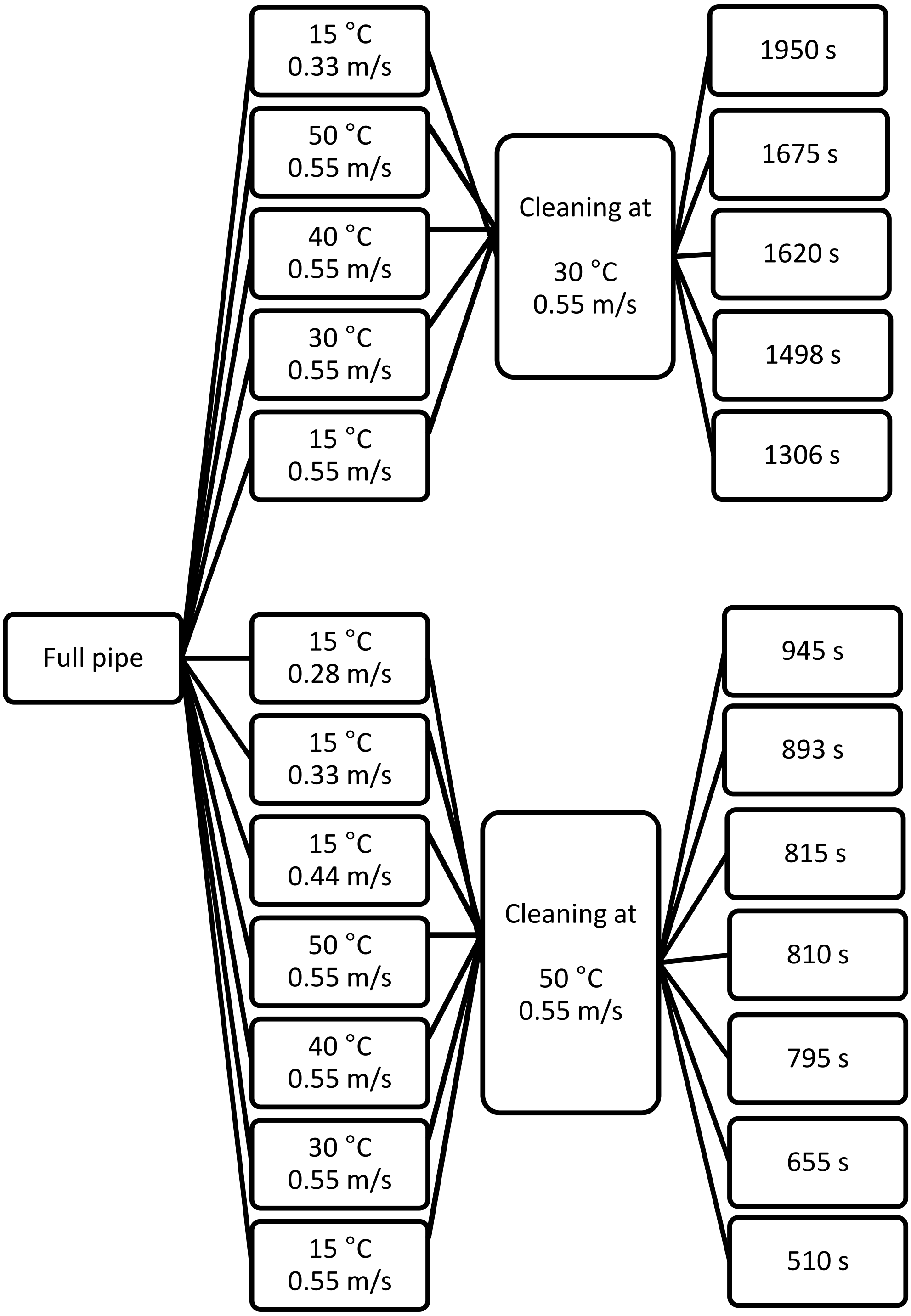


Figure 4.2: Schematic representation of the experiments which are shown in Table 4.1. a) and b) are the lab scale experiments. c) is the pilot scale experiments. Boxes in four main columns represent the cleaning process. Boxes in the 1st column represent the fully filled pipe. Product recovery stage is the 2nd column and the values inside the boxes are the studied conditions at this stage. Cleaning stage is the 3rd column and the values inside the boxes are the studied conditions at this stage. The last column is the average cleaning times of fully filled pipes resulting from the corresponding product recovery and cleaning conditions.

b)


Full pipe
15 °C 0.33 m/s
50 °C 0.55 m/s
40 °C 0.55 m/s
30 °C 0.55 m/s
15 °C 0.55 m/s
Cleaning at 30 °C 0.55 m/s
1950 s
1675 s
1620 s
1498 s
1306 s
15 °C 0.28 m/s
15 °C 0.33 m/s
15 °C 0.44 m/s
50 °C 0.55 m/s
40 °C 0.55 m/s
30 °C 0.55 m/s
15 °C 0.55 m/s
Cleaning at 50 °C 0.55 m/s
945 s
893 s
815 s
810 s
795 s
655 s
510 s

c)

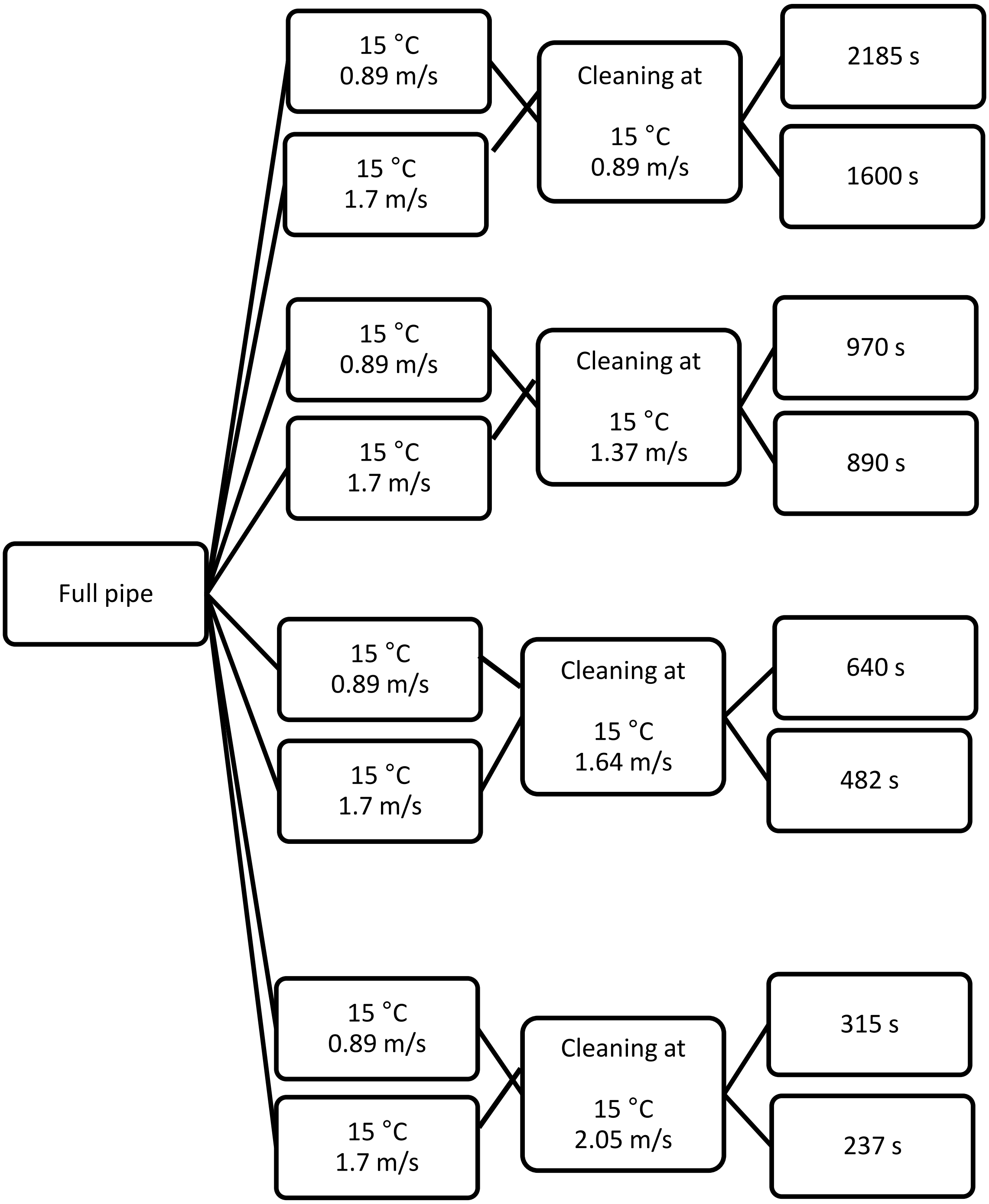


Figure 4.2 continued

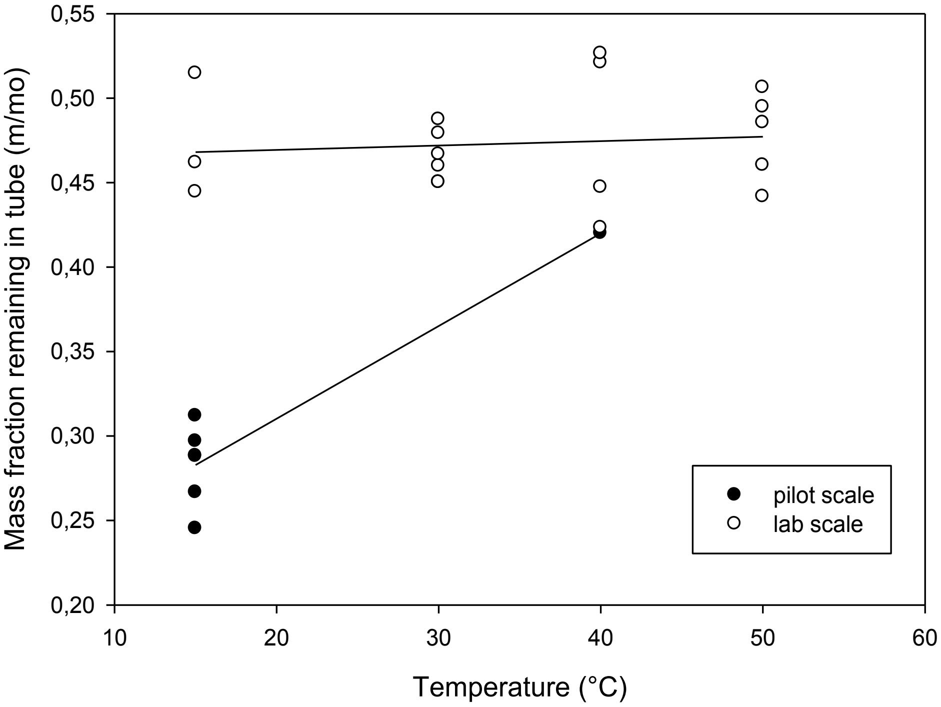


Figure 4.3: Mass fraction remaining in the pipe after the product recovery at different temperatures (T_p). m is mass of the toothpaste left in the pipe after the product recovery and m_0 is the initial mass in the fully filled pipe. Flow velocity at lab scale is 0.55 m s^{-1} and 1.7 m s^{-1} for pilot scale. Lines are plotted considering the mean values of the results at the same conditions. The pipe used in the lab scale is 1 m and has an internal diameter of 0.0239 m. In the pilot scale, it is 0.5 m and has an internal diameter of 0.0477 m.

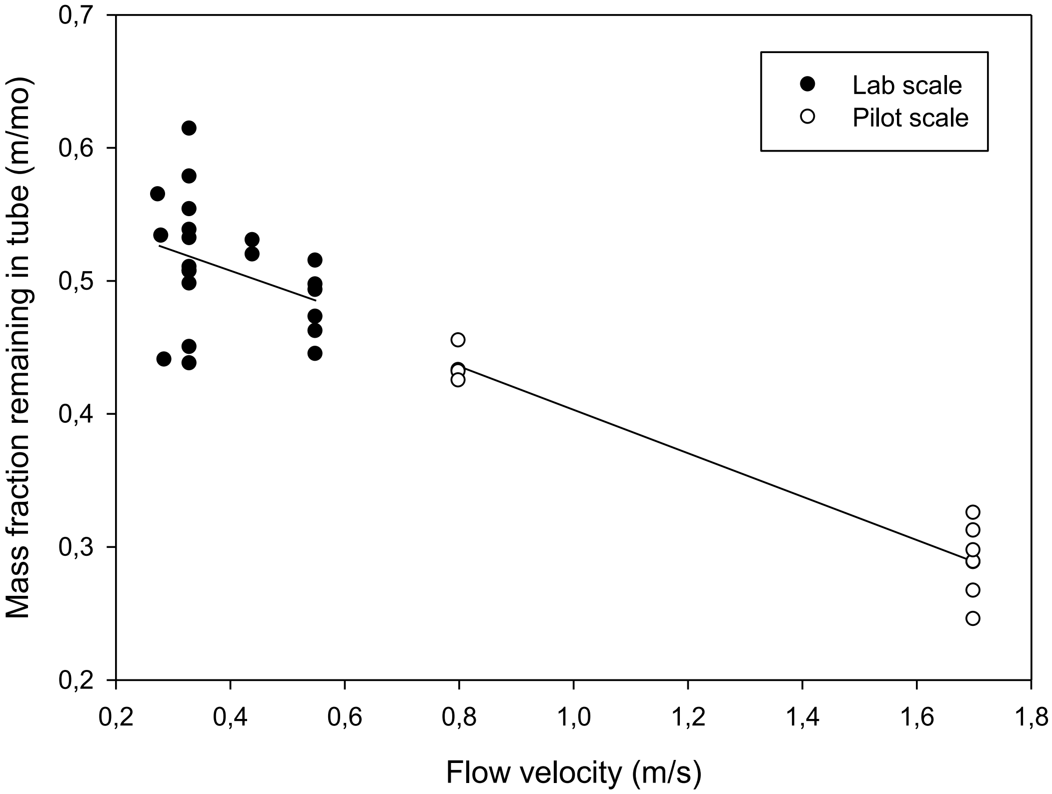


Figure 4.4: Mass fraction remaining in the pipe after the product recovery stage at different velocities at 15 °C. m is mass of the toothpaste left in the pipe after the product recovery and m_0 is the initial mass in the fully filled pipe. The pipe used in the lab scale is 1 m and has an internal diameter of 0.0239 m. In the pilot scale, it is 0.5 m and has an internal diameter of 0.0477 m. Lines are plotted considering the mean values of the results at the same conditions.

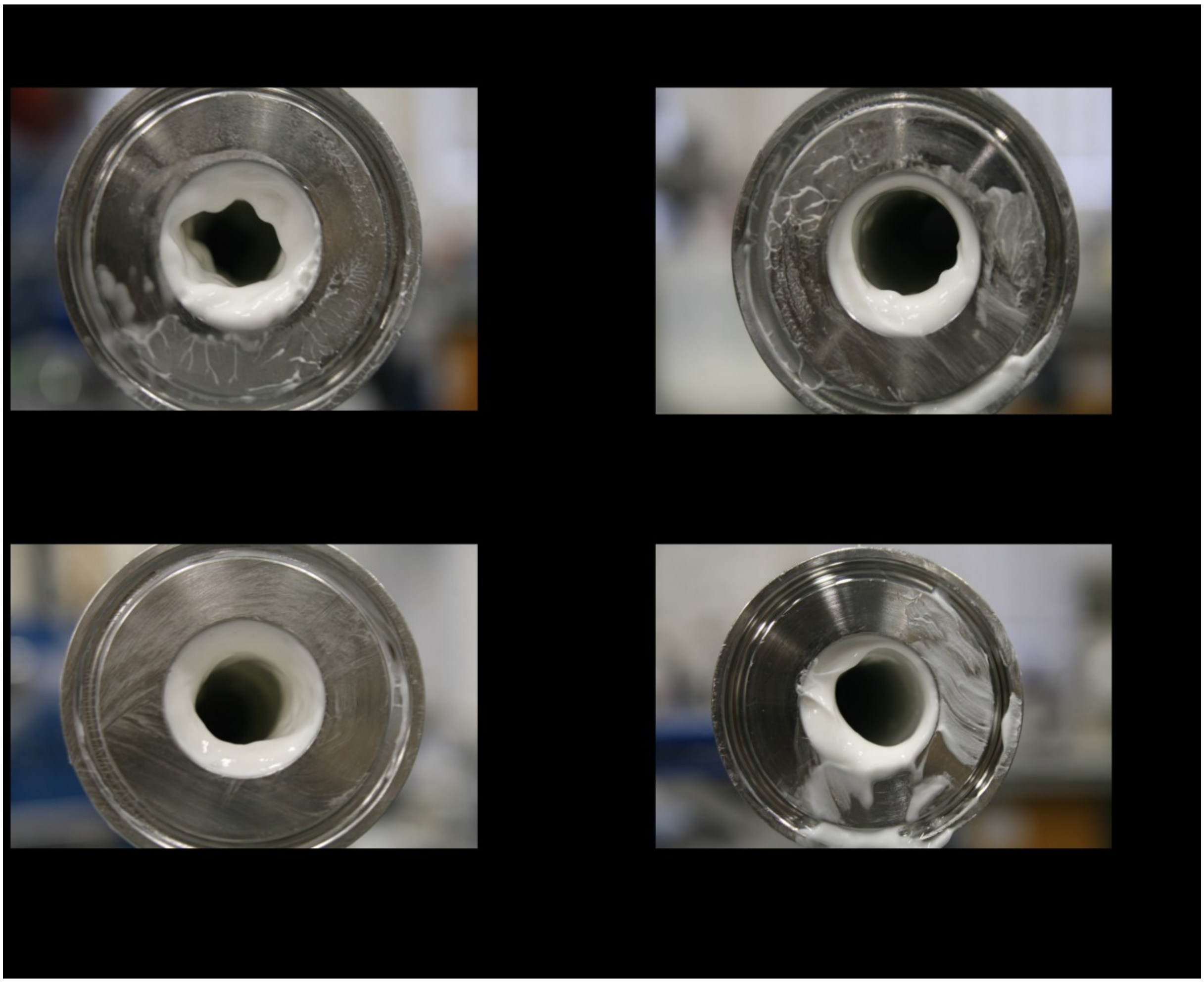


Figure 4.5: Pictures of thin film structures after different product recovery conditions. Product recovery is done at 50 °C and different flow velocities. Waviness increases by increasing flow velocity at the product recovery stage.

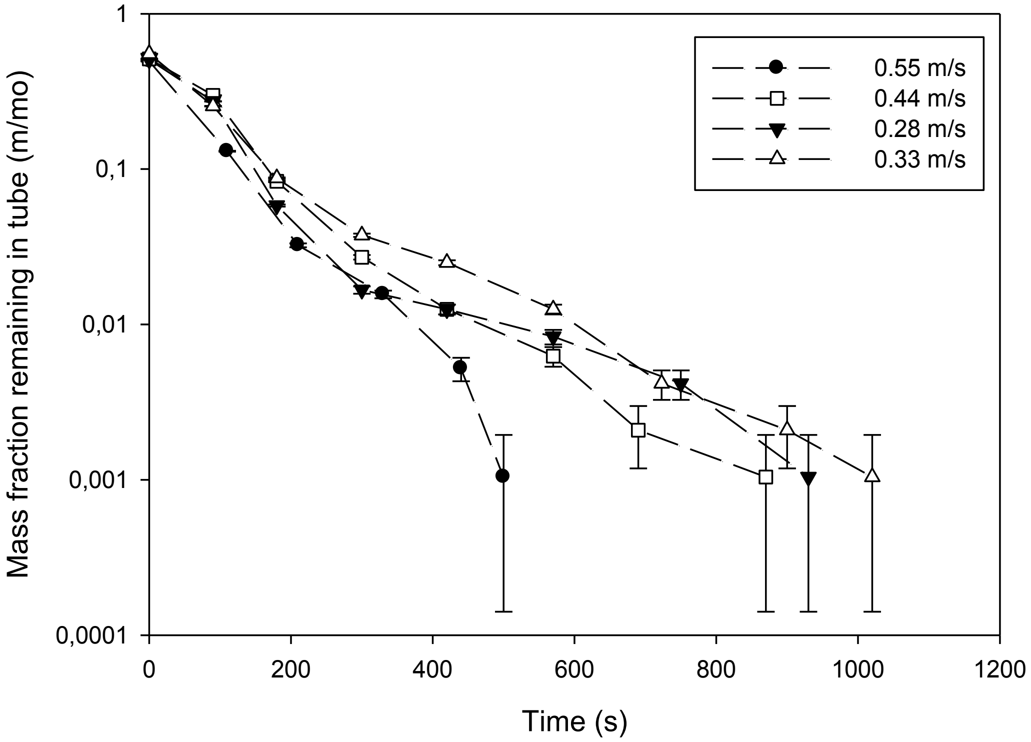


Figure 4.6: Cleaning profile of a thin film of toothpaste from a 0.0239 m pipe under the same cleaning conditions of 50 °C and 0.55 m s^{-1} water velocity. m is mass of the toothpaste left in the pipe with time and m_0 is the original mass in the fully filled pipe. Product recovery is done at 15 °C with different flow velocities of displacing water. Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.

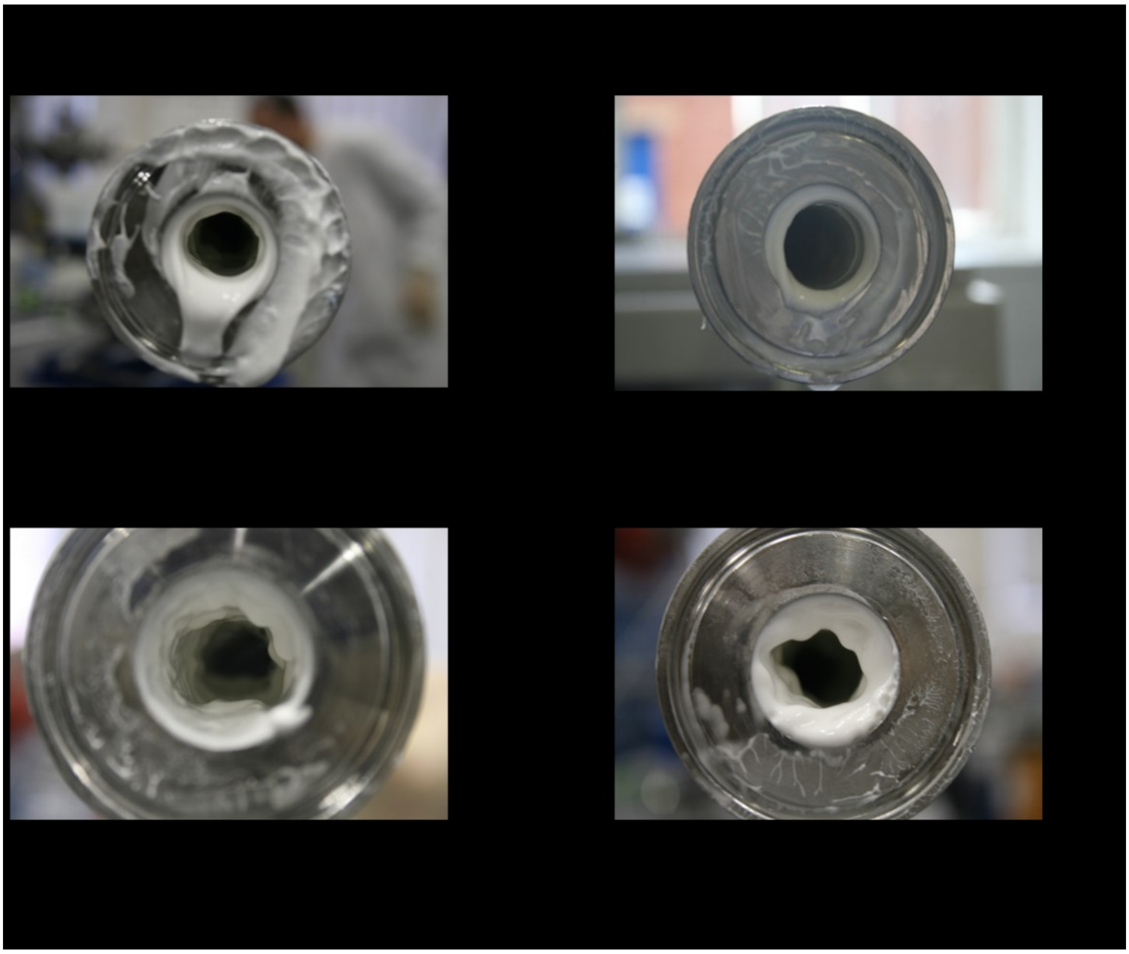


Figure 4.7: Pictures of thin film structures after different product recovery conditions. Product recovery is done at 0.55 m s^{-1} and different temperatures. Waviness seems to increase by decreasing T_p.

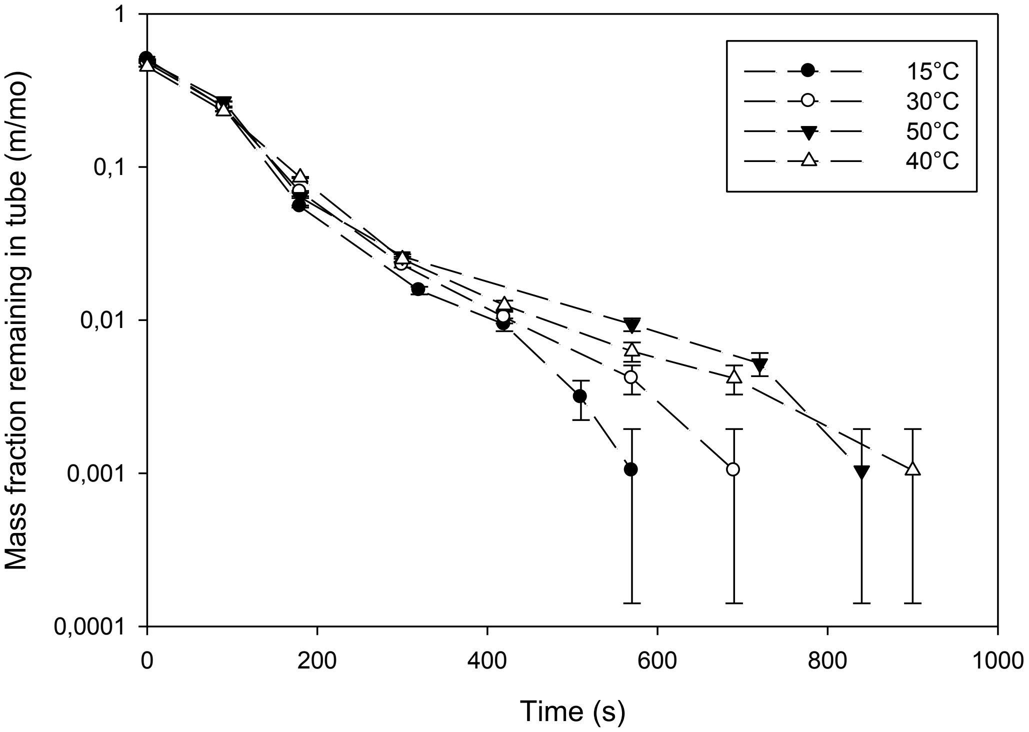


Figure 4.8: Cleaning profiles of thin films of toothpaste from 0.0239 m pipe under the same cleaning conditions of 50 °C and 0.55 m s^{-1} water velocity. m is mass of the toothpaste left in the pipe with time and m_0 is the original mass in the fully filled pipe. Product recovery is done at 0.55 m s^{-1} with different temperatures of cleaning water. Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.

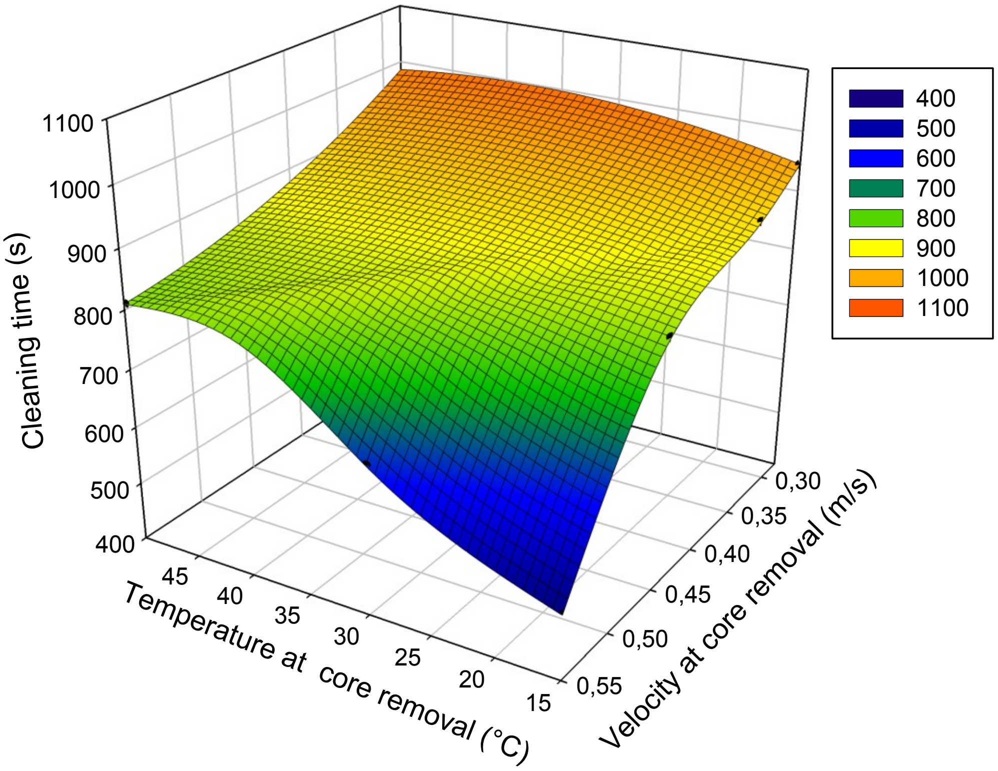
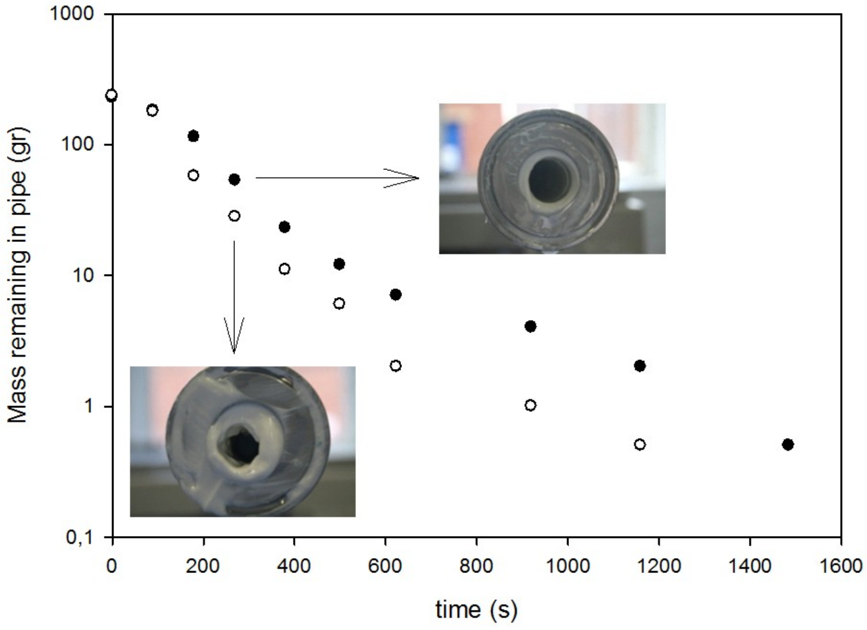
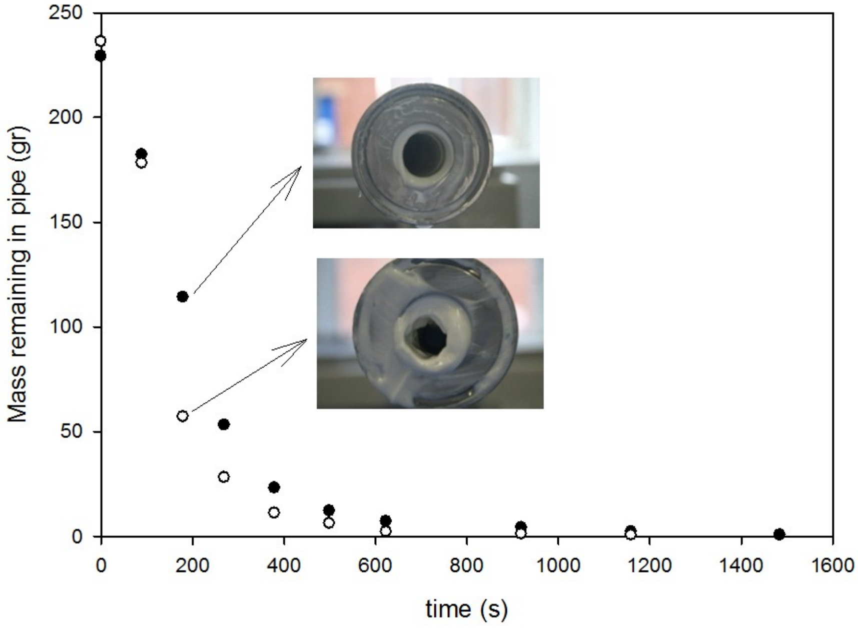


Figure 4.9: 3D representation of cleaning times of residual thin films which are formed by different flow velocity and temperature conditions at the product recovery (core removal) stage. Cleaning conditions (50 °C and 0.55 m s^{-1}) are the same for all the experiments.



a)



b)

Figure 4.10: Cleaning profiles of thin film of toothpaste deposit after the same (40 °C, 0.55 m s^{-1}) product recovery conditions. After product recovery, different film structures (smooth and wavy film) are obtained. Then, these films are cleaned at the same cleaning conditions of 30 °C, 0.55 m s^{-1}. Filled symbols show the mass data of smooth film during cleaning. Open symbols show the mass data of wavy film during cleaning. a) shows the mass remaining vs time on log scale, b) shows the mass remaining vs time on linear scale.

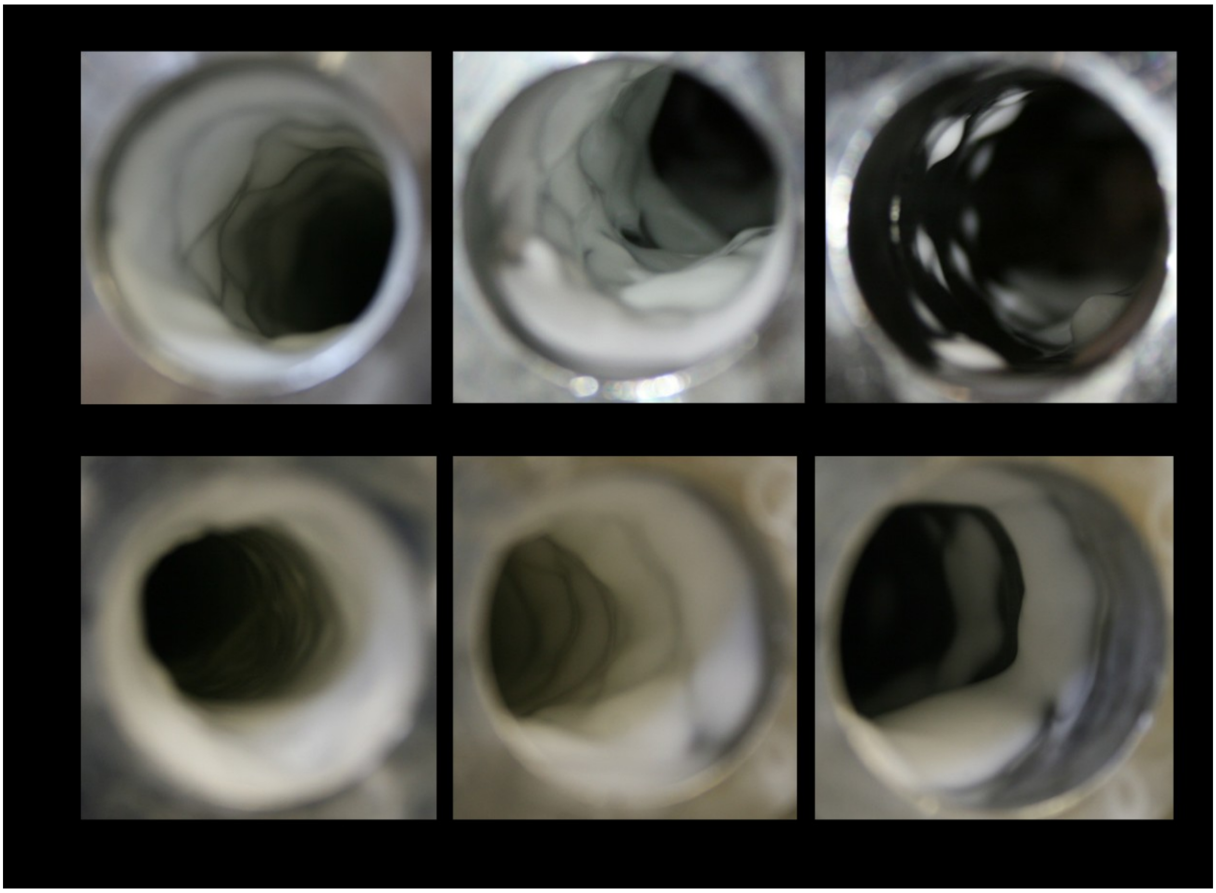


Figure 4.11: Images of thin film structures after different product recovery conditions and during cleaning (a) product recovery at 15 °C, 0.55 m s^{-1} and images are after 0 s, 100 s, 400 s from the core removal stage, (b) product recovery at 40 °C, 0.55 m s^{-1} and images are after 0 s, 100 s, 300 s from the product recovery stage. In both cases cleaning conditions are at 50 °C, 0.55 ms^{-1}.

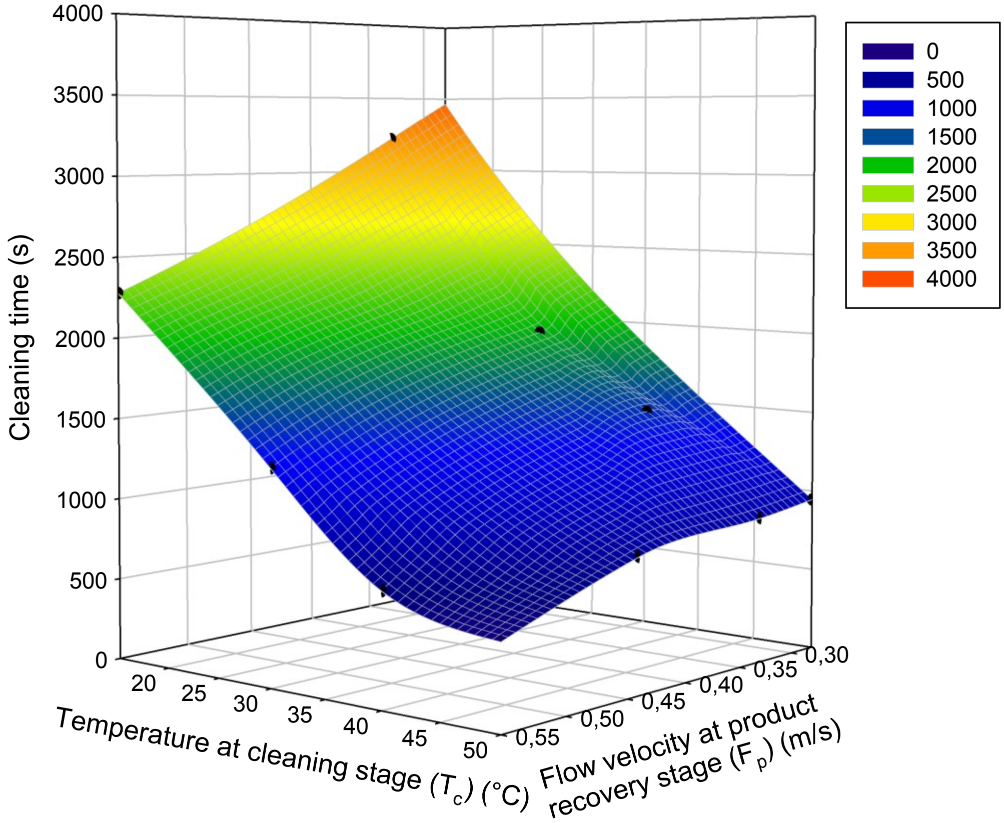


Figure 4.12: 3D representation of cleaning times of residual thin films which are formed by different flow velocity conditions at product recovery (core removal) stage at 15 °C. Different temperatures are used to clean thin films at 0.55 m s^{-1}.

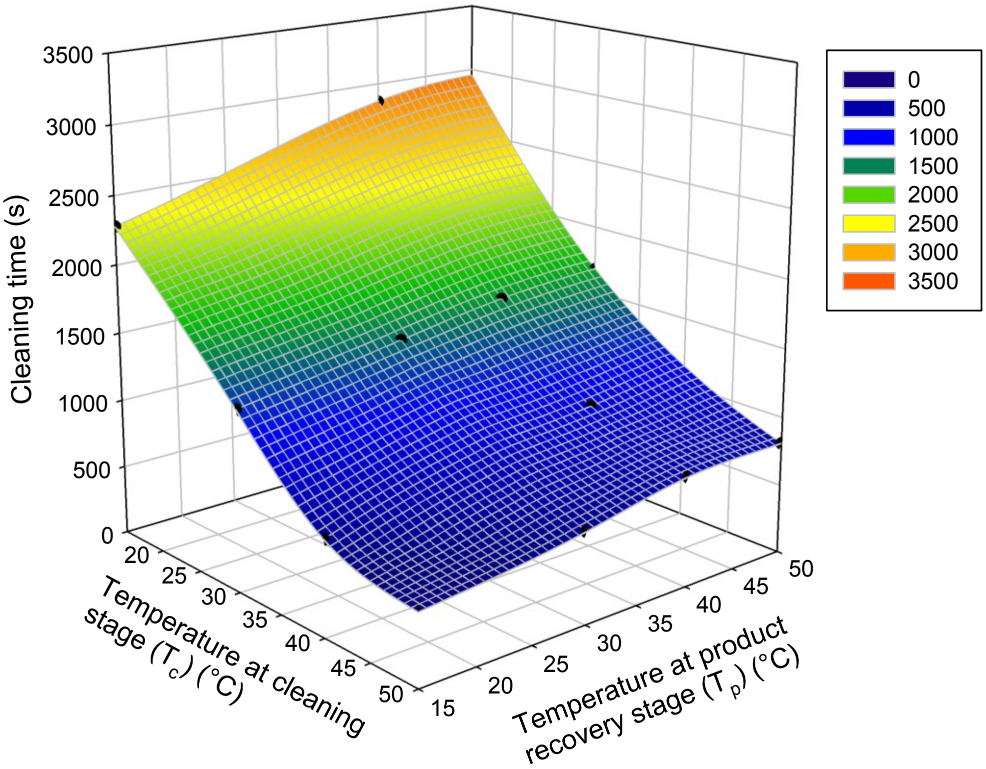
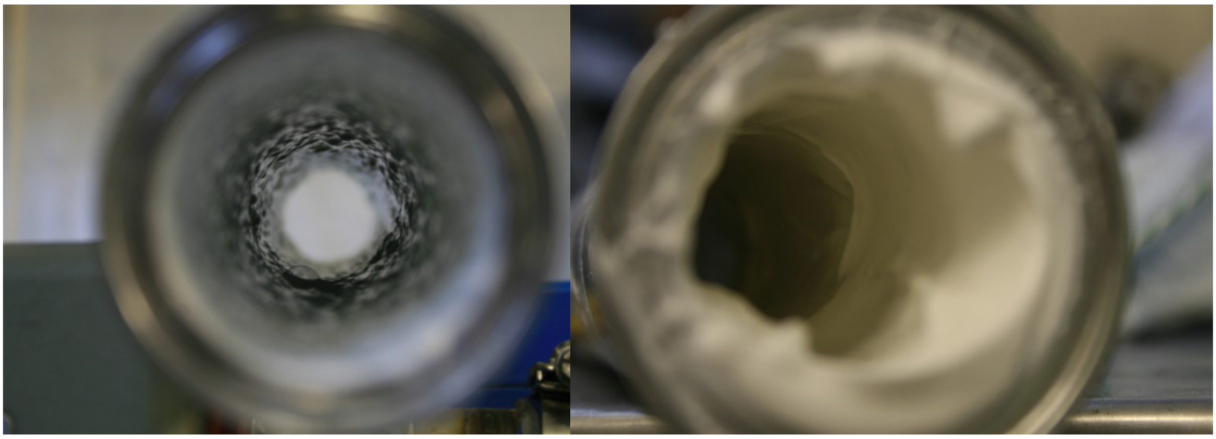


Figure 4.13: 3D representation of cleaning times of residual thin films which are formed by different temperature conditions at the product recovery (core removal) stage. Different temperatures are used to clean thin films. The same velocity of 0.55 m s^{-1} is used in both stages.



after product recovery at 1.7 m s^{-1} after product recovery at 0.89 m s^{-1}

Figure 4.14: Images of thin film structures after product recovery at different velocities at pilot scale. Temperature is at 15 °C.

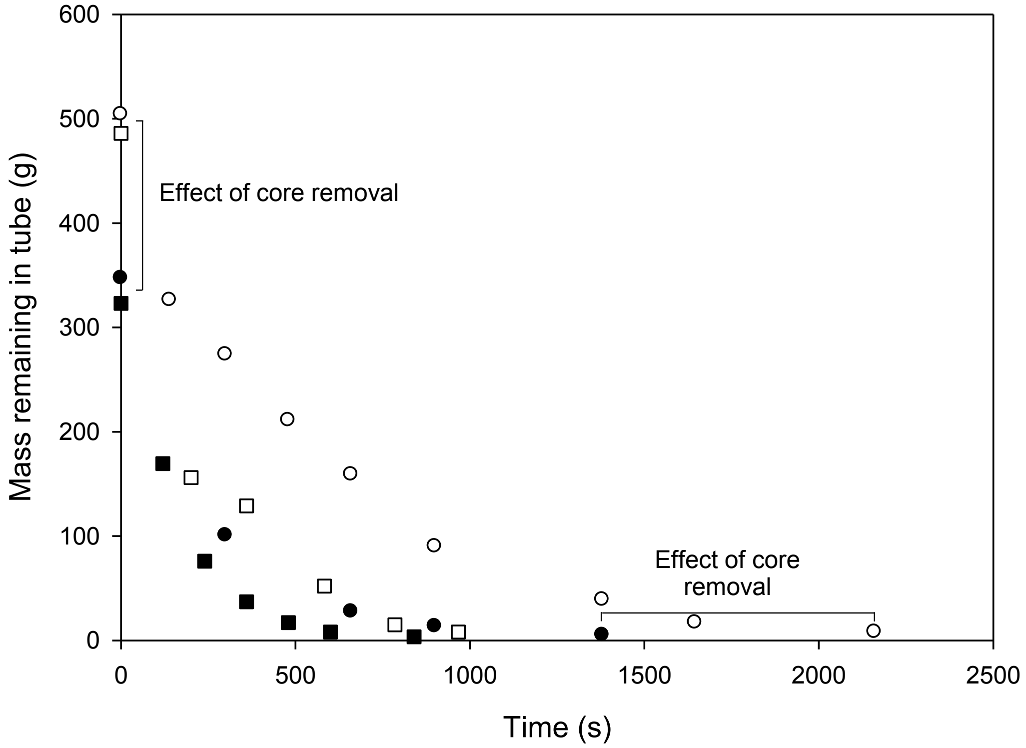


Figure 4.15: Cleaning profiles of a layer of toothpaste after the product recovery at 0.89 m s^{-1} (open symbols) and 1.7 m s^{-1} (filled symbols). Cleaning conditions are 0.89 m s^{-1} (circles) and 1.37 m s^{-1} (rectangles) at 15 °C.

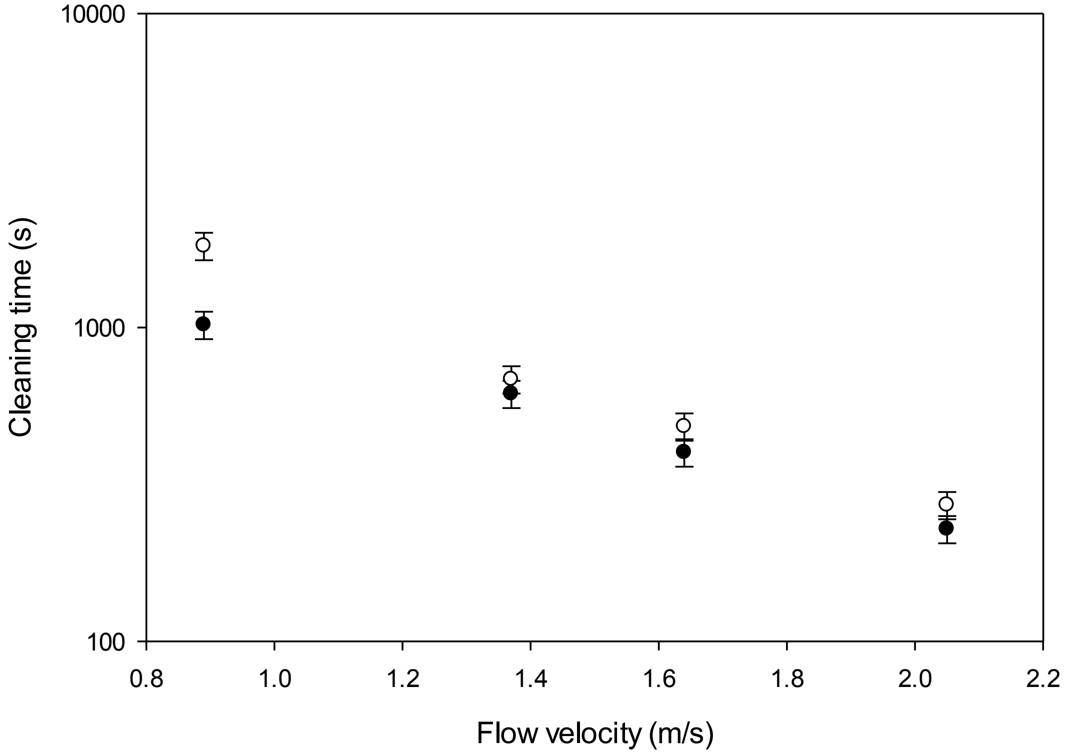


Figure 4.16: Cleaning time versus flow velocity during cleaning for different applied product recovery conditions; 0.89 m s^{-1} (open symbols) and 1.7 m s^{-1} (filled symbols). Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.

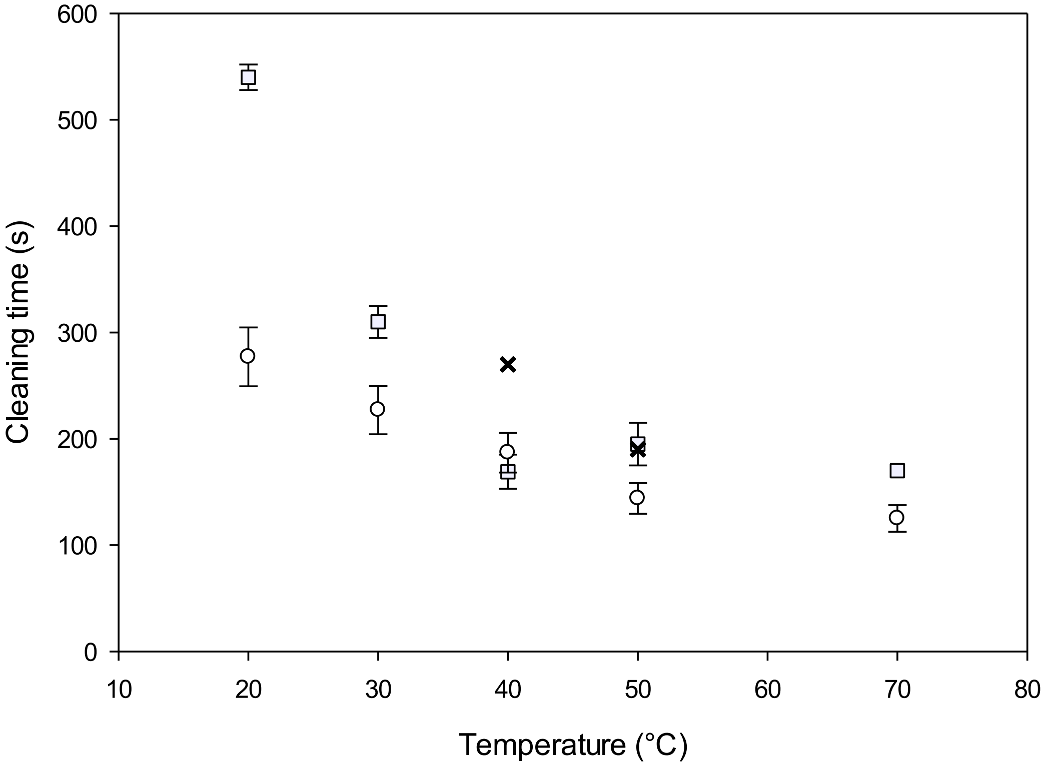


Figure 4.17: Cleaning time versus cleaning temperatures for different applied product recovery conditions: ○; product recovery is only done at 15 °C , □; results of the reference (Cole et al., 2010), product recovery is done at the same temperature to the cleaning temperatures, x; repeats of the same experiments from reference (Cole et al., 2010) at 40 and 50 °C. In the cleaning stage, 1.7 m s^{-1} water is used for all experiments. Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.

Chapter 5: FLOW REGIMES IN THE CLEANING OF PIPES

Chapter 4 has revealed that the emptying of pipes happens in two main stages; (i) the product recovery stage and (ii) the cleaning stage and it has been found that optimisation of the cleaning of pipes can be possible by the product recovery stage. However, the cleaning stage has not been fully covered in Chapter 4. Therefore, this chapter focuses on how cleaning happens after the product recovery stage in pipes and illustrates how further optimisation can be made in this cleaning stage.

Toothpaste is used as a model deposit in chapter 4 and this chapter to characterise the cleaning behaviour of pipes for two main reasons. Firstly, Chapter 2 has highlighted how complex the cleaning process is when chemicals are involved in the cleaning. Also, understanding cleaning of one soil which can be cleaned by fluid action alone may help exploration of the cleaning mechanisms for the other soils which need chemicals to be cleaned. Secondly, previous studies have generally focused on the cleaning of deposits formed after heat treatment such as milk, whey protein or tomato deposits which form thin films of mm-scale deposit (Christian, 2003, Gillham et al., 1999, Liu et al., 2007). However, many food and personal care products form a type of deposits which consists essentially of the product (such as pastes and creams) that forms cm-thick layers on the surfaces of tanks and vessels and can completely fill pipework. The problem in these soils is thus one of liquid-liquid displacement; of ensuring that pipes and tanks can be emptied as efficiently as possible. Cleaning is anticipated to depend on the material rheology for this kind of deposit (Fryer and

Asteriadou, 2009). Hence, the results of this investigation may well be appropriate for the cleaning of a wide range of yield stress materials.

It should be noted that in chapter 4, the effect of product recovery stage is appreciated. Hence, the most efficient product recovery conditions are applied in all experiments presented in this chapter (1.7 m s^{-1} and 15 °C for the pilot scale and 0.55 m s^{-1} and 15 °C for the lab scale).

This chapter aims;

- to understand the mechanisms involved in the cleaning of toothpaste from both lab and pilot scale pipework.
- to identify possible removal stages in the cleaning process,
- to investigate the effect of process variables (temperature and flow velocity) to assess the importance of each variable in different removal stages,
- according to results, to propose a more efficient CIP protocol which will decrease the energy consumption compared to the traditional CIP protocols.

5.1 Analysis of removal profiles and comparison with the other deposits

Cleaning rate is invaluable data in order to identify the removal mechanisms and thus understand and optimise cleaning. Fig. 5.1 shows a typical removal profiles of thin film of toothpaste from the pipe wall identified by the weight analysis method. Cleaning rates in this figure are calculated by the following expression;

$$Cleaning\ rate\ at\ \frac{t_i+t_{i+1}}{2}=\frac{x_i-x_{i+1}}{t_{i+1}-t_i} \qquad (5.1)$$

where X_i and X_{i+1} are the series of the measured masses, t_i and t_{i+1} are the times when X_i and X_{i+1} are measured.

It shows that there are two distinct cleaning behaviours which depend on the cleaning conditions at the lab scale:

- In Fig. 5.1 (a), cleaning is performed at 50 °C, 0.28 m s^{-1} water velocity. Initially, "induction stage" is observed where cleaning rate significantly increases from 0 to 1.3 g s^{-1}. Then, it is followed by a nearly "constant cleaning rate stage" which takes about 60 s at a rate of ca. 1.2 g s^{-1}. After that, removal rate decreases sharply to a very low value of ca. 0.03 g s^{-1}. As can be seen from the figure that at this cleaning rate, there is still 70 g of toothpaste film left on the pipe wall and cleaning is completed after a long time (5460 s).
- In Fig. 5.1 (b), toothpaste is cleaned at 15 °C, 0.55 m s^{-1} water velocity. Contrary to the removal rate behaviour in 5.1 (a), the "induction stage" is not observed and removal starts with the "constant rate stage". It is followed by the "decay stage" where cleaning rate decreases significantly from 0.8 to 0.005 g s^{-1} between 200 s and 400 s. Then, 8 g of toothpaste is still left on the pipe wall and cleaning is completed after 1496 s.

Notably, the observed cleaning rate profile in Fig. 5.1 (a) is similar to the cleaning rate profiles of whey protein and milk deposits in Bird and Fryer (1991), Gillham et al. (1999), Xin et al. (2004) (see figure 2.2 in Chapter 2). It implies that cleaning mechanisms are potentially the same although they are very different types of soils. For instance;

For the "induction stage": This stage is generally observed when the initial form of any deposit is such that it cannot be removed by the fluid shear alone. In this experiment, water diffusion and temperature presumably softens and changes the rheology of the toothpaste in this period. Similarly, in the removal of whey protein or milk deposit, fluid shear is not enough to remove the deposit in its initial form. Therefore, after treatment with chemicals, chemical diffusion and chemical reaction (which are temperature dependant processes) take place in the induction period which consequently changes the form of the deposit. It can be deduced that if the fluid shear is strong enough to remove the soil in its initial state, no induction period will be observed. For instance, in Fig. 5.1 (b), the cleaning rate directly starts with the constant rate stage because the velocity used in Fig 5.1 (b) is higher than in Fig. 5.1 (a).

The role of temperature in the cleaning can be recognised as changing the form of deposit into the removable form either by chemical reaction, chemical diffusion or rheology change. If there is a limit in the softness of the deposit where further temperature increase cannot change the state of the deposit any more, temperature increase will be ineffective and fluid shear becomes important.

For the "constant and decay stages": The form of the deposits is vulnerable to fluid shear thus any deposit can be removed rapidly in this stage. Then, decay stage is observed for the both types of deposits (toothpaste and whey protein) simply because if the amount of deposit decreases on the surface, detachment frequency will decrease thus removal rate decreases. Both periods are dominated by the fluid shear.

The reason why the same removal rate behaviour is observed in these different type of deposits can be attributed to their same cohesive-adhesive behaviour. For instance, Liu et al. (2006) showed that adhesive force between whey protein deposit and the cleaning surface was stronger than cohesive forces between the elements of whey protein deposit. Similarly, Akhtar et al. (2010) observed the same behaviour for toothpaste deposit. Thus, different removal rate profiles might be expected for deposits which show contrarily stronger cohesive forces than adhesive forces such as egg albumin and tomato deposits (Liu et al., 2007, Liu et al., 2002).

It should be noted for the removal of toothpaste deposit (shown in Fig. 5.1), after some point, removal rate distinctly decreases to a very low value and it stays nearly constant for long time until the pipe wall is visually clean. No chunk removal is observed in this stage which is the answer of why the removal rate is very low in this period. The removal here happens by the erosion of deposit since strong adhesive forces controls the removal in this final cleaning stage. The reason why this slow cleaning rate period is not observed in the removal of whey protein deposits might be that the chemical exposure also significantly weakens the adhesive bonds in whey protein deposit. Therefore, there is not any cleaning stage where strong adhesive forces control the cleaning of whey protein deposits.

5.2 Stages of cleaning

Fig. 5.2 (a) shows typical behaviour of the system; here 480 g of toothpaste (fully filled pipe) is cleaned by water at 0.55 m s^{-1} and 15 °C throughout the process. Same experimental data is plotted on log scale in Fig. 5.2 (b) in order to differentiate the cleaning regions more easily. The figure and examination of the inside of the tube, shows that there are two main stages in the cleaning of pipes after the product recovery stage:

i) In the 1st region of cleaning (film removal stage - until ca. 1000 s) there is still a continuous annular layer of material on the pipe wall; removal follows an exponential behaviour. Previously found “induction period”, “constant rate period” and “decay period” are included in this region and the deposit is typically removed by the detachment of chunks. This region can be modelled simply by using first order removal kinetics since data fits on a straight line when it is plotted on log scale (as shown in Fig. 5.2 (b)). This region is mainly controlled by the cohesive forces between deposit molecules. Notably, ca. 95 wt % of the thin film is removed in this period and it takes nearly half of the total cleaning time.

ii) In the 2nd region of cleaning (patch removal stage - here > 1000 s) the continuous film is broken up and patches of toothpaste are left on the surface which are gradually eroded away. Extremely slow removal is observed here as the rest of 5 wt % of the thin film is cleaned here by taking the other half of the total cleaning time. No chunk removal is observed anymore.

Typical stages of the cleaning process are shown visually in Fig. 5.3. Distinction between the 1st and 2nd stage of cleaning is made by analysing log scale removal data and observing the effluent water visually;

- when mass of the toothpaste is less than 10 g, 2nd stage generally starts in the lab scale.
- 2nd stage starts when the removal rate is very low.
- chunk removal is observed in the effluent turbid water during the 1st stage of cleaning whereas for the 2nd stage, effluent water nearly appears clear due to low cleaning rate.

In the next section, the effect of process variables on each cleaning stage is investigated to understand and optimise cleaning. In the following sections, data has been plotted;

i) as the weight of material left in the pipe, and

ii) As h (film thickness) against time, where it is assumed that the toothpaste film is smoothly spread throughout the pipe with same h everywhere in order to make comparison between lab and pilot scale data and experiments with different pipe lengths. h is thus found from:

$$m = \rho L \pi \left(R^2 - (R-h)^2 \right) \qquad (5.2)$$

where ρ is the density of the toothpaste and L and R are the length and radius of the pipe.

5.3 The effect of process variables at lab scale

5.3.1 The effect of temperature

The effect of temperature on the cleaning of thin film of toothpaste is studied at constant velocity of 0.55 m s^{-1}. The experimental results are shown in Fig. 5.4. It illustrates that;

- two regions are clearly distinguished and cleaning rate increases with increasing temperature in both regions.
- 2nd stage starts when h decreases below 0.01 cm which corresponds to 8 g toothpaste in the pipe.
- notably ca. 96 wt % of toothpaste is cleaned in the 1st stage and approximately half of the total cleaning time is spent in the 2nd stage.

5.3.2 The effect of flow velocity

The flow effect on the cleaning of thin film of toothpaste is investigated at flow velocity values ranging from 0.38 to 0.55 m s^{-1} at 30 °C. Figure 5.5 clearly shows that;

- the 1st cleaning stage is not affected significantly by increasing flow velocity. This may be due to the fact that studied velocity range is so narrow in the lab scale that fluid shear differences has not an impact on the removal rate of toothpaste,
- however, for the 2nd cleaning stage, distinguishable increase is observed in the cleaning rates with increasing velocity especially from 0.38 m s^{-1} to 0.45 m s^{-1}. So this finding suggests that the sensitivity of the 2nd stage to the flow velocity is more than that of 1st stage at lab scale. It should also be noted that the 2nd stage starts very early for the experiment conducted at the lowest flow velocity (0.38 m s^{-1}) and this results in very long cleaning time (ca. 4500 s).

For lab scale experiments, these conclusions can be drawn;

- For the 1st stage of cleaning process, temperature is the dominating parameter over the flow velocity.
- For the 2nd stage, both temperature and flow velocity are important in the cleaning process.

These findings suggest that optimisation can be done by applying different conditions during the cleaning process. For instance, application of low velocity in the 1st stage of cleaning will reduce the energy and water consumption without deteriorating the cleaning performance, thus it shows practical industrial value. However, the results found at the lab scale may not fit to the industrial scale. Therefore, the effect of process variables on cleaning at the pilot plant scale is illustrated in section 5.4.

5.3.3 The effect of Reynolds number

To understand the effect of Reynolds number on cleaning, experiments have been performed at the same Reynolds number (12000) by adjusting flow rate and temperature at the lab scale. Results are presented in Fig. 5.6.

- Remarkably all results collapse onto a single line in the 1st stage (initial ca. 500 s). That is, in this region, an experiment at 50 °C, 0.28 m s^{-1} gives the same cleaning performance as one at 15 °C and 0.55 m s^{-1}. Removal is well correlated by the bulk flow in this area, which is dominated by the removal of deposit in chunks; here the fluid force on the deposit will be important;
- however, in the 2nd stage, Re is not a good predictor of the cleaning process; for example, at 50 °C, 0.28 m s^{-1} extremely long removal of ca. 5700 s is found, whilst cleaning at 15 °C, 0.55 m s^{-1} results in the shortest cleaning time.

These experiments suggest that the cleaning time is more a function of the velocity of the water rather than the Reynolds number. Moreover, flow velocity rather than the rheology dominates the cleaning process here since cleaning time of toothpaste at 50 °C is longer than that of toothpaste at 15 °C although a deposit with a low viscosity is anticipated to be cleaned earlier. This is because low velocity water is used in the cleaning at 50 °C.

5.3.4 The effect of pipe length

Cleaning experiments are performed at 0.5, 0.8, 0.9 and 1 meter pipe to assess the effect of pipe length on cleaning rate and time. Fig. 5.7 clearly shows that;

- apparent deposit thickness data from the different lengths are similar and there is no significant difference between different lengths.

This suggests that removal is uniform throughout the pipe length and 1st and 2nd cleaning stages do not depend on the pipe length. This result implies that cleaning mainly happens by the help of fluid mechanical parameters, which stay uniform throughout the pipe length. This will be investigated in Chapter 7.

5.4 The effect of process variables at pilot scale

Experiments are performed at the pilot scale to investigate the effect of process variables on cleaning stages in an aim to optimise cleaning.

5.4.1 The effect of temperature

The effect of temperature on the cleaning rate is studied at constant velocity of 1.64 m s^{-1}. The experimental results are shown in Fig. 5.8. It shows that;

- increasing temperature of water clearly induces faster cleaning like the lab scale case. Very good 1st order removal is observed throughout the cleaning process as experimental data points are on a nearly straight line on logarithmic scale,
- unlike figures obtained from the lab scale experiments, different stages in the cleaning cannot be distinguished from the figure.

5.4.2 The effect of flow velocity

The flow effect on cleaning is studied at flow velocity values ranging from 0.89 to 2.05 m s^{-1} at 15 °C at the pilot scale. Fig. 5.9 clearly shows that;

- cleaning rate increases significantly with increasing flow velocity,
- again simple 1st order removal is observed for the overall cleaning process.

Experiments at pilot scale illustrate that relative effect of temperature and flow velocity on each cleaning stage cannot be identified by the used experimental method. Therefore, design of experiments method is used in the next section to study the effect of process variables on each cleaning stage.

5.5 Effect of process variables in each region of cleaning at pilot plant scale

Here, the aim is to find the degree to which cleaning of thin film of toothpaste depends on temperature and flow rate, how this dependence changes during cleaning and to find a better CIP protocol which will provide benefits in terms of time and energy consumption at pilot scale. This may allow further understanding of cleaning of deposits which show similar rheological behaviour to toothpaste.

5.5.1 Determination of cleaning times and energy consumption

The essential point is to know when the 2nd stage of cleaning starts during cleaning since in section 5.4, it could not be identified by the weight analysis method at the pilot scale. Therefore, visual observation and the online Optek turbidity meter are used. After the test section, the glass pipe is used to observe removal visually and as soon as the chunk removal stops, turbidity meter generally comes off saturation. Typical cleaning behaviour and cleaning regions are shown in Fig. 5.10, which displays the turbidity meter ppm response of cleaning behaviour at 70 °C and 11.2 m^3 h^{-1} water flow. Cleaning rate is very high initially thus turbidity meter is saturated up to 120 seconds. This region corresponds to 1st cleaning stage at which rapid removal of chunks is observed. Then, ppm response decreases exponentially until the end of cleaning, meaning that cleaning rate is decreasing. This stage is accepted as 2nd cleaning stage and lasts 100 seconds. A reading of 3 ppm on the turbidity meter is selected as the end-point of cleaning for proper comparison. The pipe is found to have only a few tiny islands left with 0.1 % of the starting weight remaining.

Once, the time is calculated for each cleaning region, energy consumption is calculated by the following expression:

$$E=(\rho g h_f/\epsilon + c_p \Delta T)\, Qt/3600 \qquad (5.3)$$

where E (MJ) is energy consumed, t (s) is the time spent during each region, ρ (kg m^{-3}) is density of water, Q (m^3 h^{-1}) is the volumetric flow rate, g (9.81 m s^{-2}) is the acceleration due to gravity, h_f (m) is the friction head loss component of the system, ϵ is pump efficiency, c_p (4.1855 kJ $kg^{-1}K^{-1}$) is heat capacity of water and ΔT (K) is temperature difference (temperature of cleaning water – datum temperature). The first term in the brackets in Equation (5.3) is the energy requirement for pumping and the other term is the energy consumed during cleaning. ϵ is found from the pump performance chart as 0.6. h is calculated as 30 m by finding the maximum flow rate that could be pumped around the system according to current rig configuration and pump capacity. Average ambient water temperature is~15 °C (datum temperature) and 20 °C is selected for the minimum temperature for experiments. Pumping energy consists of between 0.3 % and 5 % of the total energy consumption in cleaning experiments, therefore, it is found that heating of the water is more energy intensive process. Here, heating energy and pumping energy are considered together and minimisation in terms of total energy is studied.

5.5.2 Experimental design and statistical analysis

A five level, two variable central composite design (CCD) is used to determine relationship between the factors (temperature, flow rate) and the responses (time and energy). The range of flow rate and temperature are selected as 6.5-16 $m^3\ h^{-1}$ and 20-70 °C by considering the general CIP applications in plants. The CCD in the experimental design consists of ten factorial points and two replicates of the central point. The coded and actual levels of design and results are shown in Table 5.1. The model is explained by the following second degree polynomial equation:

$$y = b_0 + b_1T + b_2F + b_{11}TT + b_{22}FF + b_{12}TF \qquad (5.4)$$

y is the response function, b_0 is an intercept, b_{1-2}, b_{11-22} and b_{12} are the coefficients of the linear, quadratic and interactive terms, respectively. In addition, T and F represent the uncoded independent variables of temperature (°C) and flow rate ($m^3\ h^{-1}$), respectively. The fitted polynomial equation is expressed as surface plots in order to observe the relationship between the response and factors. According to the analysis of variance, the effect and regression coefficients of all terms are determined by using the Minitab 16 statistical software. P-values of less than 0.05 are accepted statistically significant.

5.5.3 Interpretation of the model fit

Table 5.2 and 5.3 shows the values of model coefficients and statistical parameters for the energy consumption and cleaning time for total cleaning, the 1st cleaning stage and the 2nd cleaning stage, separately and the effect of temperature and flow rate on these parameters. The statistical significance of the regression model and each coefficient are checked by p-values. Most importantly, these tables show that;

- determination coefficients (R^2) for the time and energy consumption are 0.964 and 0.938 when variables (temperature and flow rate) are fitted to the total cleaning process,
- however, when the variables are fitted to the 1st and 2nd cleaning stage separately, R^2 values for the time and energy consumption are observed to increase to 0.982 and 0.979, respectively for the 1st cleaning stage.

Therefore, the variance analysis reveals that the predictive model gives better description of the cleaning time and the energy consumption when the two cleaning stages are considered separately. This implies that the 1st and 2nd cleaning stages have different kinetics and different cleaning conditions should be applied in these stages to find an efficient CIP protocol.

Table 5.2 shows that, for modelling of the energy usage, only single effect of temperature is found as significant (p value for b_1 = 0.01) at the 1st stage and the sign in front of the coefficient of temperature is (+), which means that increasing temperature in the 1st cleaning stage will cause significant energy consumption. This implies that high temperature does not help cleaning of the deposit significantly in this stage. Neither terms are found as significant for the 2nd cleaning stage, which shows the complexity of the cleaning process in the 2nd cleaning stage.

According to statistical analysis, second degree polynomial model can adequately predict the cleaning time and the energy consumption as a function of applied temperature and flow rate of cleaning water at both stages since the p values for the model are 0.002 and 0.034 for the 1st and 2nd cleaning stages, respectively.

Table 5.3 shows that for modelling of the cleaning time, only single effects of temperature and flow rate are significant in both cleaning stages. The lower the p value, the more significant the variable. Therefore, the effect of flow rate is found more significant than that of temperature in the 1st cleaning stage (b_1=0.034,b_2=0.030) while reverse is observed for the 2nd cleaning stage (b_1=0.027,b_2=0.037). This reveals that flow rate is the dominating parameter in the 1st stage as generally removal of chunks is observed. However, for the 2nd stage, temperature is the controlling variable as adhesive forces should be overcome. Hence, the state or rheology of the deposit should be changed by increasing temperature to remove the deposit by fluid flow easily at this stage. However, quadratic and cross effects of these variables are found insignificant in both stages. It can be deduced from this finding that the cleaning process has some limiting factors, which means increasing flow rate and temperature more above some limit cannot help cleaning of the deposit significantly. In sections 5.6 and 5.7, response surface graphs are analysed to determine the best cleaning conditions for each cleaning stage.

5.6 Analysis of the 1st cleaning region in terms of cleaning time and energy consumption

Figure 5.11 shows the predictive cleaning times and energy usage obtained by varied flow rate and temperature for the 1st cleaning stage. These predictive graphs provide important clues about how effective each variable is in each region. For instance, according to Fig. 5.11 (a);

- high flow rates significantly decrease the cleaning time for the 1st stage and,
- at high flow rates (16 m^3h^{-1}), increasing the temperature has no considerable effect on the cleaning time.

This shows that flow rate is more important in the 1st stage where removal of chunks is observed, and high temperature does not significantly help to reduce the cleaning time in this stage as the same conclusion is obtained in the previous section by the statistical analysis. This is presumably due to the fact that cohesive bonds of the soil is weak enough to be broken by flow effect, thus high temperature is not needed to soften the soil more in this stage. Therefore, the use of high temperature at high flow rates will increase energy consumption and cause energy waste. Figure 5.11 (b) illustrates this perfectly. According to this figure;

- the energy usage in the 1st cleaning stage highly depends on the temperature and increase in temperature significantly increases the energy usage,
- using high flow rates causes decrease in the energy usage.

Therefore, according to predictive models in Fig. 5.11, the minimum energy consumption and cleaning time is found at the cleaning condition of 20 °C and 16 m^3 h^{-1} flow rate in the 1st cleaning stage.

5.7 Analysis of the 2nd cleaning region in terms of cleaning time and energy consumption

Figure 5.12 shows the predictive cleaning times and energy usage obtained by varied flow rate and temperature for the 2nd cleaning stage. According to Fig. 5.12 (a);

- temperature has a considerable impact on the cleaning time of the 2nd stage. At any flow rate, increasing temperature decreases the cleaning time significantly in this stage,
- increasing flow rate also induces reduction in the cleaning time at any temperature.

This shows that heat energy is needed for the removal of the deposit at the 2nd stage. The predictive energy consumption figure illustrates this point (Fig. 5.12 (b));

- although increasing temperature to 50 °C significantly increases the energy usage, increasing the temperature more above 50 °C results in dramatic fall in the energy usage especially at the highest flow rate. Hence, this shows the considerable contribution of temperature to the cleaning process at this adhesion controlling cleaning stage,
- increasing the flow rate always reduces the energy consumption at any temperature.

Therefore, for the 2nd cleaning stage, the minimum energy consumption and cleaning time is observed at 70 °C and 16 m^3 h^{-1} according to the surface response graphs in Fig. 5.12. The reason why flow rate and temperature are equally important in the 2nd cleaning stage may be that the deposit has strong adhesive bonds which should be overcome to achieve cleaning. Therefore, both high temperature and high flow rate are needed to soften and remove the soil in minimum time.

5.8 Comparison of the optimum CIP protocol with the traditional CIPs

The previous sections identify the best conditions for each cleaning stage. New experiments are conducted to validate the found conditions according to predictive models shown in Fig. 5.11 and 5.12, and to show the existence of an efficient CIP protocol. In the traditional CIP protocols, water at certain velocity and temperature is pumped for the whole cleaning process. Therefore, three main experiments are done;

i) the cold traditional CIP protocol: 20 °C water at 16 m^3 h^{-1} is used for the overall cleaning. A flow of high-velocity water at ambient temperature is often used in the prerinse stage of CIP operations.

ii) the hot traditional CIP protocol: 70 °C water at 16 m^3 h^{-1} is used for the overall cleaning which is generally applied in the industry.

iii) the optimum CIP protocol: water at 20 °C - 16 m^3 h^{-1} is used during the 1st cleaning stage and water at 70 °C - 16 m^3 h^{-1} is used during the 2nd cleaning stage. Here, the experiment is done by starting the cleaning process with the water flow at 20 °C - 16 m^3h^{-1}. When the turbidity meter comes off saturation, the pump is stopped immediately. Then, water at 70 °C - 16 m^3 h^{-1} is pumped straightaway to the system until the turbidity meter reaches to 3 ppm.

Figure 5.13 shows the turbidity readings from the cold, hot and the optimum CIP protocols. It illustrates that;

- comparable cleaning times are obtained in the hot CIP and the optimum CIP protocols, 100 s and 126 s, respectively. However, the cleaning time increases by more than 100 % (265 s) when the cold CIP is applied mainly due to the long cleaning time spent in the 2nd cleaning stage,
- in the optimum CIP protocol, water at 20 °C is applied up to 73 s at which the turbidity probe comes off saturation. After the application of water at 70 °C, the turbidity reading is saturated forthwith and during the time elapse between 73 and 106 s due to increase in the removal rate induced by the hot water. Then, a very quick 2nd stage is observed after 106th s (20 s), which validates the generated surface response models by showing the temperature sensitivity of this stage,

Figure 5.14 shows the effect of these three CIP protocols on the cleaning time and the energy consumption. It clearly shows that;

- the hot traditional CIP causes great reduction (over 75 %) in cleaning time compared to the cold traditional CIP protocol. This shows the advantage of applying hot and high velocity water in terms of increase in plant availability. However, the hot CIP would highly increase the carbon footprint as the energy consumption is four times higher than the cold CIP.
- On the other hand, the optimum CIP protocol notably results in 39 MJ energy saving (40 %) compared to the hot traditional CIP protocol and 55 % time saving compared to the cold traditional CIP protocol.

Therefore, the results have graphically demonstrated the importance of using relatively simple steps during cleaning in plants to minimise the environmental and economic impact. An alternative CIP protocol decreases the energy consumption by 40 %, while increasing the cleaning time by 25 % compared to the hot traditional CIP. However, optimisation is a complex process and depends on the industry. It needs a careful financial analysis which should take into account production needs, cleaning costs, plant availability, carbon emissions and associated taxes.

5.9 Conclusion

This chapter has analysed the cleaning stages (after the product recovery stage which is studied in Chapter 4) to understand how cleaning happens in pipes and identify any optimisation possibilities in the cleaning of pipes. Toothpaste is used as a model deposit. This chapter consists of three strands:

(i) Obtaining the cleaning rate data is invaluable to understand the cleaning process completely to make optimisation. Hence, the weight analysis method is used to monitor the cleaning rate during the cleaning of toothpaste, details of the method is explained in section 3.5.1. By the cleaning rate data, some similarities are observed between the cleaning behaviours of toothpaste and whey protein deposit although they are very different type of soils. This can be attributed to the two main reasons; (i) both are removed from top to bottom by chunks and (ii) have stronger adhesive forces than cohesive forces.

(ii) By doing lab scale experiments, it is identified that there are two distinct stages in the cleaning of toothpaste; 1st (film removal) and 2nd (patch removal) cleaning stage. About 95 wt % of toothpaste is removed and generally half of the total cleaning time is spent in the 1st cleaning stage. On the other hand, the cleaning rate is very low for the 2nd cleaning stage as only 5 wt % of the material is removed here within the other half of the total cleaning time. In the 1st stage, cohesive failure takes place since chunk removal is observed. However, for the 2nd stage, removal happens by adhesive failure as the deposit is observed to be eroded away gradually, no chunk removal is observed. Different kinetics are observed in these stages. In the 1st stage, temperature is found to be the dominating parameter whereas in the 2nd stage, both flow velocity and temperature have a significant effect on cleaning rates. These findings from lab scale experiments imply that optimisation can be made by applying different process conditions during the cleaning at pilot scale. It is very important since general practice in CIP systems is to circulate hot water rapidly from the start to the end of the cleaning process but for particular cleaning stages, hot water may not have considerable effect and this will cause waste of energy.

(iii) Pilot scale experiments are conducted to understand and optimise cleaning at higher scale. Design of experiments method is used to determine the effect of process variables in each cleaning stage. According to the statistical analysis, it is found that temperature has a negligible effect on the cleaning time in the 1st stage while flow velocity has a pronounced effect in both stages. Thus, a two step CIP protocol is proposed; application of cold water (20 °C) in the 1st stage and hot water (70 °C) in the 2nd stage at the same flow rate (16 m^3 h^{-1}). Finally, the proposed CIP protocol is compared with the traditional CIP protocols. The proposed CIP protocol results in considerable energy saving (40 %) without causing significantly longer cleaning times compared to the traditional CIP protocol.

In the cleaning of toothpaste, fluid mechanical removal is the dominating process rather than the dissolution or chemical reactions since chemicals are not used during the cleaning process and toothpaste is removed by the fluid action alone. In the cleaning of this type of deposits, deposit rheology and flow velocity will probably determine the cleaning behaviour and time (Fryer and Asteriadou, 2009). This means the results found here for toothpaste might well be applicable or related to the removal of other similar deposits. Thus, Chapter 6 investigates the effect of deposit rheology on the cleaning process.

Tables Chapter 5

Table 5.1: Central composite design for the independent variables (actual levels) and the obtained results.

Run order	**Experiment number**	**Temperature (°C)**	**Flow rate (m^3 h^{-1})**	**Cleaning times (s)**		**Energy consumption (MJ)**	
				1st region	2nd region	1st region	2nd region
6	1	20	11.20	275	495	18.75	33.75
7	2	27	7.86	352	941	39.36	105.22
2	3	27	14.63	130	270	27	56.08
9	4	45	6.50	300	487	68.55	111.28
3	5	45	11.20	164	174	64.86	68.81
10	6	45	11.20	174	186	68.51	73.24
5	7	45	16.00	85	100	47.81	56.25
1	8	63	7.86	244	216	107.33	95.02
4	9	63	14.63	80	52	65.5	42.58
8	10	70	11.20	120	60	86.31	43.16

Table 5.2: Model coefficients and statistical parameters of the second degree polynomial model for the energy usage.

Term	**Energy usage (total cleaning)**		**Energy usage (the 1st stage)**		**Energy usage (the 2nd stage)**	
	Coefficient	p	Coefficient	p	Coefficient	p
Regression	-	0.015	-	0.002	-	0.034
b_0	69.26	0.619	-99.36	0.099	168.8	0.162
b_1	8.33	0.037	4.54	0.01	3.79	0.141
b_2	-16.8	0.367	8.74	0.216	-25.54	0.113
b_{11}	-0.06	0.069	-0.02	0.096	-0.041	0.092
b_{22}	0.59	0.434	-0.28	0.317	0.866	0.169
b_{12}	-0.14	0.372	-0.12	0.066	-0.014	0.901
R^2	0.938		0.979		0.908	

Table 5.3: Model coefficients and statistical parameters of the second degree polynomial model for the cleaning time.

Term	**Cleaning time (total cleaning)**		**Cleaning time (1^{st} region)**		**Cleaning time (2^{nd} region)**	
	Coefficient	p	Coefficient	p	Coefficient	p
Regression	-	0.006	-	0.001	-	0.013
b_0	4615.11	0.005	954.49	0.003	3660.62	0.009
b_1	-65.13	0.019	-9.99	0.034	-55.14	0.027
b_2	-366.05	0.025	-63.35	0.030	-302.7	0.037
b_{11}	0.27	0.151	0.05	0.148	-0.22	0.201
b_{22}	8.08	0.132	1.19	0.207	6.9	0.163
b_{12}	2.38	0.052	0.24	0.201	2.13	0.060
R^2	0.964		0.982		0.944	

Figures Chapter 5

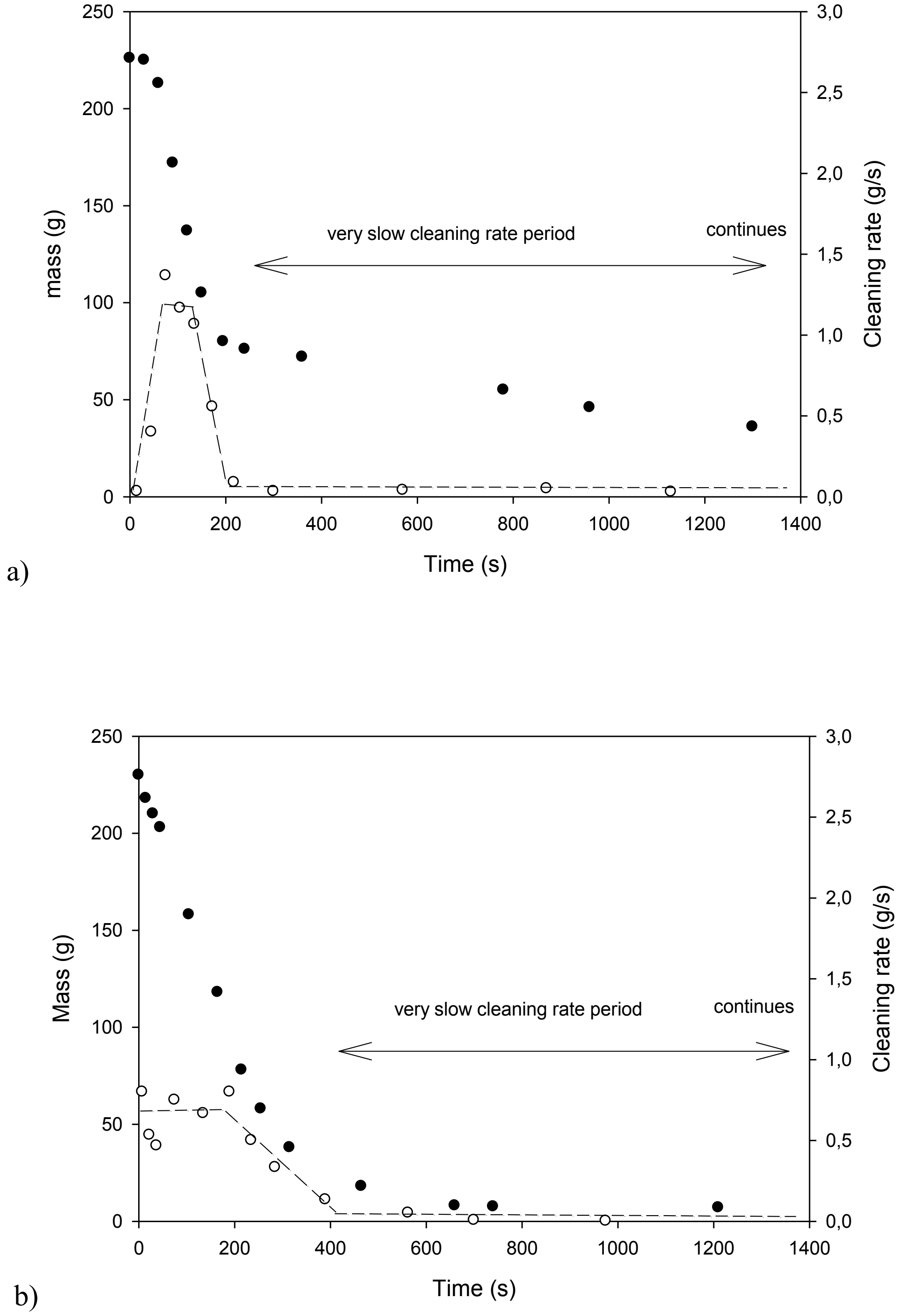
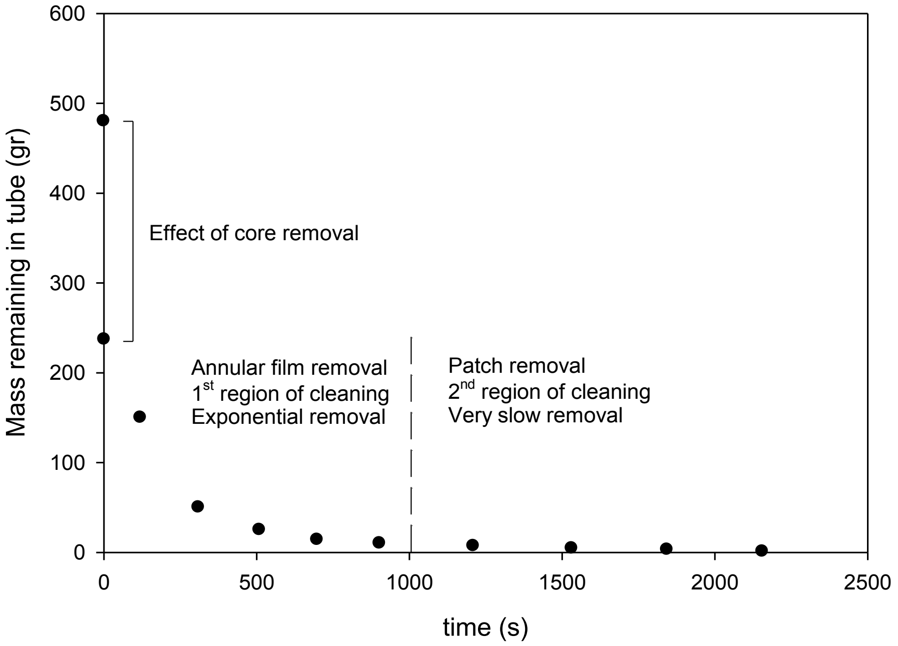


Figure 5.1: Typical mass removal (solid symbols) and cleaning rate (open symbols) data of toothpaste at (a) 50 °C and 0.28 m s^{-1} water velocity (b) 15 °C and 0.55 m s^{-1} water velocity at laboratory scale (0.0239 m ID, 1 m pipe).



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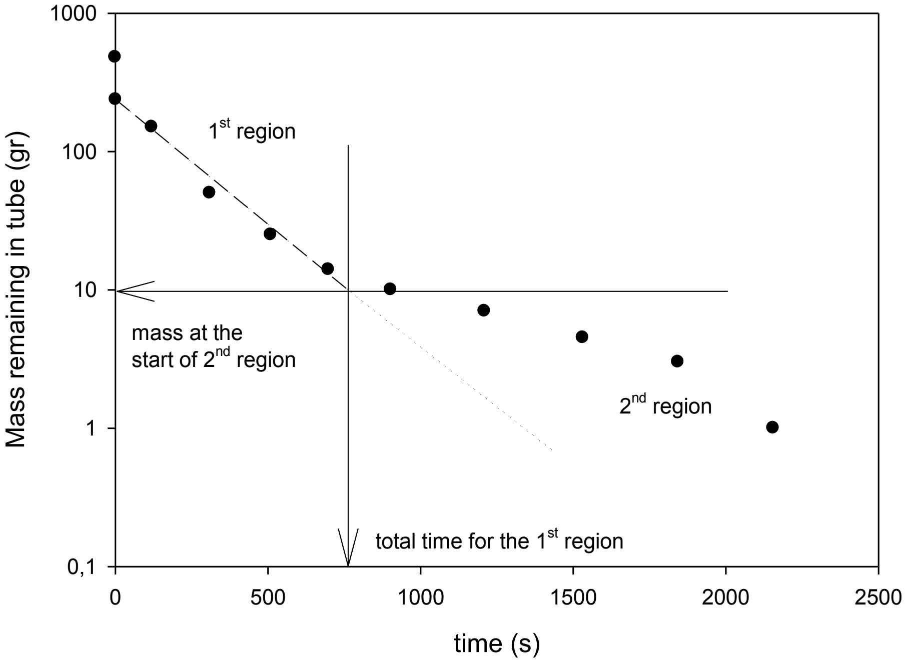


b)

Figure 5.2: Weight of toothpaste in the lab scale pipe during cleaning with water at 0.55 m s^{-1} and 15 °C, showing three stages of the cleaning process; a very short core removal process followed by an exponential removal of a thin film, followed by slow removal of small islets. Figure is plotted on a) linear scale and b) log scale.

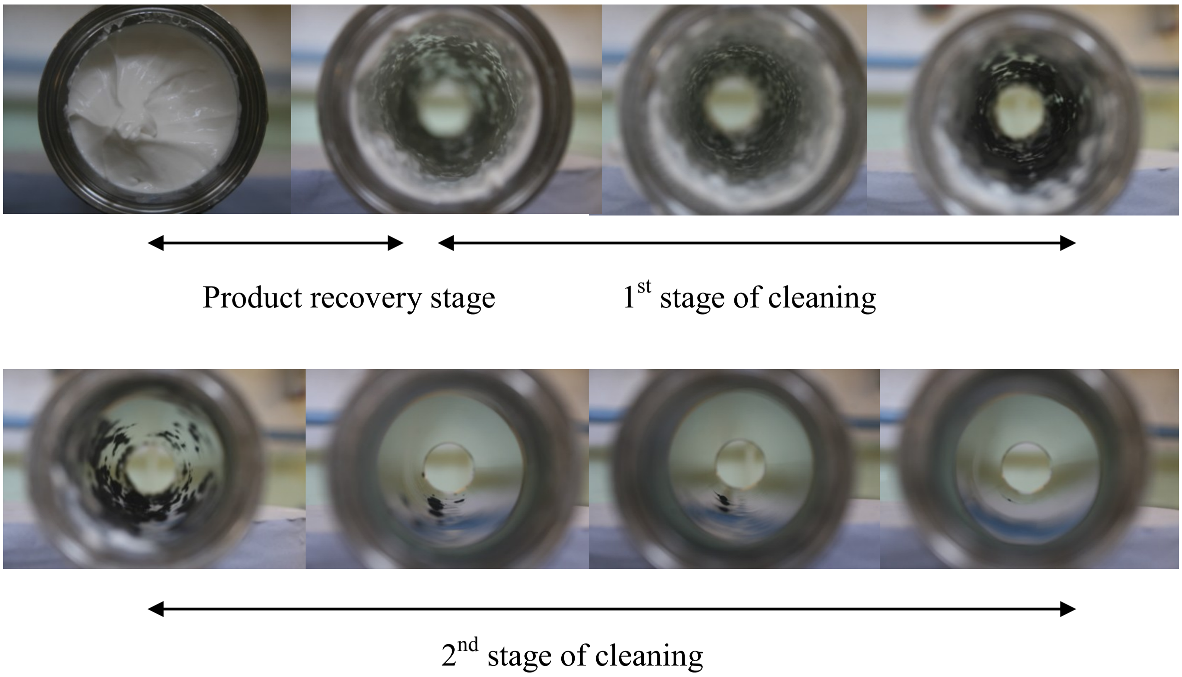


Figure 5.3: Pictures of the inside of the pilot scale pipe during the cleaning process. Cleaning conditions are 1.64 m s^{-1} and 15 °C, showing three stages of the cleaning process; a very short core removal process followed by a removal of a thin film, followed by a slow removal of small islets. Times, when pictures were taken, were 60, 180, 300, 510, 540, 600 s after the product recovery stage.

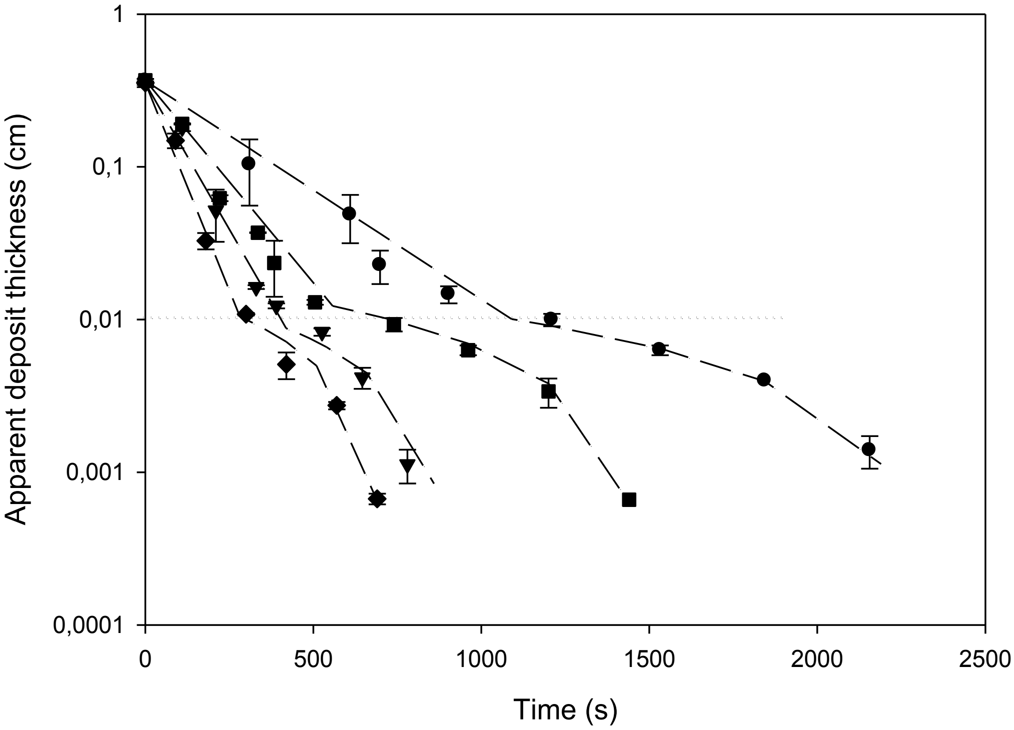


Figure 5.4 Cleaning profiles of the thin film of toothpaste at different temperatures. The product recovery is carried out at 0.55 m s^{-1} and 15 °C. Cleaning water is used at 15 °C (circles), 30 °C (squares), 40 °C (triangles) and 50 °C (diamonds) at 0.55 m s^{-1}. Line shows the start of the 2nd region. Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.

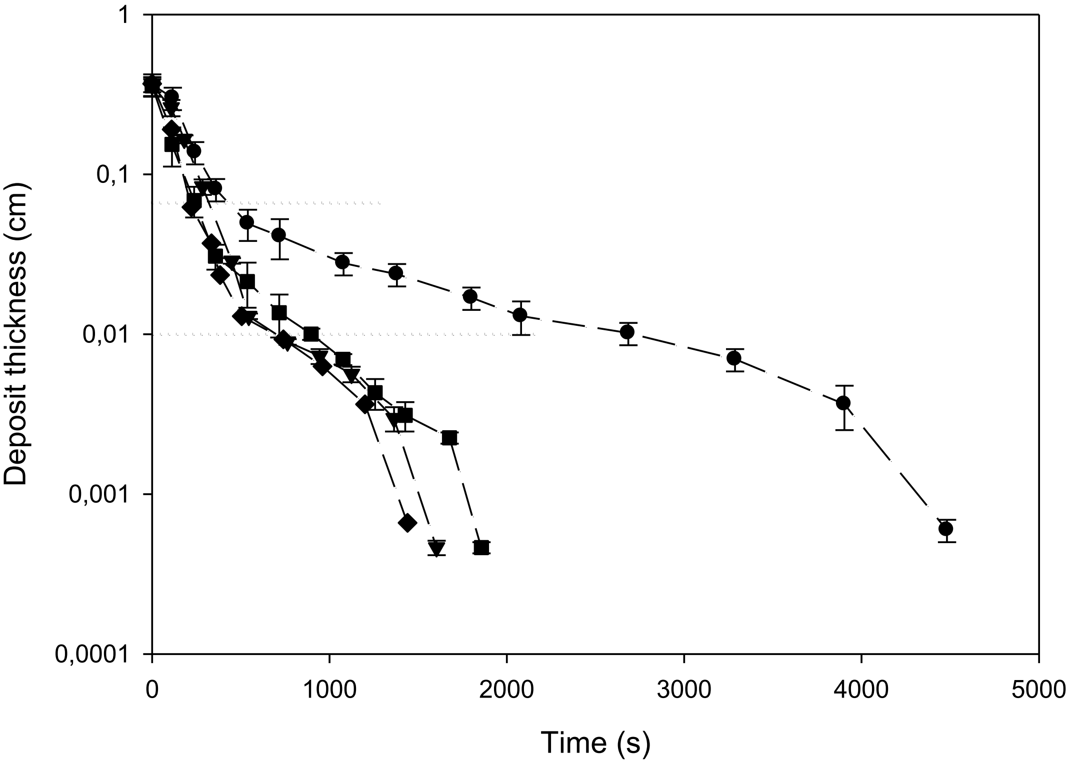


Figure 5.5: Cleaning profiles of the thin film of toothpaste at different velocity of cleaning water. The product recovery is carried out at 0.55 m s^{-1} and 15 °C. Cleaning is performed at 30 °C and velocities of 0.38 (circles), 0.45 (squares), 0.5 (triangles), 0.55 m s^{-1} (diamonds). Lines show the start of the 2nd region. Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.

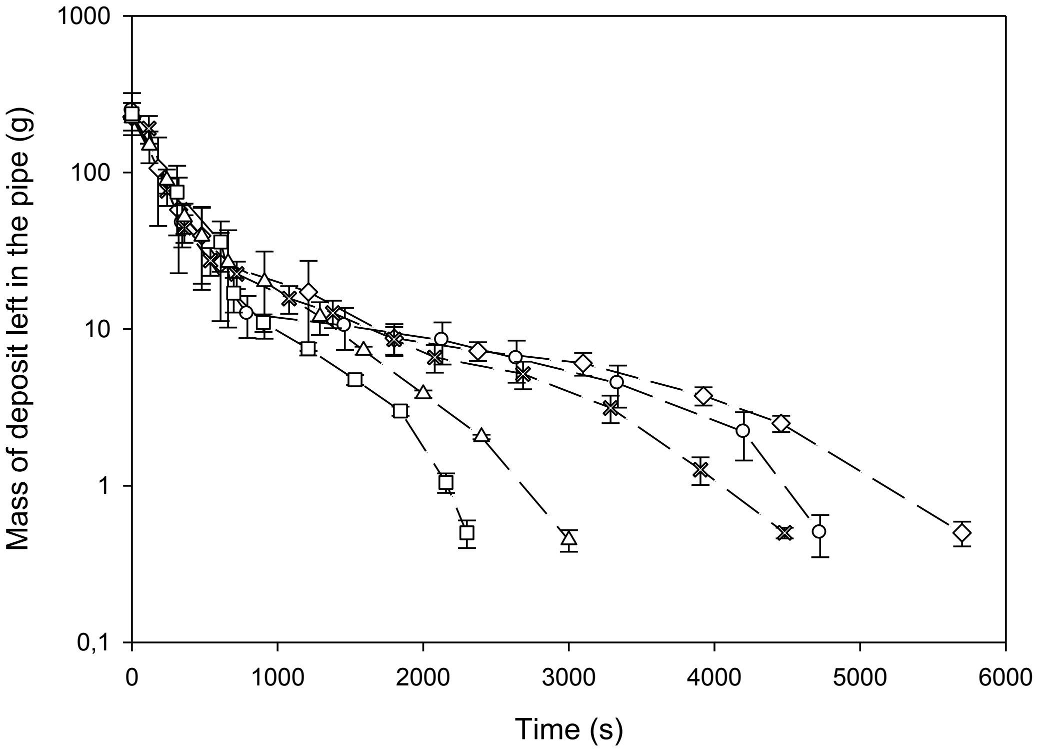


Figure 5.6: Cleaning profiles of the thin film of toothpaste at same Reynolds number of 12000. The product recovery is carried out at 0.55 m s^{-1} and 15 °C. Cleaning is performed at 15 °C - 0.55 m s^{-1} (squares), 20 °C - 0.48 m s^{-1} (triangles), 30 °C - 0.38 m s^{-1} (crosses), 40 °C - 0.31 m s^{-1} (circles) and 50 °C - 0.28 m s^{-1} (diamonds). Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.

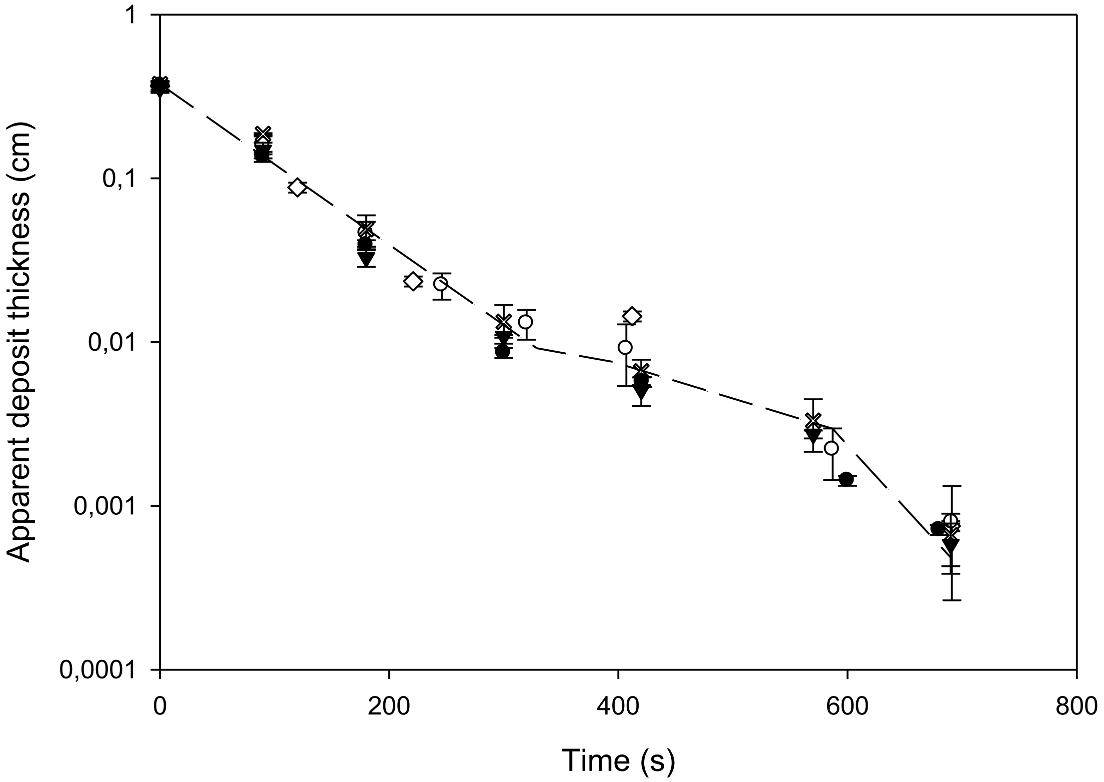


Figure 5.7: Cleaning profiles of the thin film of toothpaste from 0.5 m. (circles), 0.8 m. (crosses), 0.88 m. (diamonds) and 1 m. (triangles) pipe at 50 °C, 0.55 m s^{-1} of cleaning water. The product recovery is carried out at 0.55 m s^{-1} and 15 °C. Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.

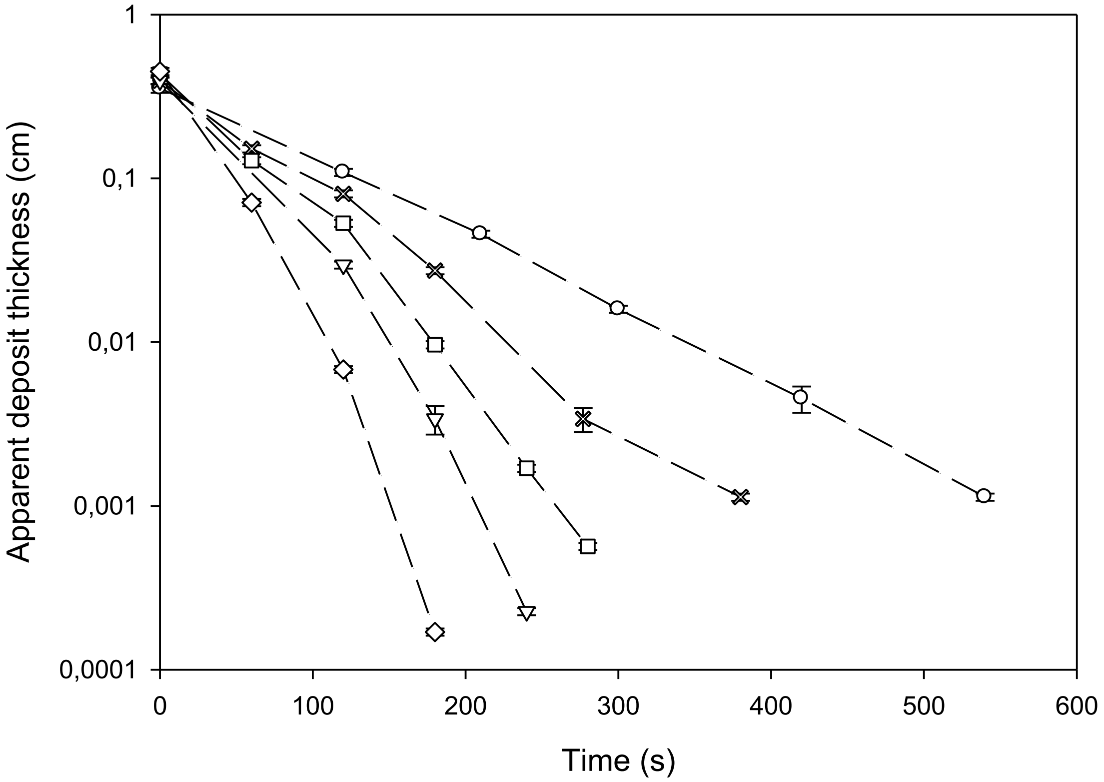


Figure 5.8: (a) Cleaning profiles of thin film of toothpaste at different temperatures of 15 (circles), 20 (crosses), 30 (squares), 40 (triangles) and 50 °C (diamonds). Cleaning water is used at 1.64 m s^{-1} in 0.5 m, 0.0477 m ID pipe. The product recovery is carried out at 1.7 m s^{-1} and 15 °C. Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.

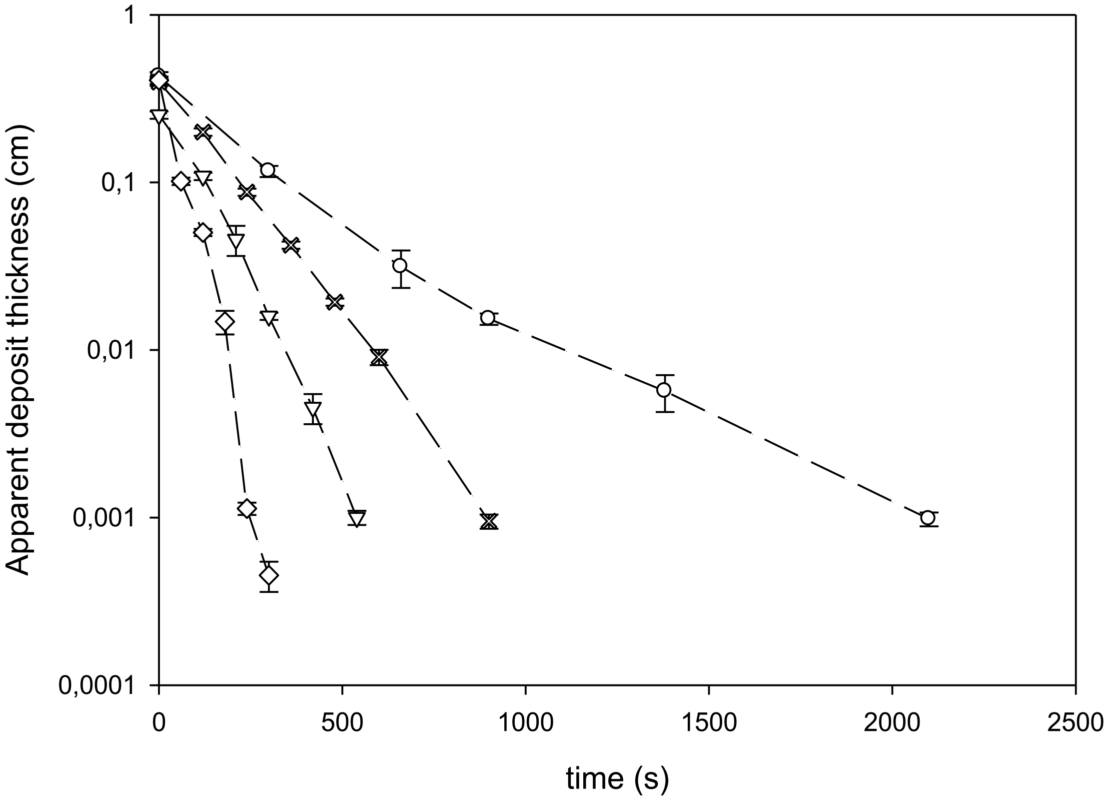


Figure 5.9: Cleaning profiles of thin film of toothpaste at different velocity of cleaning water. Cleaning is performed at 15 °C and velocities of 0.89 (circles), 1.37 (crosses), 1.64 (triangles), and 2.05 m s^{-1} (diamonds). The product recovery is carried out at 1.7 m s^{-1} and 15 °C. Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.

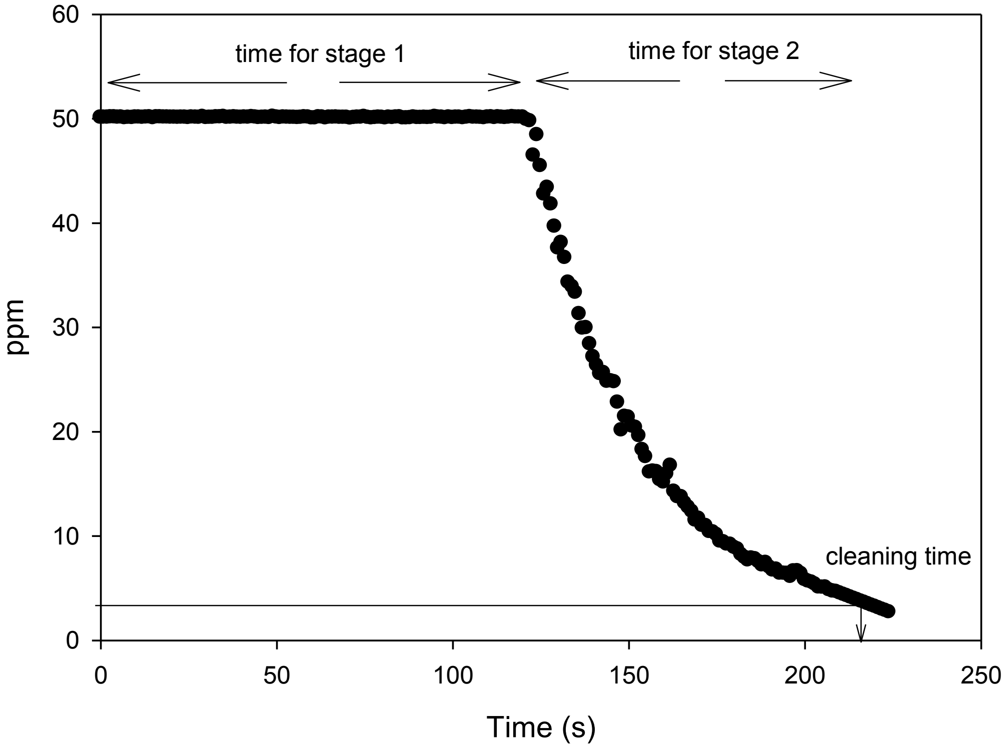
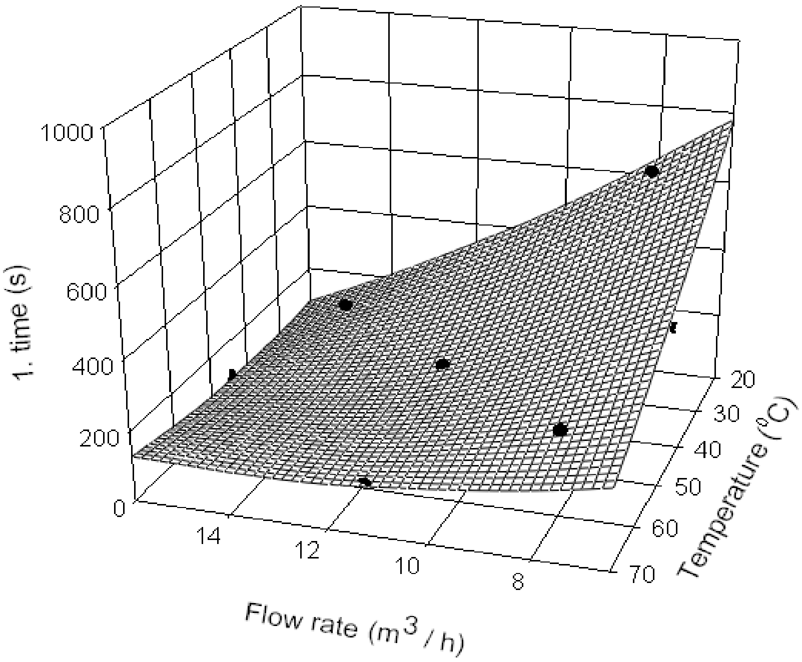


Figure 5.10: Typical turbidity reading during the cleaning of toothpaste at 70 °C and 11.2 m^3 h^{-1}. Cleaning rate is high in the film removal (1st cleaning) stage thus turbidity meter saturates up to 120 s.



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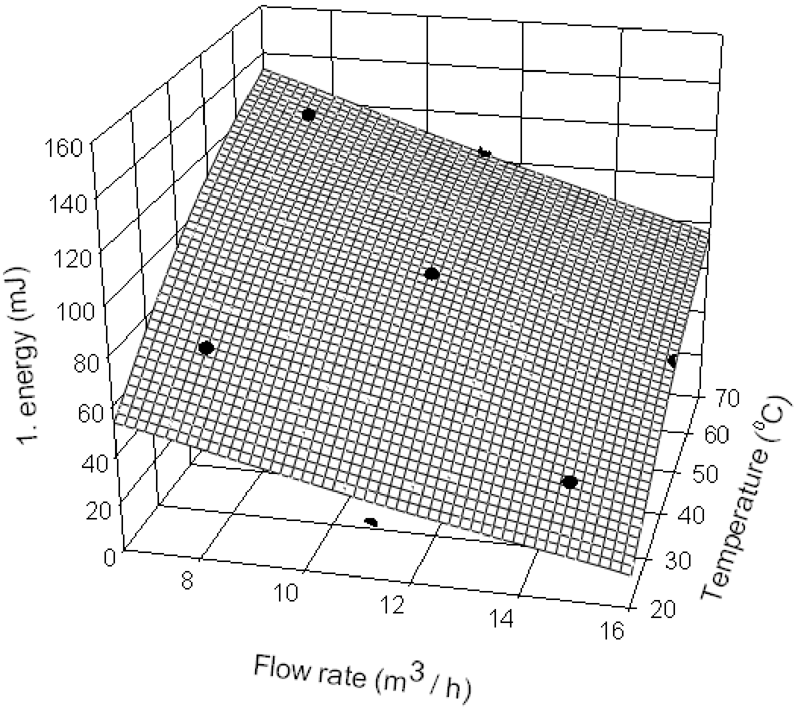
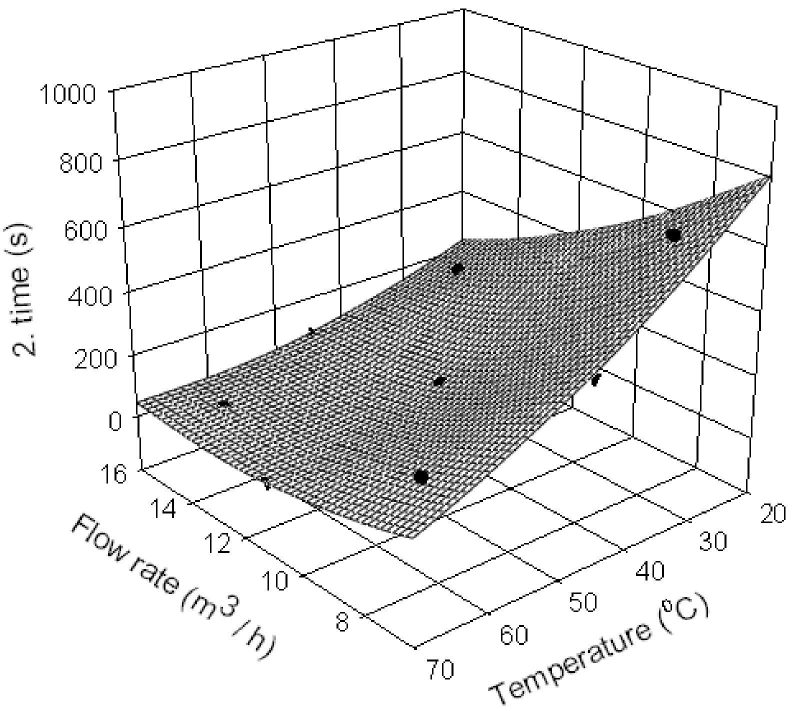


b)

Figure 5.11: Predictive response surfaces and experimental results obtained as a function of temperature and flow rate. (a) Spent time in the 1st stage which ends when the turbidity meter unsaturates. (b) Energy consumption for the 1st cleaning stage.



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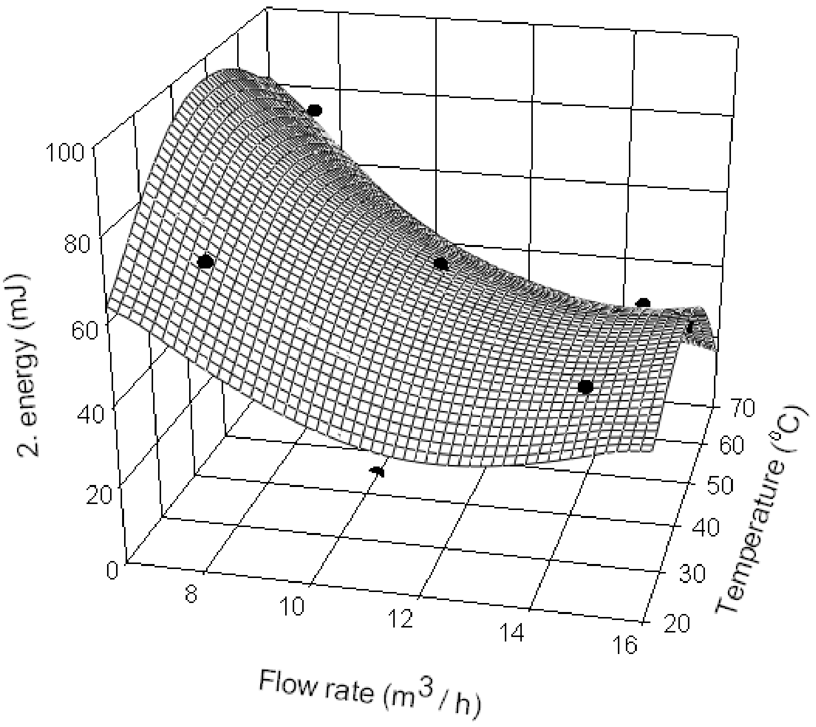


b)

Figure 5.12: Predictive response surfaces and experimental results obtained as a function of temperature and flow rate**.** (a) Spent time in the 2nd stage which starts after the turbidity meter unsaturates. (b) Energy consumption for the 2nd stage.

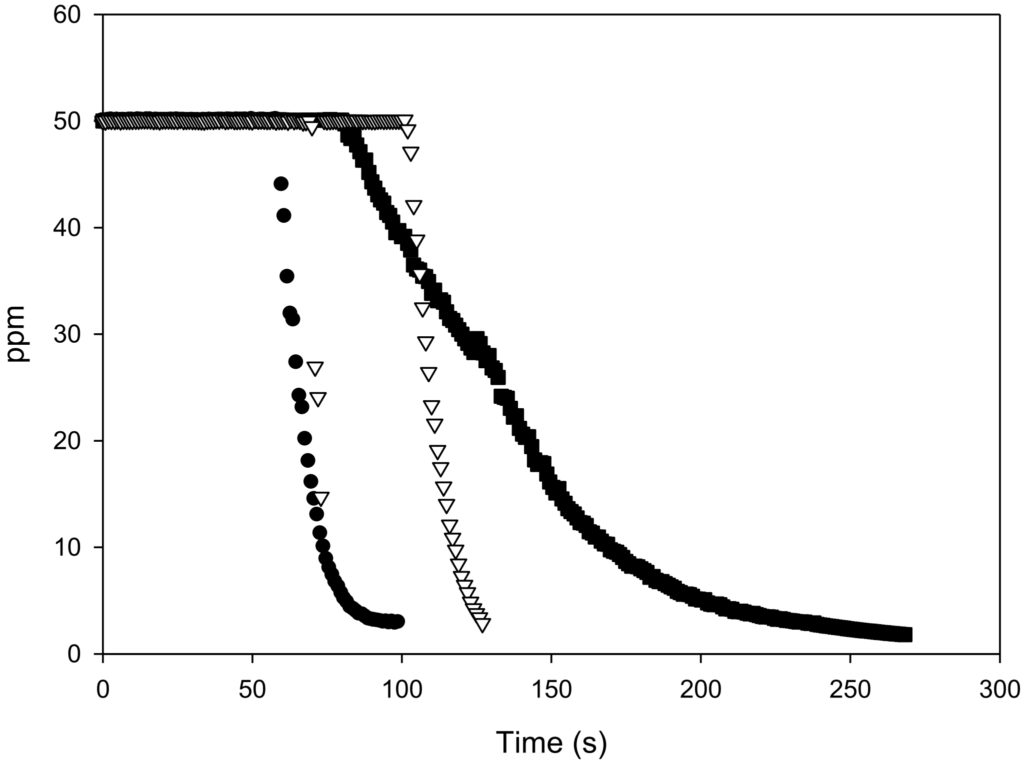


Figure 5.13: Turbidity readings from the 3 different CIP protocols. Flow rate is 16 m^3h^{-1} in all systems. Water is used at 20 °C (squares) in the cold CIP protocol. Water is used at 70 °C (circles) in the hot CIP protocol. Water is used at 20 °C up to 73 s and then water at 70 °C is used until the end of cleaning (triangles) in the optimum CIP protocol.

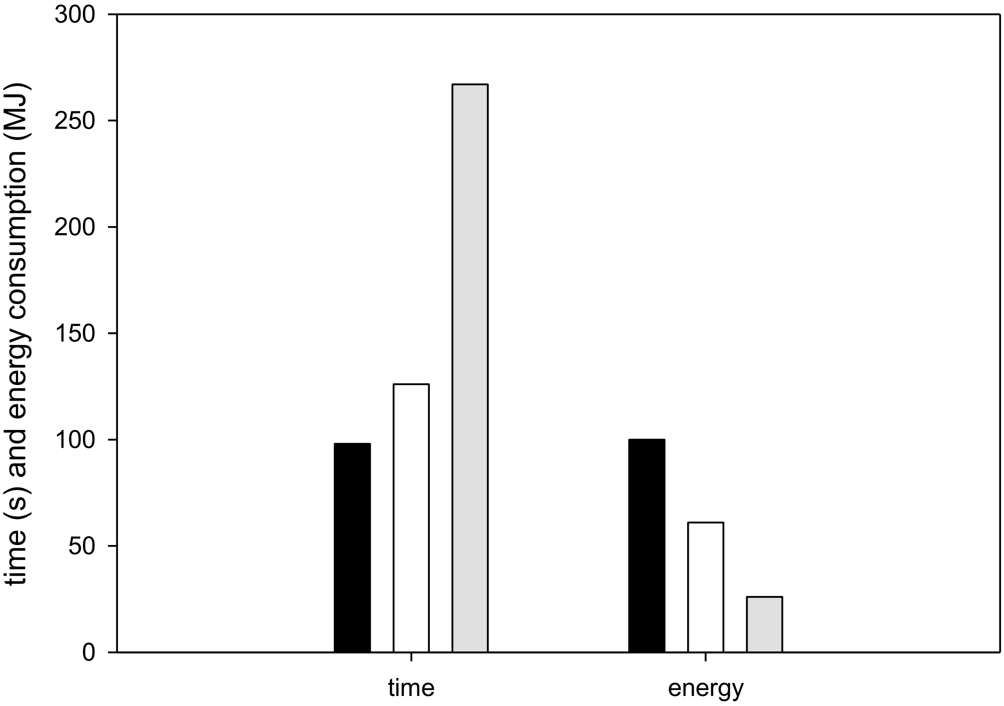


Figure 5.14: Time and energy consumption during the three different CIP protocols. Flow rate is 16 m^3 h^{-1} in all systems. The hot traditional CIP protocol is shown in black, the optimum CIP protocol in white, and the cold traditional CIP protocol in grey.

Chapter 6: THE EFFECT OF DEPOSIT RHEOLOGY ON THE CLEANING PROCESS

Chapter 4 and 5 have identified the cleaning behaviour and stages of a Herschel-Bulkley type of deposit (toothpaste) from pipes thoroughly, however, it is not known yet whether other fluids show the same cleaning behaviour or not. Therefore, in this chapter, the cleaning of various simple deposits is studied in order to improve understanding of cleaning by examining the relation between deposit rheology and its cleaning.

For other type of deposits, the effect of fluid flow may not be enough so dissolution or diffusion might be the controlling mechanism in the cleaning process. In some systems, both physical and chemical cleaning process take place to remove deposit from a surface (Fryer et al., 2006). Therefore, for the cleaning of all types of deposits, there should be a relationship between fluid mechanical parameters and deposit properties, probably determining the dominating cleaning mechanism. A relationship may be found that can relate the cleaning behaviour of numerous deposits.

The main aim of the work presented here is to quantify the effect of deposit properties on cleaning through studying the effect of deposit rheology on cleaning time and behaviour. For this purpose, various food and personal care products are cleaned from fully filled pipes. The deposit types selected could all be removed with water alone to simplify the problem.

Nonetheless, the findings here may provide better understanding of cleaning mechanisms of all types of deposits.

6.1 The effect of deposit rheology on cleaning time

Cleaning of thirteen different materials from a pilot scale straight pipe is investigated at 1.5 m s^{-1}, 20 °C. As shown in section 3.4, the materials show different rheologies: Newtonian, Herschel-Bulkley and shear thinning behaviour. Toothpaste is diluted with water to give different weight percentages soils with different rheologies. In order to relate their cleaning time with their rheologies, cleaning times of materials are plotted against their viscosity and yield stress values separately. As mentioned in section 3.5.2, the Optek turbidity probe is used here to find cleaning times. Rheological types and parameters of materials and obtained cleaning time results are shown in Table 6.1.

Figure 6.1 (a) shows the cleaning time of each material vs. its viscosity. A shear rate of 15 s^{-1} is chosen to rank the materials in the order of their viscosity values. As can be seen in Figure 3.4 (b), the order of materials is not changed between the shear rate range of 10 - 100 s^{-1}. Cleaning times with respect to material viscosity reveals a scatter of data points, which does not show any clear relationship:

- For instance, although glucose syrup has the highest viscosity value among the studied materials (70 Pa s), its removal time from the pipe is only 25 ± 5 s. This very quick cleaning time is not due to dissolution since glucose syrup is very sticky material. And, it takes at least 300 s to dissolve it in water by rigorous manual mixing although this mixing is more applied shear than in pipe flow.

- Hand cream has a moderate viscosity value of 12 Pa s but has the highest cleaning time of 1200 ± 150 s.

Cleaning times with respect to material yield stress shown in Figure 6.1 (b) shows a good linear relationship except for hand cream cleaned at 20 °C (open circle symbol). However, hand cream cleaned at 70 °C (rectangle open symbol) appears to fit this linear relationship. This implies that the effect of temperature in cleaning might be to decrease deposit yield stress. Figure 6.1 (b) shows that yield stress of deposits is one of the governing parameters which determine cleaning time. However, cleaning of hand cream and toothpaste should be investigated further in order to determine why the cleaning time of hand cream is high despite having a reasonably low yield stress compared to toothpaste.

6.2 Comparison between the cleaning behaviours of hand cream and toothpaste

Figure 6.2 (a) shows a 3D mesh plot of the cleaning time with temperature and flow velocity for hand cream and toothpaste. It is clear that at most of the cleaning conditions hand cream is cleaned in a shorter time than toothpaste which is expected result according to the found trend in Fig. 6.1 (b). However, at low velocities and low temperatures, hand cream has higher cleaning times than toothpaste.

The weight analysis method is done to determine the mass fraction of materials left on the pipe wall with time at a low velocity to further understand the higher cleaning time of hand cream. Figure 6.2 (b) shows a comparison between removal behaviour of hand cream and toothpaste at 1 m s^{-1}, 20 °C. This figure interestingly illustrates that;

- in the 1st cleaning stage (up to 1000 s), the cleaning of hand cream is much faster than that of toothpaste, as expected from the yield stress values of the two materials,
- however during the 2nd cleaning stage (> 1000 s), the cleaning rate of hand cream is significantly slower than toothpaste, causing a longer cleaning time.

In the 2nd cleaning stage, the erosion of thin islands takes place as identified in Chapter 5. Water may not wet the hand cream properly due to the oily nature of hand cream thus this may cause slower removal overall. In order to investigate wettability issue, contact angle measurements were made of a water droplet on (i) hand cream, (ii) undiluted toothpaste and (iii) 80 % diluted toothpaste at 20 °C. This was done by capturing an image of the side-view of the water droplet and material under a microscope. The angle between the water droplet and air was determined using the angle function in Image J. The same amount of material was placed on a glass slide and levelled out using a film thickness gauge (supplied by GSK, UK) to give a smooth surface. The contact angle for each material was approximately (i) 18° for hand cream, (ii) 23° for toothpaste and (iii) 24° for 80 % diluted toothpaste. Similar results suggest that it is not a material wettability issue in the 2nd cleaning stage. However, this problem is overcome by increasing velocity in the cleaning of this type of deposit. As a result, yield stress is found to be the main relevant rheological parameter in the cleaning of materials which could be cleaned by fluid action alone.

6.3 The effect of deposit rheology on the cleaning behaviour

The effect of deposit rheology on the cleaning behaviour of deposits is compared for three materials with three different rheologies shown in Figure 6.3 (a): shampoo (shear thinning), glucose syrup (Newtonian) and 70 % diluted toothpaste (Herschel-Bulkley). Figure 6.3 (b) shows oscillatory stress sweeps of these deposits. Glucose and shampoo are predominantly viscous. Up to a shear stress of 50 Pa, toothpaste is predominantly elastic (G'>G"). As the maximum achievable shear stress in this study is 25 Pa, this means only the 70 % diluted toothpaste remains predominantly solid during cleaning.

Figure 6.4 (a) shows the mass fraction of materials left in the pipe at specific times during cleaning:

- Around nine-tenths of the total amount of shampoo and glucose syrup in the pipe is removed in a very short time i.e. in the product recovery stage (studied in Chapter 4). However, the amount of 70 % diluted toothpaste removed from the pipe wall after the product recovery is significantly less than the other materials (eight-tenths). This might be the result of its elastic behaviour in the applied shear stress range. Also, this finding has an industrial value that product recovery of shear thinning and Newtonian fluids can be achieved with high yield.
- Out of these three materials 70 % diluted toothpaste has the longest cleaning time. This is due to the existence of a very slow patch removal stage observed between cleaning at 280 to 600 s.
- A shear thinning deposit (shampoo) is cleaned faster than a Newtonian deposit (glucose syrup). This may be due to the decreasing viscosity of the shampoo with increased shear.

It is clear from these findings that deposits can be grouped into two of removal modes: (i) zero-yield stress deposits and (ii) yield stress deposits. These two removal modes are depicted in Figure 6.4 (b) according to visual observations:

i) Zero-yield stress deposits are removed by shearing. They simply flow out of the pipe, thus their removal is quicker and probably a function of viscosity. A typical patch removal stage is not observed in the cleaning of these deposits.

ii) On the other hand, yield stress deposits essentially support shearing forces without being flowed by these forces and are removed by fracturing initially and finally slow erosion. This removal mode of yield stress deposits shows similarities with that of other more complex soils such as whey protein, milk, mineral deposits and Biofilms (Christian, 2003, Bird and Fryer, 1991, Gillham et al., 1999, Goode, 2011) as illustrated in section 5.1. This might be due to the fact that all of these deposits show solid like behaviour rather than fluid behaviour. Yield stress value would be important for this group of materials as in materials science, yield stress means the lowest stress that produces a permanent deformation in a material then at some point fracture occurs without applying high stress.

6.4 The effect of process parameters on cleaning time

So far yield stress is found to be the key rheological parameter here to affect cleaning. As well as velocity of cleaning water, temperature of the water is also important in affecting cleaning time. Therefore, understanding the relationship between bulk forces (velocity and temperature) and intrinsic forces (yield stress) in affecting cleaning time might allow us to understand current cleaning and predict cleaning time of another material not yet characterised. For this purpose an experimental programme is designed to find the effect of yield stress, temperature and flow velocity on cleaning time by the most efficient use of data.

6.4.1 Experimental design and statistical analysis

Toothpaste is diluted to produce different weight % concentrations with different yield stress values. A five level, three variable central composite design (CCD) is used. The 16 designed experiments, experimental cleaning times and predicted cleaning times by the model equation (Equation 6-1) are shown in Table 6.2. According to surface response analysis (which explores the relationships between several explanatory variables and one response variables) 2nd degree polynomial model is used:

$$t = b_0 + b_1Y + b_2T + b_3F + b_{33}F^2 + b_{12}YT + b_{13}YF + b_{23}TF \qquad (6.1)$$

t (s) is the cleaning time, b_0 is an intercept, $b_{1\text{-}2\text{-}3}$, b_{22} and $b_{12\text{-}13\text{-}23}$ are the coefficients of the linear, quadratic and interactive terms, respectively. In addition, Y, T and F represent the uncoded independent variables of yield stress (Pa), temperature (°C) and flow rate (m^3h^{-1}), respectively. Note that quadratic terms of yield stress and temperature are not used in the model as the variance analysis reveals that this predictive model gives the best description of the cleaning time as a function of variables with an R^2 of 0.953. According to the analysis of variance, the effect and regression coefficients of all terms are determined by using Minitab 16 statistical software. P-values of less than 0.05 are accepted to be statistically significant.

6.4.2 Interpretation of the surface plots

The fitted polynomial equation is expressed as surface plots in order to observe the relationship between the response and factors. Figure 6.5 (a) shows the effect of yield stress and flow rate on cleaning time at 45 °C and 6.5 (b) shows the effect of yield stress and temperature on cleaning time at 11.2 m^3h^{-1}.

Figure 6.5 illustrates that;

- for deposits having low yield stress, increasing velocity and temperature has a negligible impact on cleaning time. Therefore, energy and water should not be wasted in the cleaning of low yield stress deposits by applying high velocity water at high temperature,
- for high yield stress deposits, overall cleaning time is a strong function of temperature and velocity of cleaning fluid.

Figure 6.5 (b) interestingly shows that the cleaning time decreases with similar slopes when temperature increases and yield stress decreases, which suggests a close interrelationship between yield stress and temperature. Therefore, this finding is further investigated by the statistical analysis of the results.

6.4.3 Interpretation of the model fit

Table 6.3 shows the values of model coefficients and statistical parameters for the cleaning time and the effect of the yield stress, temperature and flow velocity on these parameters. The statistical significance of the regression model and each coefficient are checked by the p-values ($p \leq 0.05$ is chosen as a significance level). This table graphically shows that;

- the coefficients for yield stress and temperature are very close to each other, 12.273 and -11.989, respectively. This closeness and signs in front of the coefficients imply that an increase in cleaning time would be similar when yield stress increases or temperature decreases.

- although single effects of yield stress and flow velocity are found as significant (p values = 0.000, 0.033), that of temperature is found insignificant (p value = 0.077). This importantly indicates that temperature is not an independent factor like yield stress and flow velocity since temperature also changes the yield stress of the deposit,
- the coefficient of interactive term of “yield stress*temperature” is very close to zero (-0.098). This indicates that the cleaning time would not be affected when yield stress and temperature are increased at the same time.

All of these findings suggest that cleaning time might only be a function of yield stress and flow velocity since temperature affects cleaning by changing the yield stress of a deposit. In order to confirm this argument, the weight analysis method is used to further investigate the relationship between temperature and yield stress.

6.5 A relationship between yield stress and temperature

In order to assess the individual effects of yield stress of a deposit and temperature of cleaning water on the cleaning process, the cleaning of undiluted toothpaste at different temperatures is compared with the cleaning of diluted toothpaste. Firstly, rheology tests are performed to identify yield stress of the tested toothpaste samples, which are;

1) (undiluted) toothpaste at 15 °C,
2) 60 % diluted toothpaste at 20 °C,
3) (undiluted) toothpaste at 30 °C,
4) 90 % diluted toothpaste at 20 °C,
5) (undiluted) toothpaste at 50 °C,
6) 80 % diluted toothpaste at 20 °C.

Figure 6.6 shows yield stress of the tested deposits obtained by the oscillation test.

- According to Figure 6.6 (a), undiluted toothpaste at 15 °C has the highest yield stress (203 ± 5 Pa) while the 60 % diluted toothpaste at 20 °C has the lowest yield stress (35 ± 1 Pa) amongst the studied deposits.
- Figure 6.6 (b) shows that yield stress of undiluted toothpaste at 30 °C is slightly higher (146 ± 4 Pa) than that of 90 % diluted toothpaste (126 ± 4 Pa) at 20 °C.
- However, comparable yield stress values are observed in Figure 6.6 (c) for the undiluted toothpaste at 50 °C (75 ± 2 Pa) and 80 % diluted toothpaste (73 ± 2 Pa) at 20 °C.

After the identification of the yield stress values, the cleaning of these deposits from fully filled pipe is investigated by the weight analysis method in order to compare their cleaning behaviours. Cleaning is performed at the corresponding temperatures at which yield stresses are determined. Also, the same product recovery conditions are used due to the results found in Chapter 4.

Figure 6.7 shows the mass of deposits left on the pipe wall during the cleaning process. It is observed that yield stress values and cleaning profiles match well: The higher the yield stress of a deposit, the slower the cleaning rate and cleaning takes longer time. Moreover, similar removal trends are observed for;

- 90 % diluted toothpaste cleaned at 20 °C and undiluted toothpaste cleaned at 30 °C, and
- 80 % diluted toothpaste cleaned at 20 °C and undiluted toothpaste cleaned at 50 °C.

Especially for the latter group of deposits, cleaning profiles appear to lie on almost one line in the 2nd cleaning stage and comparable cleaning times are obtained for them. This result is in accordance with the rheology results as very close yield stress values are found for these deposits (can be seen in Figure 6.6 (c)). In order to analyse how cleaning of these deposits happens in the fully filled pipes, cleaning profiles of 80 % diluted toothpaste cleaned at 20 °C and undiluted toothpaste cleaned at 50 °C are plotted on both log and linear scale in Figure 6.8. It graphically shows that;

- substantially less amount of material is left (125 g – 22 wt % of the initial amount) for 80 % diluted toothpaste compared to undiluted toothpaste after the product recovery stage. The product recovery conditions are the same, hence, this finding strongly suggests that yield stress is also the key factor which influences the amount of product recovered at the product recovery stage. Therefore, yield of product recovery will be poor for deposits which have high yield stress,
- the amount of deposit left after the product recovery stage does not affect the cleaning time. The cleaning of fully filled pipes is controlled by the cleaning of patches which are present in the 2nd cleaning stage (this stage is previously identified in Chapter 5) and comprises ca. 2 % (ca. 10 g) of the total mass in the pipe (ca. 450 g).
- interestingly 2nd cleaning stage of both deposits starts at similar times (ca. 270 s) with similar amounts (ca. 10 g), and the cleaning rates at this stage are observed to be identical. Hence, they are cleaned at similar times. This can be because the yield stresses of both deposits become similar during the cleaning process as shown in Fig 6.6 (c).

All of these investigations have illustrated that the effect of cleaning water temperature on cleaning is to decrease the yield stress of the toothpaste deposit.

6.6 A dimensionless number – the effect of yield stress and flow velocity on cleaning

In the previous section, the cleaning time is found to be essentially a function of yield stress and velocity and not temperature. Consequently, many attempts are made to make Equation 6.1 simpler by only a function of velocity and yield stress. Flow rate is converted to velocity by considering 0.0477 m ID pipe. Figure 6.9 (a) shows a plot of cleaning time vs. ratio of velocity^2 to yield stress of the deposit. In this graph, cleaning times are predicted by Equation (6.1) by inputting different yield stress and flow velocity values into the equation. Temperature is input as 20 °C in order to exclude its effect on yield stress. It is clear in Figure 6.9 (a) that the cleaning times appear to lie on almost one line.

It can also be anticipated that the cleaning time would decrease with increasing density of a cleaning fluid as well as velocity. For instance, if air was used instead of water to clean the deposit at the same velocity, cleaning would not be achieved. Therefore, a new dimensionless number can be formed as shown in Equation 6.2:

$$(v^2 \rho)/\tau \qquad (6.2)$$

where v is flow velocity (m s^{-1}), ρ is density (kg m^{-3}) of the cleaning fluid and τ is yield stress of a deposit (kg m^{-1} s^{-2}). A plot of cleaning time vs. the dimensionless number is shown in Figure 6.9 (b) including experimental data in Fig. 6.1 (b) and chapter 5. The cleaning time data collapse onto a one curve for different deposits with an R^2 of 0.955 when they are plotted against this dimensionless number. Equation 6.3 shows the relationship found with the cleaning time (s):

$$\text{cleaning time} = 54800\,(v^2\rho/\tau)^{-1.5};\ R^2 = 0.955 \qquad (6.3)$$

Since the term in the numerator of the dimensionless number (Equation 6.2) is similar to wall shear stress in Equation 2.2, an alternative approach is to make the system dimensionless using the ratio of wall shear stress to yield stress of the materials. Figure 6.9 (c) illustrates the relationship between the cleaning time data and this number. Slightly a better fit is observed as shown in Equation 6.4:

$$\text{cleaning time (s)} = 6.17\,(\tau_w/\tau)^{-1.5};\ R^2 = 0.958 \qquad (6.4)$$

Both dimensionless numbers can be used to describe the system in one scale. However, they have different physical meanings as can be understood from their magnitudes and only one of them should be related with the mechanical cleaning process. Therefore, cleaning at different scales will be investigated in Chapter 7 to identify the related variables in detail.

6.7 Discussion

The dimensionless number (Equation 6.2) can be physically interpreted as a ratio of a kind of flowing fluid energy to the internal forces (yield stress or cohesive/adhesive bonds) of a deposit. Therefore, this finding suggests that physical phenomena behind the cleaning might be that the energy produced by flowing water due to flow turbulence and/or friction is also dissipated by the breakage of cohesive and adhesive bonds of a deposit, and yield stress is a good predictor of the magnitude of these bonds. Hence, the effect of flow turbulence and friction will be further investigated in Chapter 7.

Here simple soils which can be removed by water alone are studied and the found dimensionless number can quantify the effect of properties of simple soils on cleaning.

Nevertheless, the cleaning mechanisms of complex soils may also be identified by this number if yield stress term is replaced with the cohesive or adhesive forces term in the found number. For instance, the cleaning of complex soils which need chemicals to be cleaned may depend on dissolution process as well as fluid mechanical process because the magnitude of cohesive and adhesive bonds is probably much higher than that of the soils studied here. Since complex soils have strong bonds, high values in the denominator make the number very low. Therefore, it can be hypothesized that there may be a critical threshold in this number under which dissolution process might dominate the cleaning and above which fluid mechanical removal might control the cleaning. Supportively in literature, Christian (2003) visually observed that whey protein deposit was removed fluid mechanically or chemically depending on the chemical concentration, temperature and flow rate of the cleaning solution used. When the flow rate was low, whey protein deposit was always cleaned dominantly by the chemical removal (dissolution) and when it was high, the cleaning generally happened by fracturing of the deposit by the fluid action. Similarly, Xin et al. (2002) found that dissolution-based cleaning model equation was no longer applicable when the Reynolds number was above 20,000 for their system. Schöler et al. (2012) also realised that dissolution was the governing mechanism in the cleaning of starch deposit, however, Weibull parameter, which was defined as a kind of indicator of a cleaning mechanism, changed at velocities above 1.5 m s^{-1} for their system. The change in this parameter at the high velocity might indicate that the cleaning mechanism changed into fluid mechanical removal.

The relationship between cleaning time and the dimensionless number (Equation 6.3) also suggests that if dissolution does not take place in the cleaning of any deposit which has very strong adhesive or cohesive bonds, completely clean state cannot be attained because the cleaning time goes to infinity as this dimensionless number approaches to zero (see Figure 6.9

(b)). This is encountered in daily life such that milk deposits, which are strongly adhered to surfaces, cannot be cleaned completely even by the application of high mechanical force unless chemicals are used.

6.8 Conclusion

Cleaning of various food and personal care products are studied in an aim to investigate the effect of deposit rheology on the cleaning process. Cleaning behaviours are analysed by the weight analysis method. The previously found (in Chapter 5) two distinct cleaning regions are observed in the cleaning of yield stress deposits. Therefore, yield stress fluids behave like a solid during cleaning and they are removed by fracturing and slow erosion. Whereas, Newtonian and shear thinning deposits simply flow out from the pipe during cleaning. Shear thinning deposit is cleaned earlier than Newtonian deposit. However, it takes much longer time to clean yield stress deposits.

It is found that yield stress is the critical rheological parameter and the cleaning time is proportional to yield stress. A crossover point of elastic and viscous modulus obtained by stress sweep oscillation test is used to find yield stress value of deposits. Also, yield stress has a large impact on the amount of product recovered; the lower the yield stress of a deposit, the higher the amount recovered in the product recovery stage.

A new dimensionless number is found which is a ratio between flowing energy of a cleaning fluid and the yield stress of a deposit. It successfully collapses cleaning time data onto a one curve for different deposits cleaned at different velocities. It suggests that there is a close relationship between the yield stress of a deposit and its cleaning.

Tables Chapter 6

Table 6.1: Rheology type, viscosity, yield stress and cleaning time of materials tested at 20 °C. Values for hand cream tested at 70 °C are also shown.

Material	**Material Type**	**Yield stress (Pa)**	**Viscosity (Pas) at 15 s^{-1}**	**Cleaning time (s)**
Shower gel	Herschel-Bulkley	23 ± 1	3 ± 0.1	80 ± 10
Glucose	Newtonian	0	70 ± 2	25 ± 5
Hand cream	Herschel-Bulkley	165 ± 4	12 ± 1	1200 ± 150
Yoghurt	Herschel-Bulkley	14.5 ± 0.5	1.7 ± 0.1	14 ± 5
Apple sauce	Herschel-Bulkley	12 ± 0.5	8 ± 0.5	12 ± 5
Shampoo	Shear thinning	0	6.2 ± 0.5	20 ± 5
100% toothpaste	Herschel-Bulkley	203 ± 5	24 ± 1	1000 ± 100
90% toothpaste	Herschel-Bulkley	126 ± 4	16 ± 1	650 ± 50
80% toothpaste	Herschel-Bulkley	73 ± 2	6 ± 0.1	430 ± 40
70% toothpaste	Herschel-Bulkley	52 ± 2	5.8 ± 0.1	220 ± 25
60% toothpaste	Herschel-Bulkley	35 ± 1	4 ± 0.1	130 ± 15
50% toothpaste	Herschel-Bulkley	13 ± 0.5	2 ± 0.1	25 ± 5
Hand cream at 70 °C	Herschel-Bulkley	26 ± 1	0.8 ± 0.1	100 ± 15

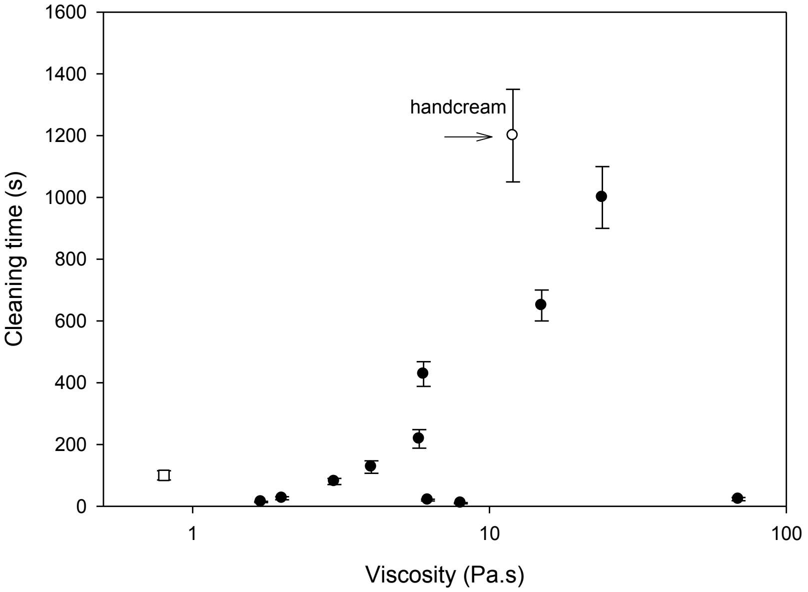
Table 6.2: Central composite design for the independent variables (actual levels) and the obtained and predicted cleaning time results.

Run	**Temperature (°C)**	**Flow rate ($m^3 h^{-1}$)**	**Yield Stress (Pa)**	**Weight % concentration**	**Experimental cleaning time (s)**	**Predicted cleaning time (s)**
1	60	8.5	32	58	100	76
2	60	14	126	90	65	29
3	45	11.2	79	83	170	172
4	30	8.5	126	90	720	683
5	20	11.2	79	83	340	301
6	30	8.5	32	58	135	203
7	60	8.5	126	90	220	281
8	45	11.2	158	100	340	301
9	45	16	79	83	75	53
10	45	11.2	79	83	165	172
11	60	14	32	58	20	88
12	30	14	126	90	158	214
13	45	11.2	0	40	10	-6
14	70	11.2	79	83	85	39
15	30	14	32	58	30	0
16	45	6.5	79	83	465	437

Table 6.3: Model coefficients and statistical parameters of the second degree polynomial model for the cleaning time.

Term	**Cleaning time**		
	Coefficient	p	F values
Regression	-	0.000	23.16
b_0	964.182	0.038	-
b_1 (yield stress)	12.273	0.000	32.54
b_2 (temperature)	-11.989	0.077	4.13
b_3 (velocity)	-133.663	0.033	6.65
b_{33} (velocity*velocity)	3.318	0.138	2.71
b_{12} (yield*temperature)	-0.098	0.009	11.81
b_{13} (yield*velocity)	-0.498	0.011	10.97
b_{23} (temperature*velocity)	1.289	0.027	7.35
R^2	0.953		

Figures Chapter 6



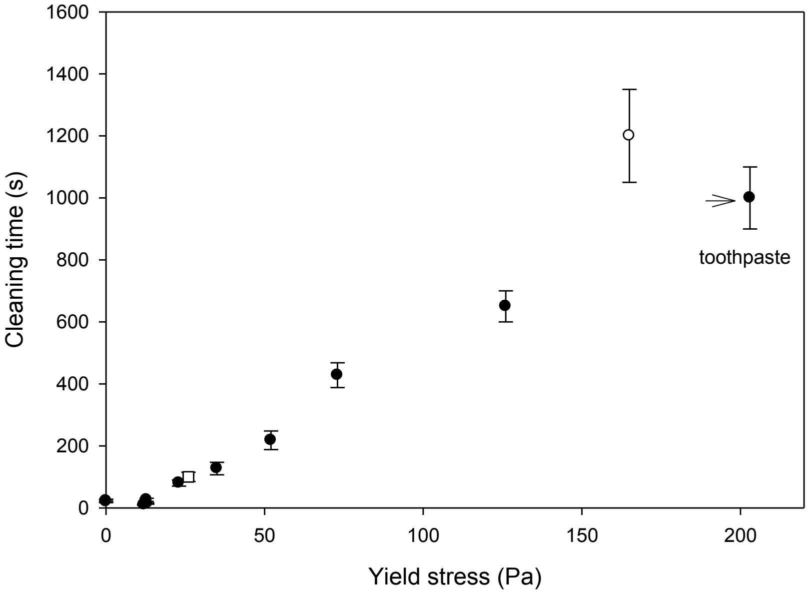
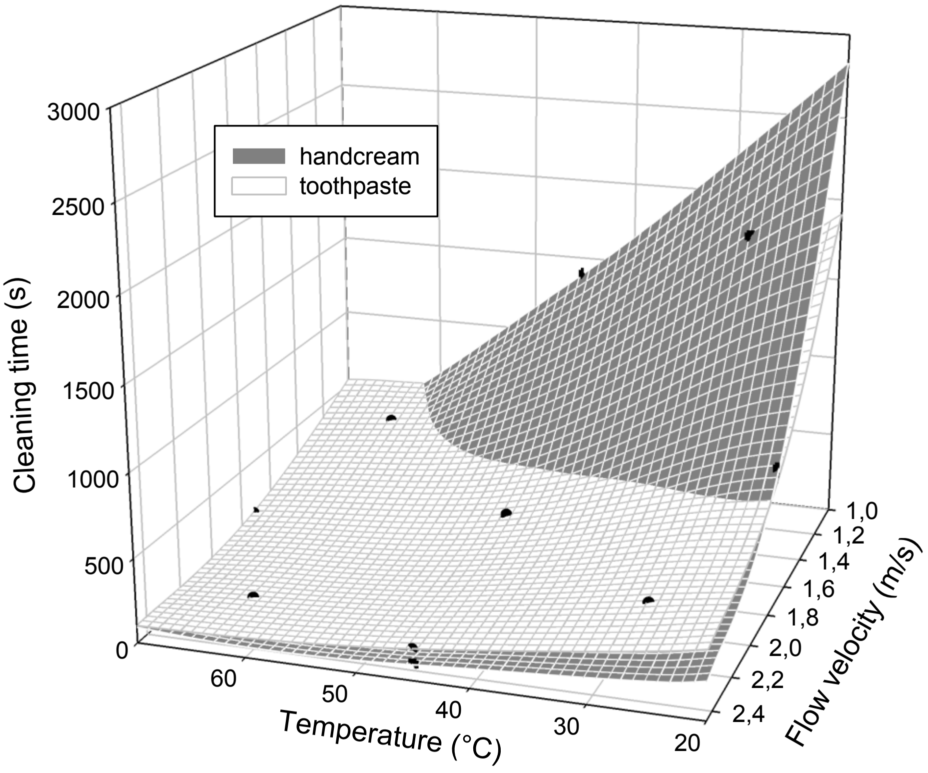
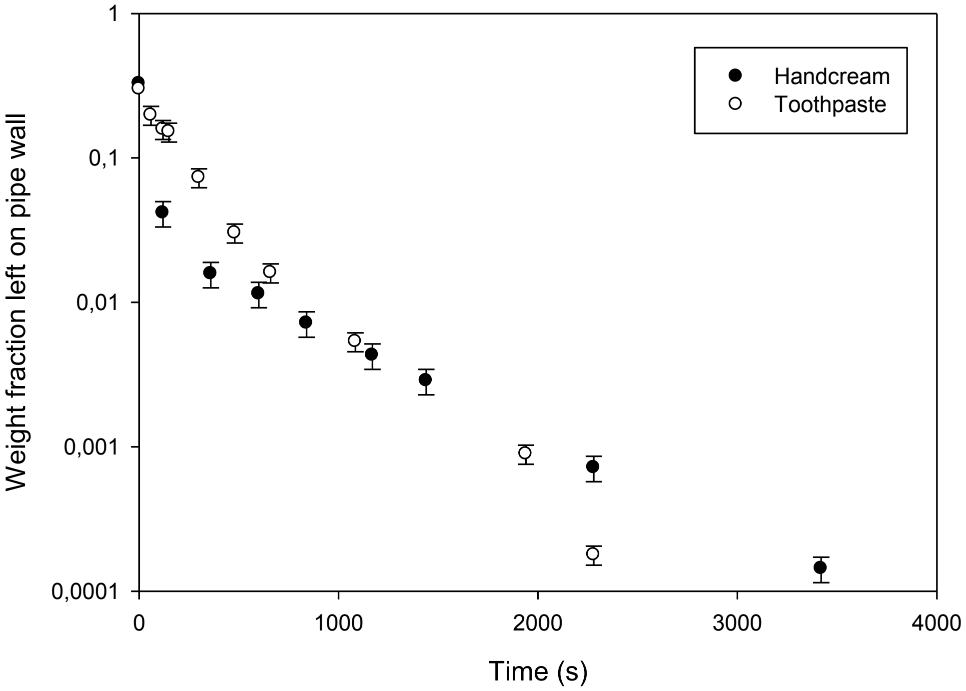


Figure 6.1: (a) Cleaning time vs viscosities of deposits at a shear rate of 15 s^{-1}. (b) Cleaning time vs yield stress of deposits. Cleaning was done at 1.5 m s^{-1} at 20 °C from 0.0477 m diameter pipe at pilot scale. Yield stress and viscosity values used in the graphs were obtained at 20 °C. Handcream was also cleaned at 70 °C. Thus, cleaning time was matched by its viscosity and yield stress values found at 70 °C (open rectangle symbols).



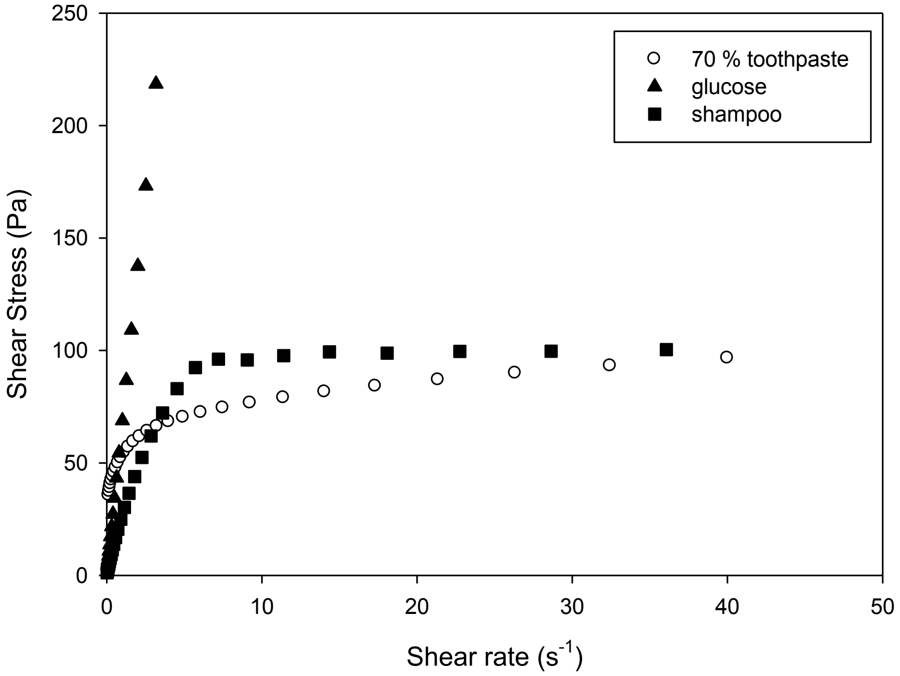
a)

Figure 6.2: (a) 3D mesh plot of hand cream and toothpaste showing the effect of temperature and velocity of cleaning water on cleaning time. Cleaning was performed at pilot scale and cleaning time was determined by the turbidity probe. Product recovery was done at 1.7 m s^{-1} and 20 °C for all experiments.

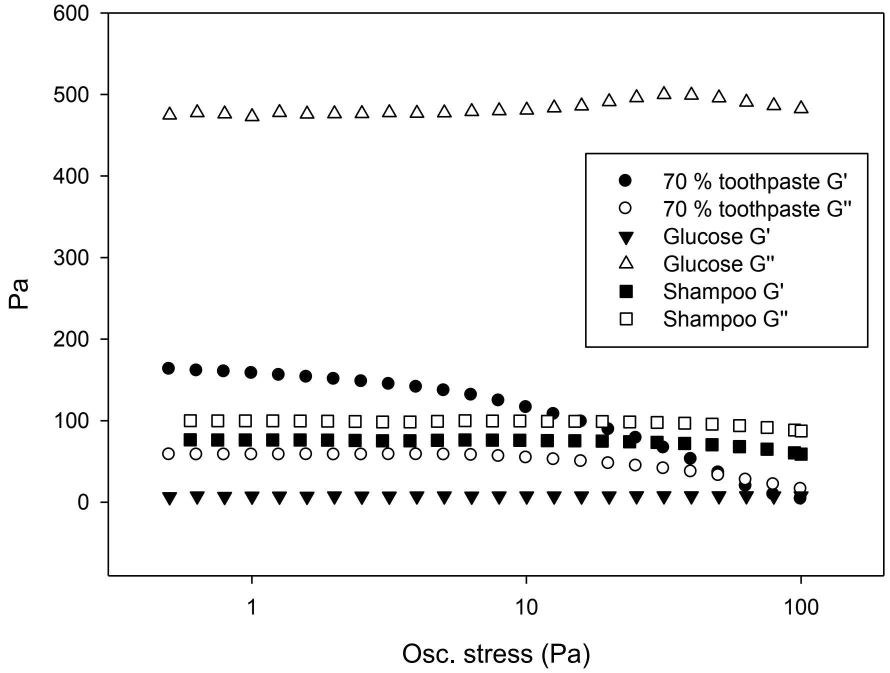


b)

Figure 6.2: (b) Graph showing removal behaviour of toothpaste and hand cream after core removal. Mass fraction left vs time data was obtained from cleaning at 1 m s^{-1} and 20 °C from the 0.0477 m diameter pipe at pilot scale.



a)



b)

Figure 6.3: (a) Shear rate ramp of deposits. (b) Stress sweep oscillatory test of deposits. Both of them are performed at 20 °C.

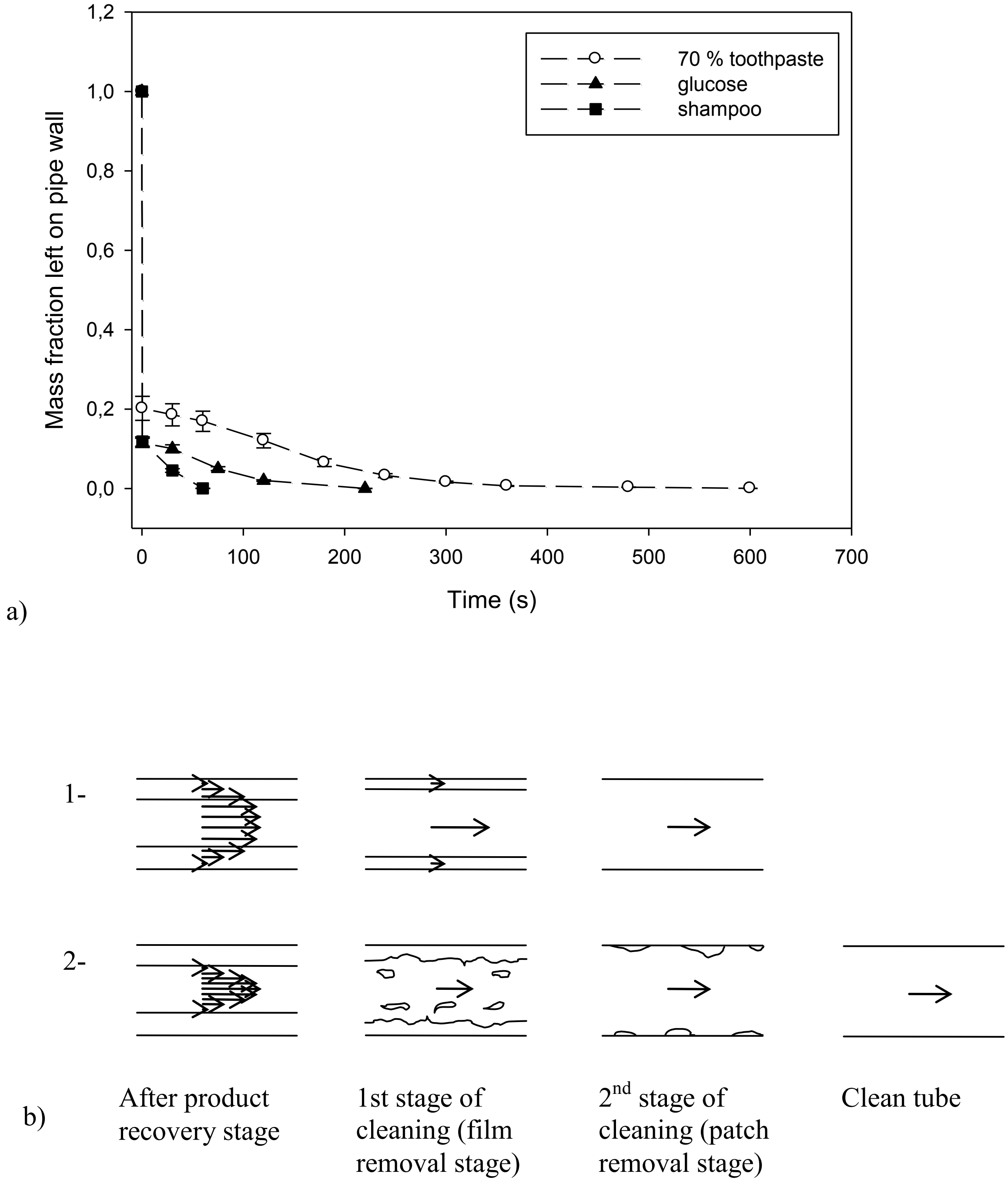
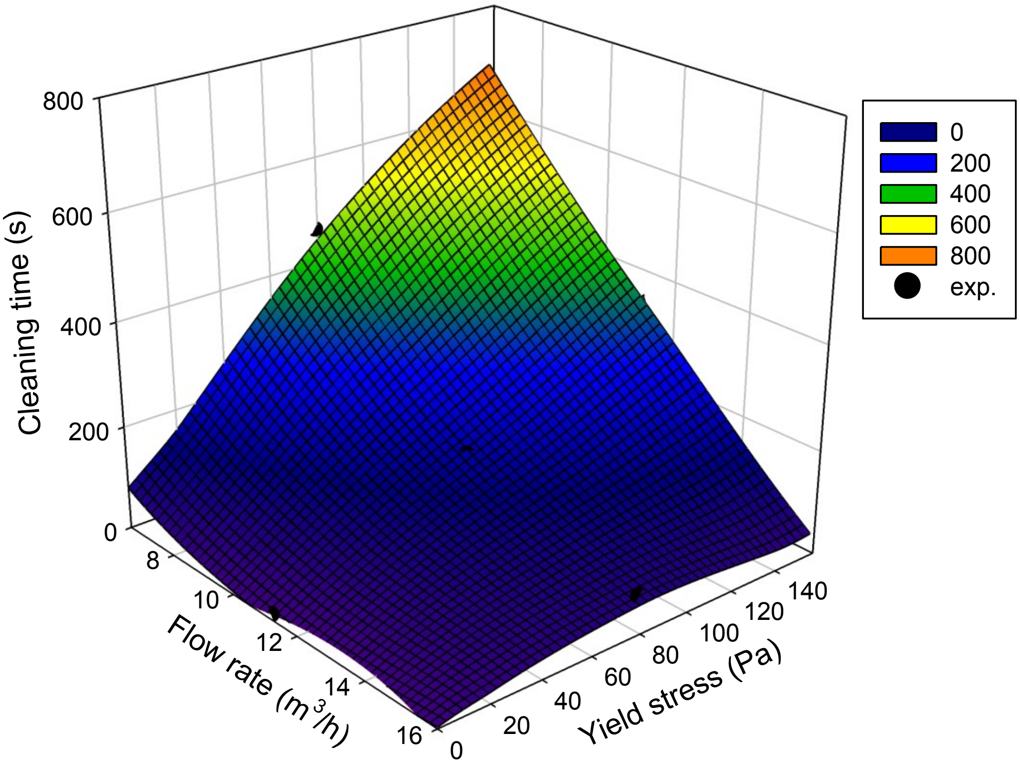
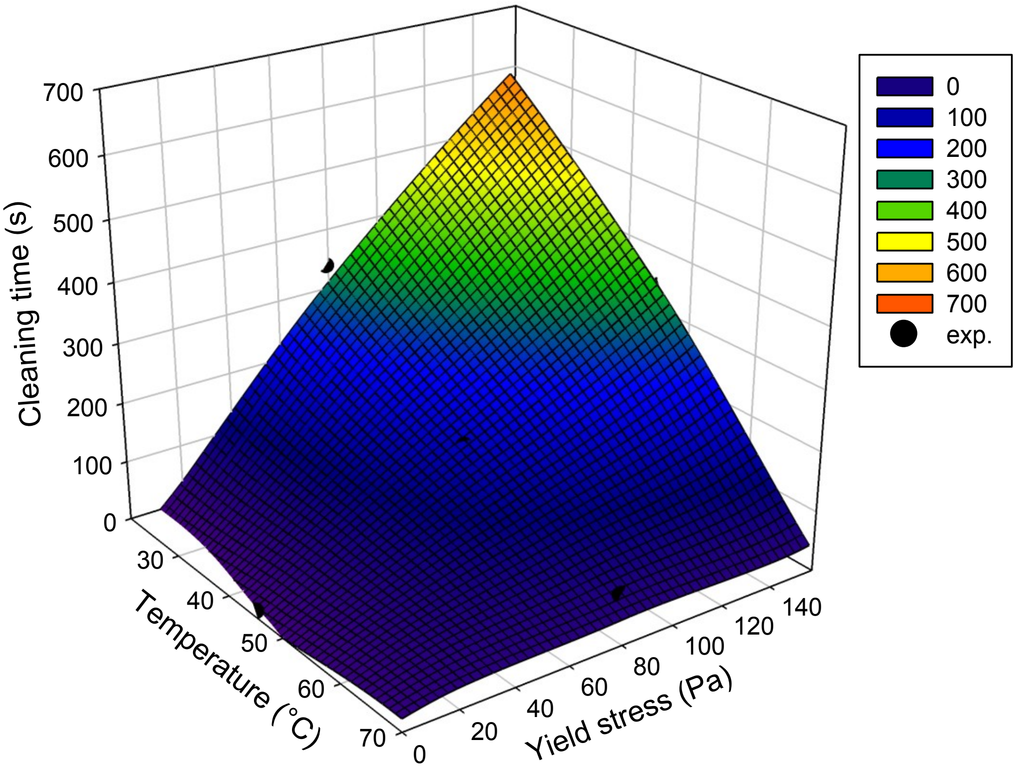


Figure 6.4: (a) Mass fraction left vs time graph of deposits studied in 0.0239 m diameter pipe at 0.55 ms^{-1} and 20 °C. (b) Removal modes of soils. 1- Shear thinning and Newtonian soils (shampoo, glucose) and 2- Yield stress soils. 2nd stage of cleaning is not observed in the removal stages of zero-yield soils.



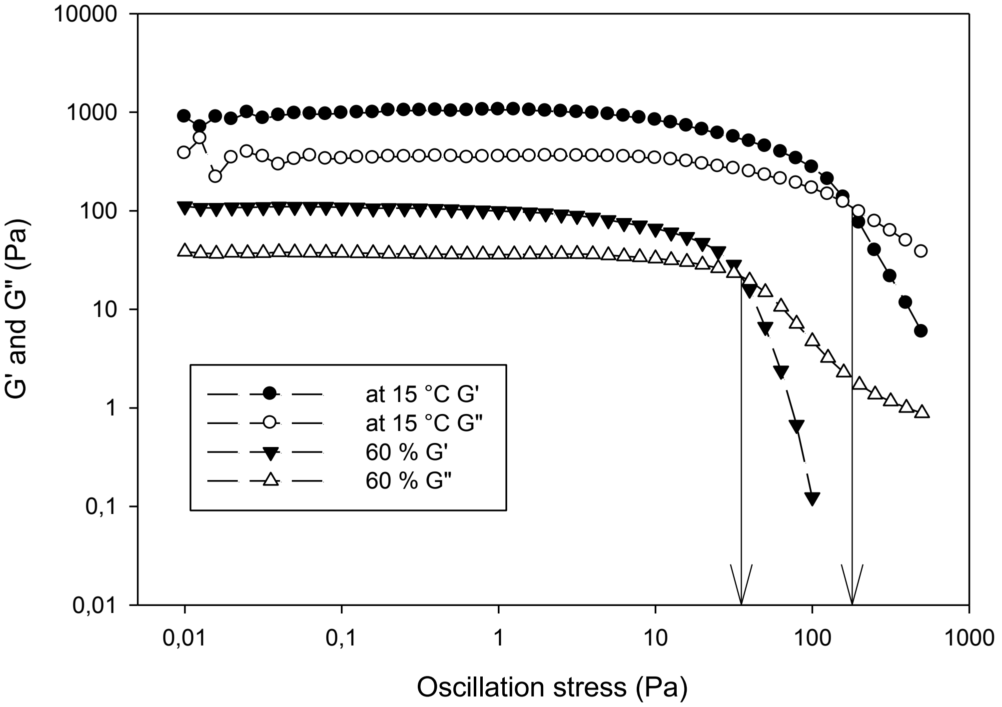
a)

Figure 6.5: (a) Predictive cleaning times are shown as surface plots. It shows the effect of yield stress and flow rate on cleaning time at 45 °C. Cleaning is performed at pilot scale and cleaning time is determined by the turbidity probe.



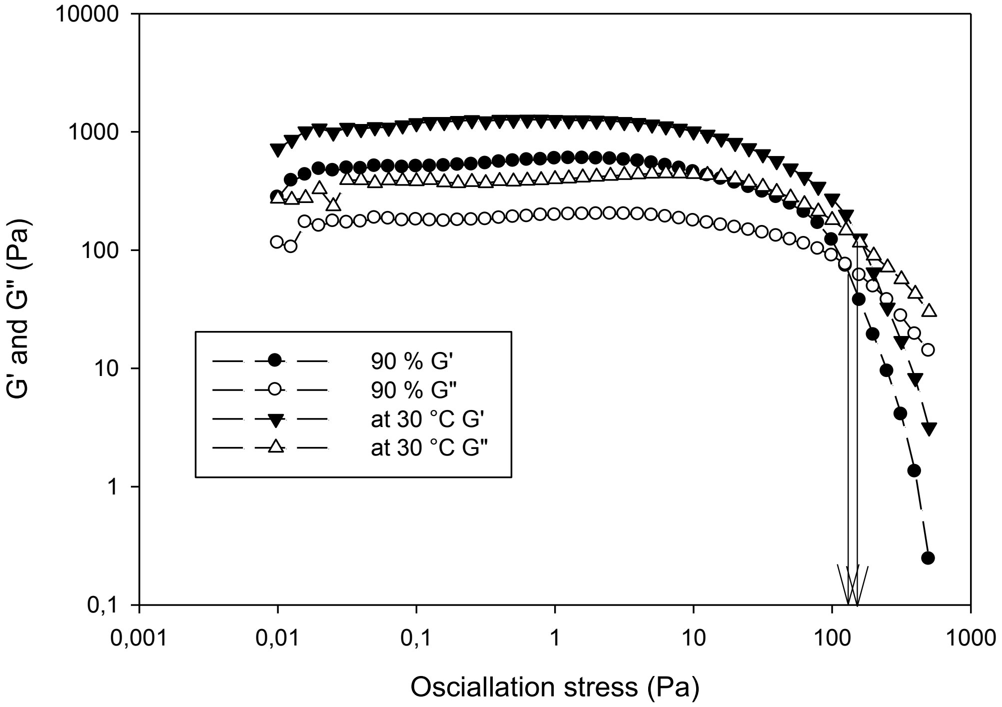
b)

Figure 6.5: (b) shows the effect of yield stress and temperature on cleaning time at 11.2 m^3h^{-1}. Points on the graphs are the experimental cleaning times. Product recovery is done at same conditions for all experiments (20 °C and 17 m^3 h^{-1}). Cleaning is performed at pilot scale and cleaning time is determined by the turbidity probe.



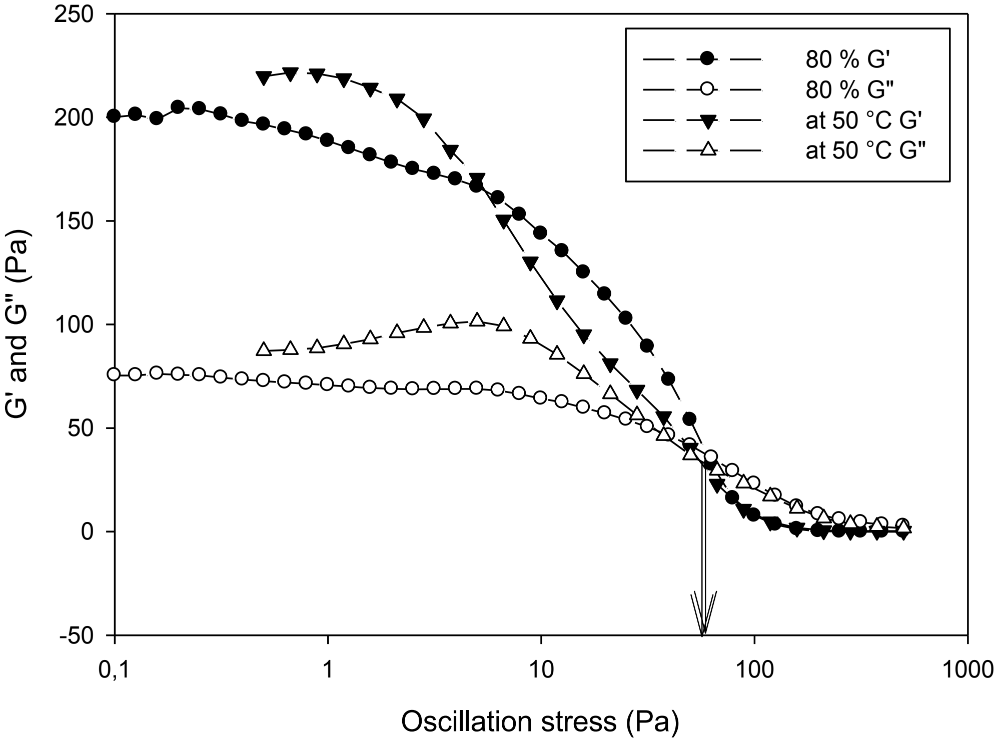
10000
1000
100
10
1
0,1
0,01
G' and G'' (Pa)
0,01
0,1
1
10
100
1000
Oscillation stress (Pa)
at 15 °C G'
at 15 °C G''
60 % G'
60 % G''

a)



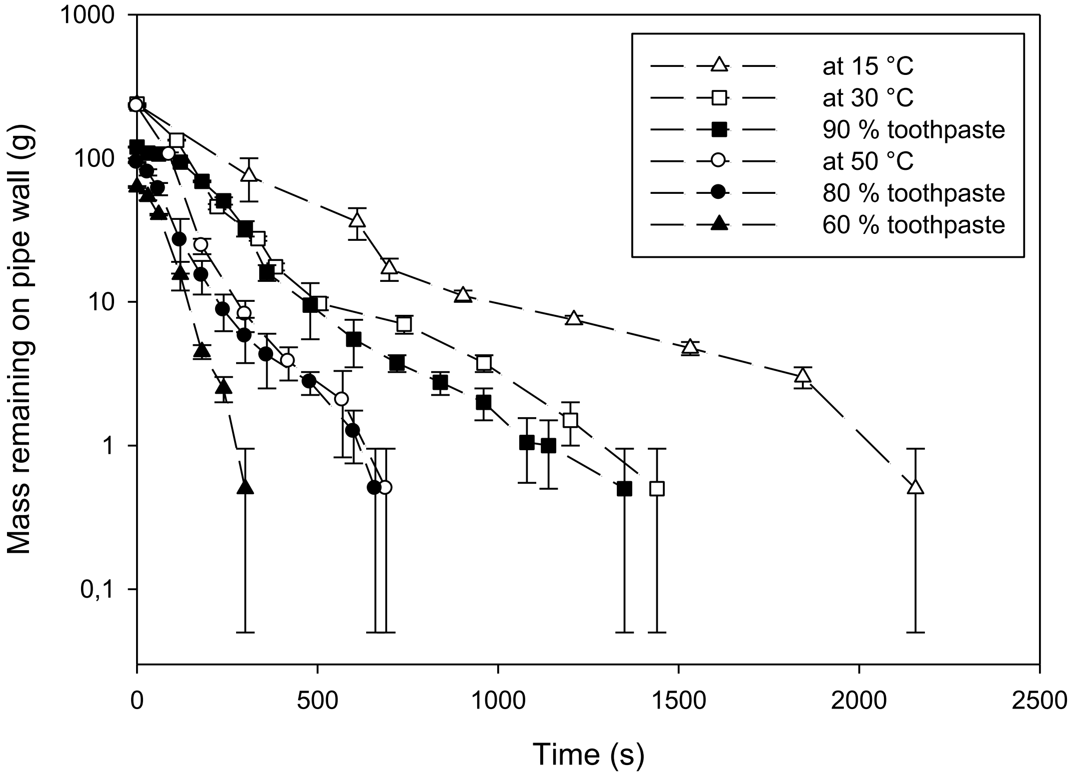
10000
1000
100
10
1
0,1
G' and G'' (Pa)
0,001
0,01
0,1
1
10
100
1000
Osciallation stress (Pa)
90 % G'
90 % G''
at 30 °C G'
at 30 °C G''

b)



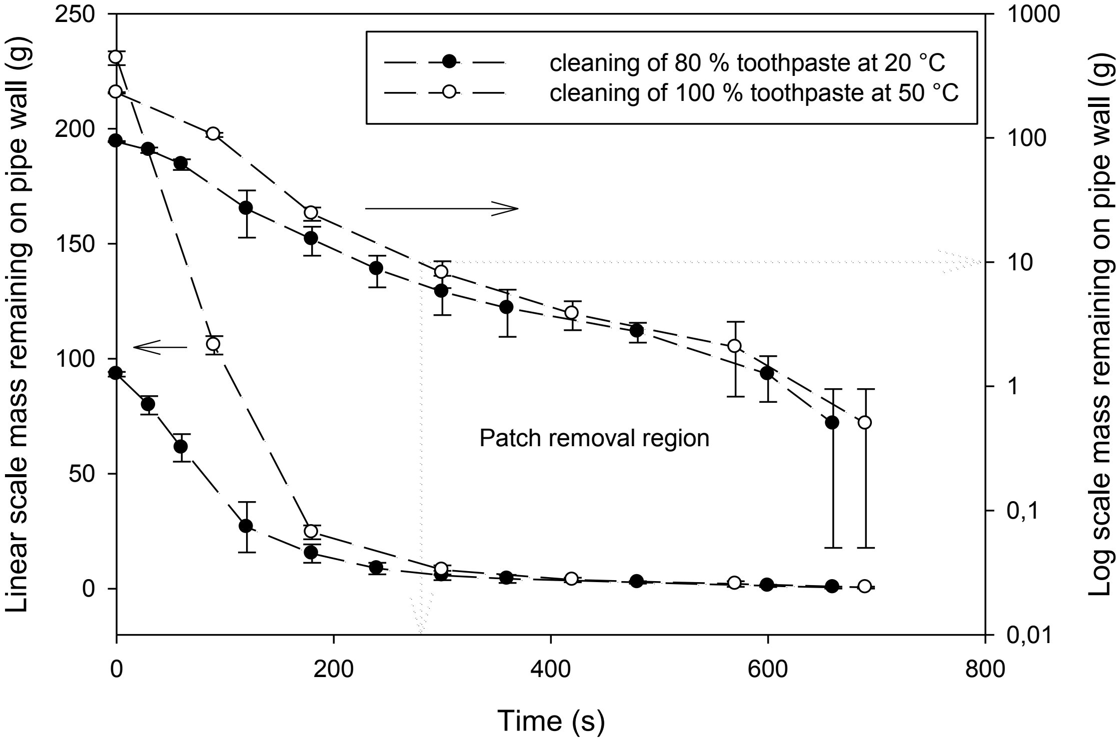
c)

Figure 6.6: Typical oscillatory stress sweep results to determine yield stress values of materials. I.e. oscillation stress at the crossover of G’ and G’’ is chosen as the yield stress value of the deposits and indicated by the arrows. (a) includes the data for toothpaste (not diluted) at 15 °C and 60 wt % toothpaste at 20 °C, (b) includes the data for toothpaste (not diluted) at 30 °C and 90 wt % toothpaste at 20 °C, (c) includes the data for toothpaste (not diluted) at 50 °C and 80 wt % toothpaste at 20 °C.



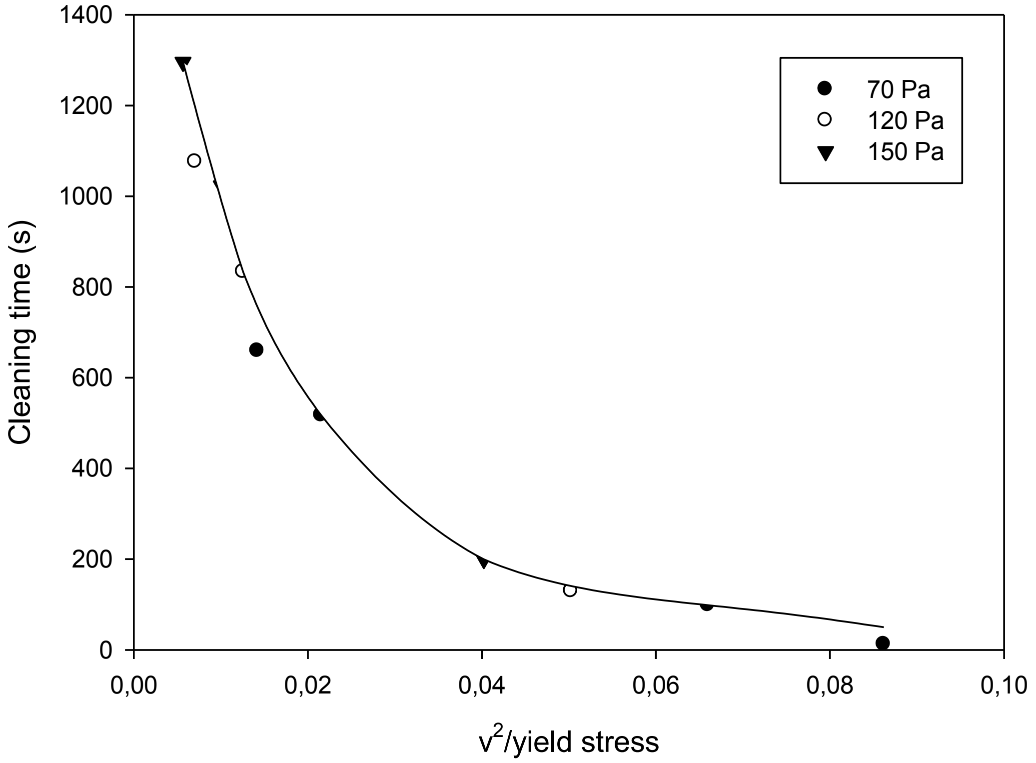
a)

Figure 6.7: Mass left on pipe wall vs time graph of deposits studied in 0.0239 m diameter pipe at 0.55 m /s. Product recovery is done at the same conditions in all experiments (0.55 m s^{-1} and 20 °C). (a) Cleaning profiles of toothpaste at 15 °C, 30 °C and 50 °C. It also includes the cleaning profiles of 60, 80 and 90 % diluted toothpaste at 20 °C.



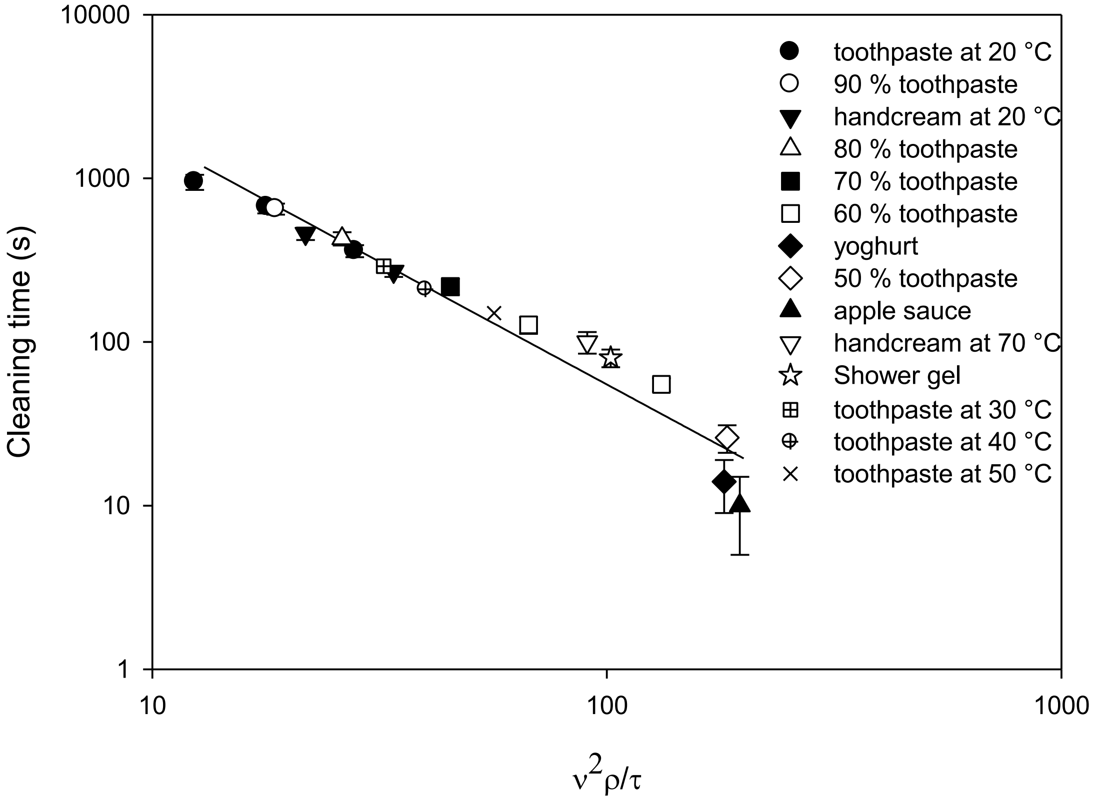
b)

Figure 6.8: Mass left on pipe wall vs time graph of deposits studied in 0.0239 m diameter pipe at 0.55 m /s. Product recovery is done at the same conditions in all experiments (0.55 m s^{-1} and 20 °C). (b) Cleaning profiles of undiluted toothpaste at 50 °C and 80 % diluted toothpaste at 20 °C plotted in linear and logarithmic scale. Each data point is averaged from at least two experiments and the maximum and minimum values are plotted as error bars.



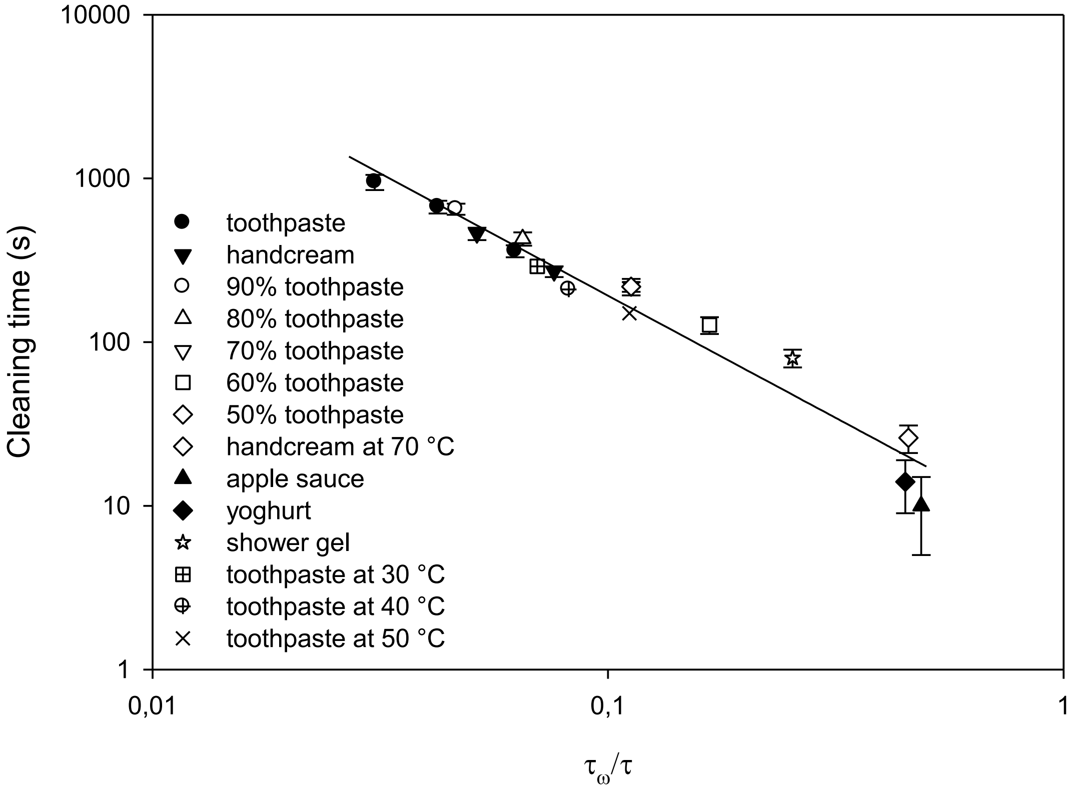
a)

Figure 6.9: (a) Cleaning time versus velocity square over yield stress plot obtained from response surface equation (Equation 6-1). A power law fit gives: time = 2.13 $(v^2/\tau)^{-1.35}$ with an R^2 of 0.8. Cleaning is performed at pilot scale.



b)

Figure 6.9: (b) Cleaning time versus dimensionless number plot including experimental data in Fig. 6.1 (b) and chapter 5. It gives: time = 54800 $(v^2\rho/\tau)^{-1.5}$ with an R^2 of 0.955. Cleaning is performed at pilot scale and cleaning time is determined by the turbidity probe.



c)

Figure 6.9: (c) Cleaning time versus dimensionless wall shear stress over yield stress of the deposit plot including experimental data in Fig. 6.1 (b) and chapter 5. It gives: time = 6.17($\tau_w/\tau)^{-1.5}$ with an R^2 of 0.958. Cleaning is performed at pilot scale and cleaning time is determined by the turbidity probe.

Chapter 7: SCALE UP & CHARACTERISATION OF THE FLOW EFFECT

Chapter 1 has highlighted that due to considerable use of heated chemicals in cleaning, the cost and environmental impact of the process is considerable. Chapter 2 has pointed out that one of the limitations in decreasing the cleaning cost is the difficulty of scale up of laboratory data due to the lack of true understanding of the cleaning process.

In order to remove deposit from the surface, a certain amount of energy from either mechanical, heat or chemical energies is brought to the surface by pumping the cleaning solution. The parameter with the largest impact is no doubt mechanical action because material would not be removed and disposed of in an acceptable time without it. How to relate this mechanical action with cleaning is essential because it may help to understand the cleaning process in detail. Consequently, optimisation and scale up of cleaning can be achieved which probably result in great cost reductions. Thus, many studies have been tried to characterise the effect of flow on cleaning. As stated in Chapter 2, all of the studies have agreed that increasing mechanical action by increasing flow velocity increases cleaning rate, however, it is not clear until now how to express this. There is a big controversy in the literature about this issue and Reynolds number, velocity and wall shear stress have been separately argued to be the potential parameters which can characterise the flow effect on cleaning. In Chapter 6, a kind of flow energy of cleaning fluid is found to be a good fluid mechanical parameter which might characterise the flow effect. Also, a hydrodynamic parameter of turbulence is predicted to affect cleaning but how to quantify this effect is also unknown in literature. Therefore, the aims of this work are to:

i) characterise the effect of flow on cleaning by studying cleaning of fully filled straight pipes at lab and pilot scale at different velocities.

ii) investigate the effect of turbulence on cleaning by studying cleaning of fully filled straight pipes placed just after various fittings.

As mentioned in section 3.5.4 a discontinuous visual observation method is used in this chapter to determine the end point of cleaning. The weight analysis method is used to compare cleaning rates. Graphs are plotted by using the data of mass left per unit surface area in order to compare the results of lab and pilot scale experiments. The product recovery stage is done at the same conditions (1.5 m s^{-1} at 20 °C, unless stated) in all experiments presented here to minimise the effect of product recovery stage found in Chapter 4.

7.1 The effect of flow velocity

Experiments are done at similar range of velocities in lab and pilot scale pipes to investigate the effect of flow velocity on cleaning. Since yield stress deposits are found to show the same cleaning behaviour in Chapter 6, the cleaning of hand cream is studied here but also comparison between the cleaning of toothpaste and hand cream will be presented in section 7.4. Figure 7.1 (a) shows cleaning time of hand cream data against velocity for both scales. It clearly illustrates that;

- Cleaning at an increased flow velocity decreases the cleaning time. And, cleaning happens substantially faster at lab scale, which suggests that decreasing the diameter of the pipe causes rapid cleaning.
- For instance, at the same velocity of 1 m s^{-1} , lab scale pipe is cleaned at 800 ± 20 s, while pilot scale pipe is cleaned at 3420 ± 240 s.
- Therefore, it is proved that previously given guidance rules (1.5 m s^{-1}) which only depend on velocity is deceptive and is only valid for one geometry. It implies that velocity parameter solely does not control cleaning.

Figure 7.1 (b) shows mass per unit area against time during cleaning of hand cream at the same velocity of 1 m s^{-1} at both length scales. It is clear that cleaning rate is higher in the lab scale experiment and cleaning ends much more quickly in the lab scale pipe than in the pilot scale pipe.

7.2 The effect of Reynolds number

Figure 7.2 (a) shows cleaning time of hand cream versus Reynolds number for both length scales. It illustrates that;

- small scale pipe is again observed to be cleaned significantly earlier than the pilot scale pipe at the same Reynolds number,
- for instance, at the same Reynolds number of 42000, the lab scale pipe is cleaned at 120 ± 20 s, while the pilot scale pipe is cleaned at 3420 ± 240 s.

Figure 7.2 (b) depicts mass per unit area against time during cleaning at Re 42000. It can be observed that at the same Reynolds number, hand cream is removed exceedingly fast from the lab scale pipe, which implies that Reynolds number has also no relation with cleaning.

Previously, it was argued that cleaning could be enhanced by decreasing boundary layer thickness (De Goederen et al., 1989). Therefore, increasing Reynolds number was believed to have a considerable effect on cleaning. However, this finding suggests that boundary layer thickness itself might not have a significant effect on the cleaning process since cleaning happens much faster in the lab scale experiments despite having much lower Reynolds number values as shown in Figure 7.2 (a).

7.3 The effect of wall shear stress

Figure 7.3 (a) shows the relation between cleaning time and wall shear stress which is so far reputed to be the most relevant variable to cleaning. It shows that;

- again cleaning times are shorter in the lab scale pipe, for instance, cleaning times are 375 ± 60 s and 730 ± 90 s for lab and pilot scale pipes, respectively at the same wall shear stress, 5.8 Pa,
- although the differences in the cleaning times for both scales are smaller than for Reynolds Number and velocity, still it takes double the time to clean the larger diameter pipe than the smaller diameter pipe at the same wall shear stress.

Figure 7.3 (b) shows the mass removed per unit surface area in both experimental scales at the same wall shear stress, 11.5 Pa. This figure also clearly shows that cleaning rate in the lab scale pipe is faster than that in the pilot scale pipe which means that the wall shear stress is also not a satisfactory fluid mechanical parameter to characterise the flow effect on cleaning and scale up cleaning.

7.4 The effect of a dimensionless number

So far different cleaning times have been obtained at the lab and pilot scale when cleaning is performed at the same velocity, Reynolds number and wall shear stress separately in these scales. Therefore, all data are analysed in an aim to find an expression such that cleaning times at both scales will be similar when cleaning is carried out at the same value of this expression. A dimensionless number is found as shown in Equation 7.1;

$$v^2/(gD) \qquad (7.1)$$

where g ($m\ s^{-2}$) is acceleration due to gravity (taken as 9.8 $m\ s^{-2}$), v ($m\ s^{-1}$) is flow velocity and D (m) is the pipe diameter. This dimensionless number is the head loss (energy loss) in flow due to friction per unit length of a straight pipe. Figure 7.4 (a) shows the cleaning time data re-plotted against this dimensionless frictional loss number. All data collapse onto a one line showing that cleaning times are similar at both length scales. For instance, the cleaning time is 720 ± 90 s for the small scale pipe and 730 ± 60 s for the pilot scale pipe at the same frictional loss value of 5.

Mass removed per unit area vs. cleaning time at a frictional loss parameter of 7 is shown in Figure 7.4 (b). Similar cleaning rates can be seen between the cleaning of two length scales. Hence, this finding notably suggests that this number can be used to scale up lab data.

In the previous Chapter, a new dimensionless number has been defined, shown in Equation 6.2:

$$(v^2\rho)/\tau \qquad (6.2)$$

where v and ρ are the flow velocity (m s^{-1}) and density (kg m^{-3}) of water and τ is a yield stress of a deposit (kg m^{-1} s^{-2}). This dimensionless number collapsed the all cleaning time data, which obtained from cleaning of various materials at different velocities at pilot scale, onto a one curve. Hence, another set of experiments are performed with the another yield stress material (toothpaste) to investigate if any further relation can be found which can combine all parameters such as scale, type of material and process conditions. Experiments and results are shown in Table 7.1.

Cleaning times of toothpaste and hand cream at different velocity and scales are plotted against Equation 7.2 which is formed by introducing a diameter term into the denominator of Equation 6.2:

$$(v^2\rho)/(D\tau) \qquad (7.2)$$

A plot of the all data is shown in Figure 7.5 (a). Cleaning times are found to lie on a one master curve with an R^2 of 0.985. This shows that the expression successfully achieve to scale up of the cleaning data of different materials which are cleaned predominantly by the flow action. Equation 7.3 shows the relationship found with the cleaning time:

$$\text{Cleaning time} = 13*10^5\,(v^2\rho/(D\tau))^{-1.328},\ R^2 = 0.985 \qquad (7.3)$$

In chapter 6, the dimensionless ratio of wall shear stress to yield stress of materials also successfully collapsed the cleaning time data onto one curve at one scale (Figure 6.9 (c)). Therefore, the relevance of this ratio with the cleaning process is tested with the cleaning time data at two scales in Figure 7.5 (b). The figure clearly illustrates that different cleaning times are observed at different scales which implies that in addition to wall shear forces, different fluid mechanical actions contributes the mechanical removal process. The results indicate that Equation 7.2 can adequately characterise the fluid mechanical effect on the cleaning of similar type of materials.

7.5 The effect of turbulence on cleaning

In a pipe line, some energy is lost due to friction in straight pipes according to the following Darcy-Weisbach phenomenological equation in terms of pressure loss (Coulson et al., 1999);

$$f\frac{1}{2}L(\frac{v^2\rho}{D}) \qquad (7.4)$$

where f is friction factor, L (m) is pipe length, v (ms^{-1}) is the mean velocity in the pipe, ρ (kgm^{-3}) is the density of the fluid and D (m) is the diameter of the pipe.

In addition to this, extra energy is lost by reason of additional turbulence arising when fluid flows through some components which cause a change of direction, change of area, e.g. branching or bends. Pressure loss for such cases is generally expressed by the following empirical equation (Coulson et al., 1999):

$$K\frac{{v_f}^2\rho}{2} \qquad (7.5)$$

where K is the resistance coefficient and depends on the type of the fitting and v_f ($m\ s^{-1}$) is the mean flow velocity on a section not affected by the section where the energy loss is produced. Therefore, additional turbulence occurs inside or just after the fittings and this may affect the cleaning. To study the effect of turbulence on cleaning, the straight pipe test section is positioned after various fittings in pilot plant. Figure 7.6 shows the pipe configurations used and dimensions of the fittings. 0.3 m (6.25D) straight pipe is used as the test section here. It should be noted that;

- in all experiments here, initial product recovery is done in configuration (d) in Fig. 7.6 with a water velocity of 2.45 m s^{-1} at 20 °C to minimise the effect of product recovery mentioned in Chapter 4. This is crucial to identify the effect of turbulence purely. Generally, 0.18 ± 0.01 wt fraction of toothpaste is left on the pipe wall. This will be referred to as "cored".
- Then, the set up is changed to study cleaning of the test section in each configuration shown in Figure 7.6. Cleaning is done at the same conditions, 1.5 m s^{-1} at 20 °C. Experiments are done at least in duplicate at each configuration.
- The weight analysis method is used to detect cleaning end-point and investigate cleaning rates. This method is explained in section 3.5.1.

Figure 7.7 illustrates the effect of turbulence caused by flow through fittings on cleaning by showing weight fraction left in the straight pipe placed after various fittings with time. The figure shows that;

- cleaning rate is observed to be the slowest when no fitting is used (set up (d) in Fig. 7.6), which shows the importance of turbulence caused by the fittings on cleaning.
- on the other hand, cleaning rate after the gradual expansion is the fastest suggesting that the expansion causes the highest turbulence (set up (e) in Fig. 7.6). In these divergent flows, boundary layer separation is generally observed due to the positive

pressure gradients in the flow as pressure increases in the downstream of the expansion. And vortices occur at the edge of the separated boundary layer, where the velocities change direction. Consequently, this boundary layer separation and increase in the turbulence because of the vortices cause very large energy losses in the flow (Kundu and Cohen, 2002). Therefore, these fluid dynamical actions are presumably responsible for the rapid cleaning in these divergent flows which are inherently unstable,

- when the 90° elbow is used (set up (a) in Fig. 7.6), the cleaning rate in the test section is a little higher than the no fitting case,
- however, flow is highly disturbed and cleaning rate increases considerably when the closed return bend and the 90° elbow are used back-to-back (set up (b) in Fig. 7.6),
- after the tee with branched flow (set up (c) in Fig. 7.6), cleaning rate is moderate among the studied configurations.

To quantify the turbulence production and the effect of this turbulence on cleaning, pressure loss caused by the fittings is calculated according to Equation 7.5. K and v_f values are shown in Table 7.2. Cleaning time is plotted against the dimensionless ratio of pressure loss due to fittings to yield stress of the deposit in Figure 7.8. A good linear relationship is observed between the dimensionless number and the cleaning time.

A linear fitting gives: Cleaning time = -68.62 (P_{loss}/yield stress) + 1161 ($R^2 = 0.96$).

This suggests that although the cleaning of only toothpaste is studied here, this number may characterise the effect of turbulence on the cleaning of various deposits. A study of cleaning of various deposits in these pipe configurations might be valuable as a future work.

Production of higher turbulence levels causes increase in the energy loss and this contributes the cleaning process accordingly.

The results suggest that there is a strong relationship between the cleaning time and the flow actions which are responsible for the energy losses in a system. Complex fluid mechanical actions involve in the cleaning process. Nevertheless, the effect of fluid mechanical action on cleaning can be quantified by calculating the energy losses caused by the flow either in straight pipes or in the fittings.

7.6 Discussion

According to the results, the cleaning number (Equation 7.2) is found to be a very good parameter to predict and scale up cleaning data in straight pipes. Physically, it is a ratio of pressure drop (Pa) per unit length due to friction loss in straight pipe flow to yield stress (Pa) of a material. Moreover, the effect of turbulence level in flow on cleaning is also characterised by the pressure loss calculation for the flow in fittings. These findings strongly suggest that fluid mechanical parameters, which induce energy losses in the flow, are involved in the removal process of a deposit from surfaces. These parameters might be skin friction (wall shear stress), turbulence, swirl and recirculation motions, vortices etc. Therefore, fluid mechanical removal in the cleaning process is also very complex, and single parameters like Reynolds number, velocity or wall shear stress cannot be used to characterise the flow effect on cleaning.

There are many implications derived from the findings. For instance, energy loss depends on the friction factor (f) which is influenced by the roughness of pipe surfaces. Energy loss increases with increase in the roughness value due to fluid actions such as wake formations behind roughness elements. Therefore, rough soil surfaces may increase the cleaning rates accordingly. Indeed, this is observed in section 4.2.3, (see Fig. 4.10). The cleaning of wavy and smooth films formed by changing the product recovery conditions is studied. It is found that cleaning rate of rougher film is higher than that of smoother film. Gordon et al. (2013) also observed the same behaviour. They investigated the influence of soil roughness on the cleaning of pre-gelatinised starch-based layers from stainless steel substrates by aqueous NaOH solutions at ambient temperature. They found that rough layers were cleaned more readily than those containing small inclusions.

Another implication is that the length of a straight pipe to be cleaned should have no effect on cleaning time since energy loss per unit length is the same throughout the pipe length. Therefore, cleaning should be completed at the same time for all parts of the straight pipe in steady flows provided that deposit is distributed evenly along the pipe. This is in good agreement with the work in section 5.3.4. It is observed in Figure 5.7 that cleaning rates and times of pipes are similar regardless of the pipe length. If the pipe length affects the cleaning, cleaning rates will not be similar in the pipes with different lengths.

The effect of friction also clarifies many conflicting results in the literature. Some researchers have reported that there is a threshold Re value in order to commence cleaning mechanically by flow action (Jackson and Low, 1982, Jennings et al., 1957). However, Bird and Fryer (1991) found that cleaning rate did not change substantially from laminar to turbulent flow in very narrow 6.35 mm OD pipes and cleaning started even at very low Reynolds number in their system. This might be due to the very small pipe diameter used, which made the friction loss very high in the pipe. Hence, cleaning could happen at low velocities in their system.

Early studies have used Reynolds number and wall shear stress in cleaning and found fairly good relationship between these parameters and cleaning like the relation observed in Figure 6.9 (c). The reason for this might be that both terms and energy loss parameter are a function of velocity. Thus, the cleaning time of simple geometries at one scale may be described as a function of Reynolds Number or wall shear stress. However, after studying the cleaning of mixed proof valves, Jensen and Friis concluded that wall shear stress was not a sole parameter to affect cleaning and they stated that complex geometries might be cleaned earlier than straight pipes (Jensen and Friis, 2005). This is also due to the fact that different geometries generally impose more energy losses than straight pipes.

Pulsed flows have also been shown to increase cleaning rates (Blel et al., 2009, Gillham et al., 2000). It was revealed that pressure losses in pulsed flows were in the range of 4 to 19 times larger than average pressure loss during one directional flows (Plett and Grasshoff, 2006). Therefore, pulsed flow also causes high energy losses in flow and enhances cleaning.

In brief, the energy loss due to friction per unit length is found to be the best parameter to characterise the flow effect on cleaning in both simple and more complicated geometries. Consequently, some values given previously in the literature as a rule of thumb of cleaning e.g. a minimum flow velocity of 1.5 m s^{-1} or a threshold Re are likely to be only valid for a specific system. In some cases, 2 m s^{-1} may not be enough to clean large diameter pipelines within acceptable time while this flow velocity will be overdesigned and wasteful for relatively small diameters of pipeline. For instance, according to Equation 7.3, it can be predicted that the cleaning of toothpaste from 0.2 m ID pipeline will take about 48 minutes at a water velocity of 2 m s^{-1}. However, the cleaning in 0.025 m ID pipeline will only take 3 minutes at the same cleaning velocity.

7.7 Conclusion

Cleaning of toothpaste and hand cream from lab and pilot scale straight pipes is studied at different flow velocities to characterise the flow effect on cleaning. A straight pipe section is also placed after various fittings to quantify the effect of turbulence on cleaning. After the investigation of these two groups of experiments, energy loss in flow is found to have a good relationship with cleaning times of the deposits.

Velocity, Reynolds number and wall shear stress solely are proved to have insignificant effect on the cleaning process. A dimensionless number (Equation 7.1), which is present in the empirical head loss equation, is observed to be the best parameter to characterise the flow effect on cleaning and scale up of cleaning time data for one type of deposits. It is found that straight pipes at different scales are cleaned at similar times when this dimensionless number is the same at both scales. Further development of the dimensionless number has enabled a new expression (Equation 7.2). It collapses all cleaning time data for different materials cleaned at different velocities and pipe diameters onto a one curve. This expression is a ratio between pressure loss in flow per unit pipe length and yield stress of a deposit.

Additional turbulence occurs when water flows through fittings e.g. a valve, a bend or a T-piece. Here, cleaning rate of the straight pipe section is found to increase when placed just after these types of fittings. Moreover, the magnitude of head loss caused by the fittings is also used to quantify the turbulence effect on cleaning. A linear relationship is found for increasing head loss and decreasing cleaning time. Therefore, these findings suggest that determination of energy loss in a hydrodynamical system might be valuable for scaling up of cleaning data or be used to predict and compare cleanability of any system where cleaning is governed by the fluid mechanical removal.

Tables Chapter 7

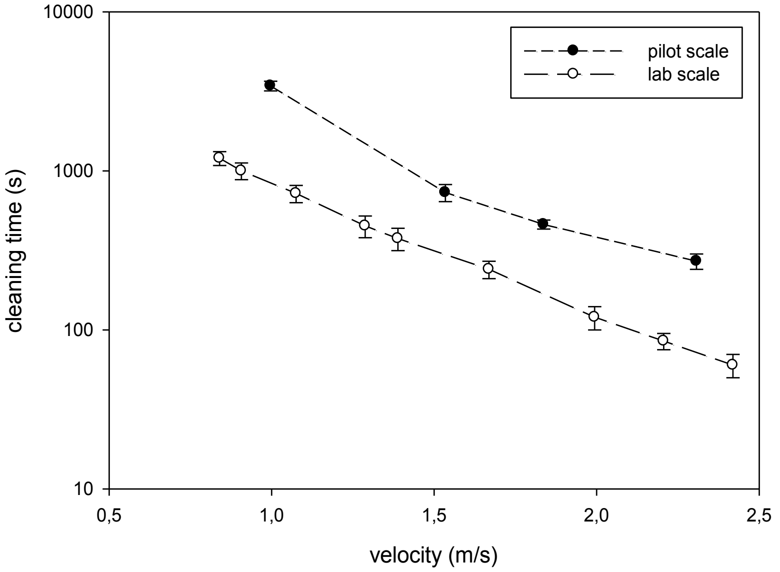
Table 7.1: Cleaning of toothpaste and hand cream at different velocities from lab and pilot scale pipes.

Pipe ID (m)	Flow velocity ($m\ s^{-1}$)	Cleaning time (s) of hand cream (τ = 165 Pa)	Flow velocity ($m\ s^{-1}$)	Cleaning time (s) of toothpaste (τ = 203 Pa)
0.0239	0.84	1200 ± 120	0.84	1590 ± 120
	0.91	1000 ± 120	1.07	930 ± 80
	1.07	720 ± 90	1.28	540 ± 60
	1.29	450 ± 70	1.61	370 ± 40
	1.39	375 ± 60	1.67	320 ± 30
	1.67	240 ± 30	2.00	180 ± 20
	1.99	120 ± 20	2.08	150 ± 10
	2.21	85 ± 10	2.21	130 ± 10
	2.42	60 ± 10	2.59	90 ± 10
0.0477	0.99	3420 ± 240	0.99	2280 ± 180
	1.53	730 ± 90	1.53	950 ± 90
	1.84	460 ± 30	1.84	630 ± 60
	2.31	270 ± 30	2.31	360 ± 30

Table 7.2: Resistance coefficients (Sinnott, 2005) and velocity values used in pressure loss calculations.

Fittings	K values	V_f (m s^{-1})	Head loss (m)
90° standard elbow	0.57	1.5	0.068
Tee (with flow through branch)	1.14	1.5	0.136
3 elbow (closed return bend and 90° elbow)	1.52	1.5	0.181
Gradual 13° expansion	0.12	6.14	0.23
No fitting	-	1.5	0.014

Figures Chapter 7



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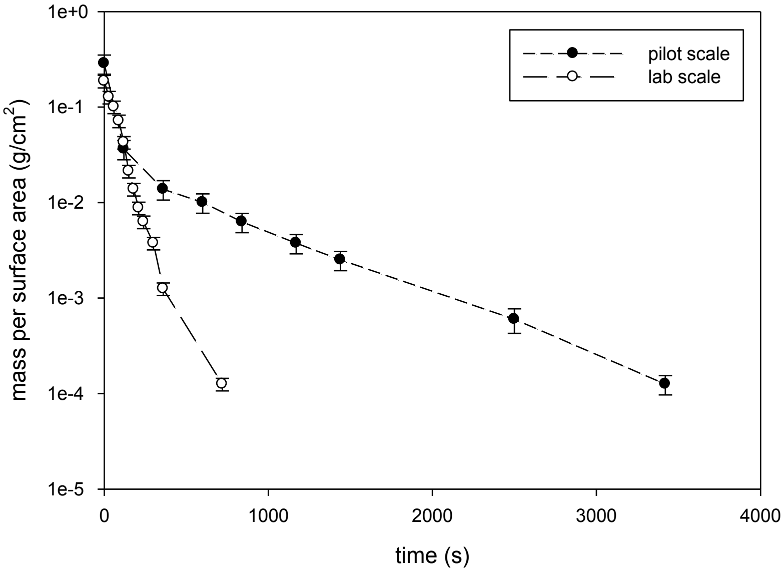
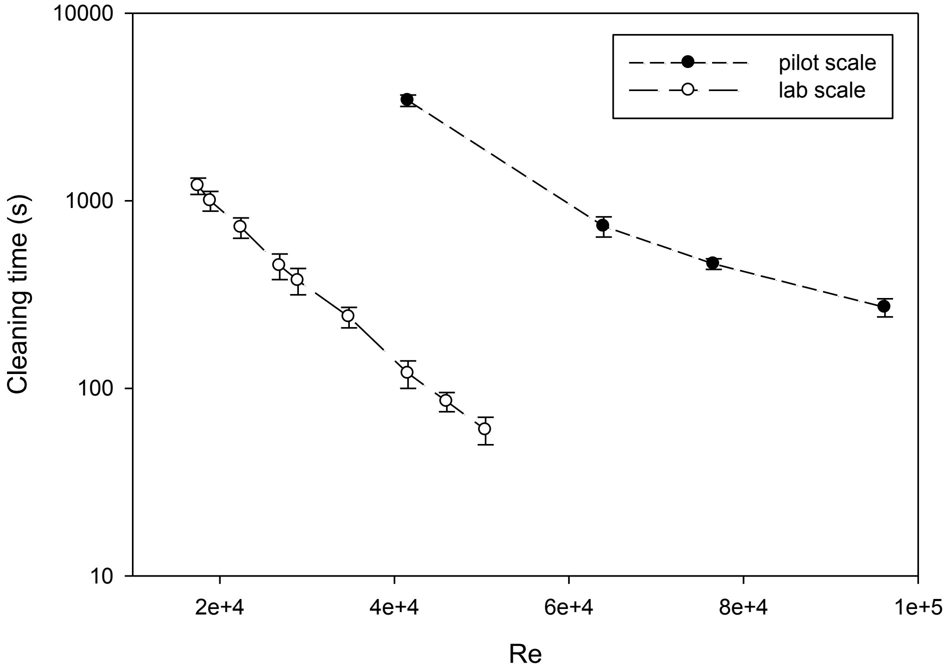
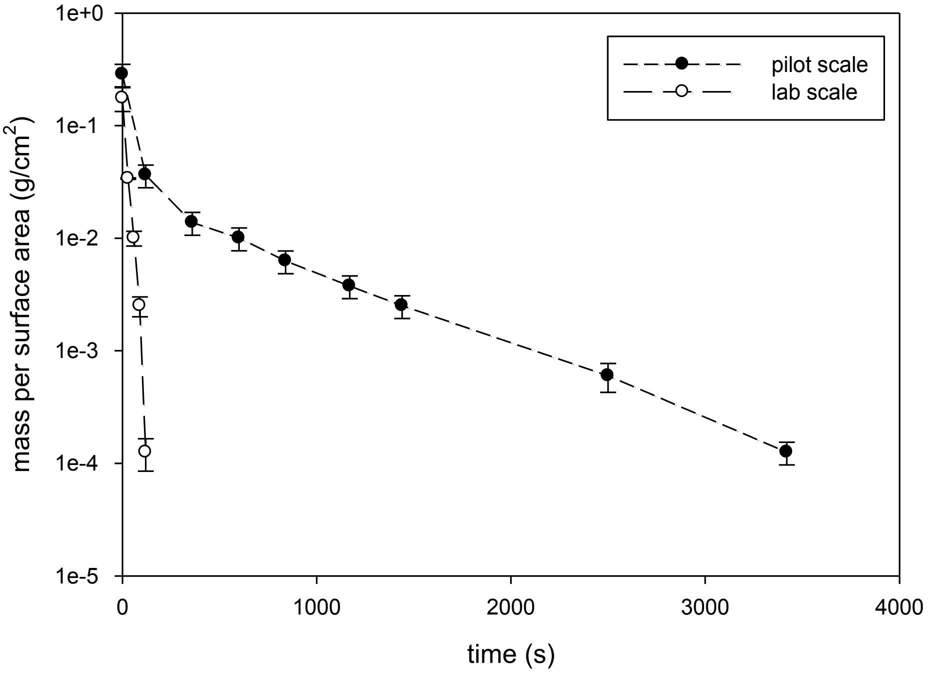


b)

Figure 7.1: (a) Graph of cleaning time of hand cream vs. velocity including the data in both scales. (b) Mass per unit surface area vs. time at the same velocity of 1 m s-1 at both scales at 20 °C. Pilot scale test pipe 0.5 m long (ID - 0.0477 m), lab scale test pipe 1 m long (ID - 0.0239 m).



a)



b)

Figure 7.2: (a) Graph of cleaning time of hand cream vs. Reynolds number including the data in both scales. (b) Mass per unit surface area vs. time at Re of 42000 at both length scales at 20 °C. Pilot scale pipe - 0.5 m (ID - 0.0477 m), lab scale pipe - 1 m (ID - 0.0239 m).

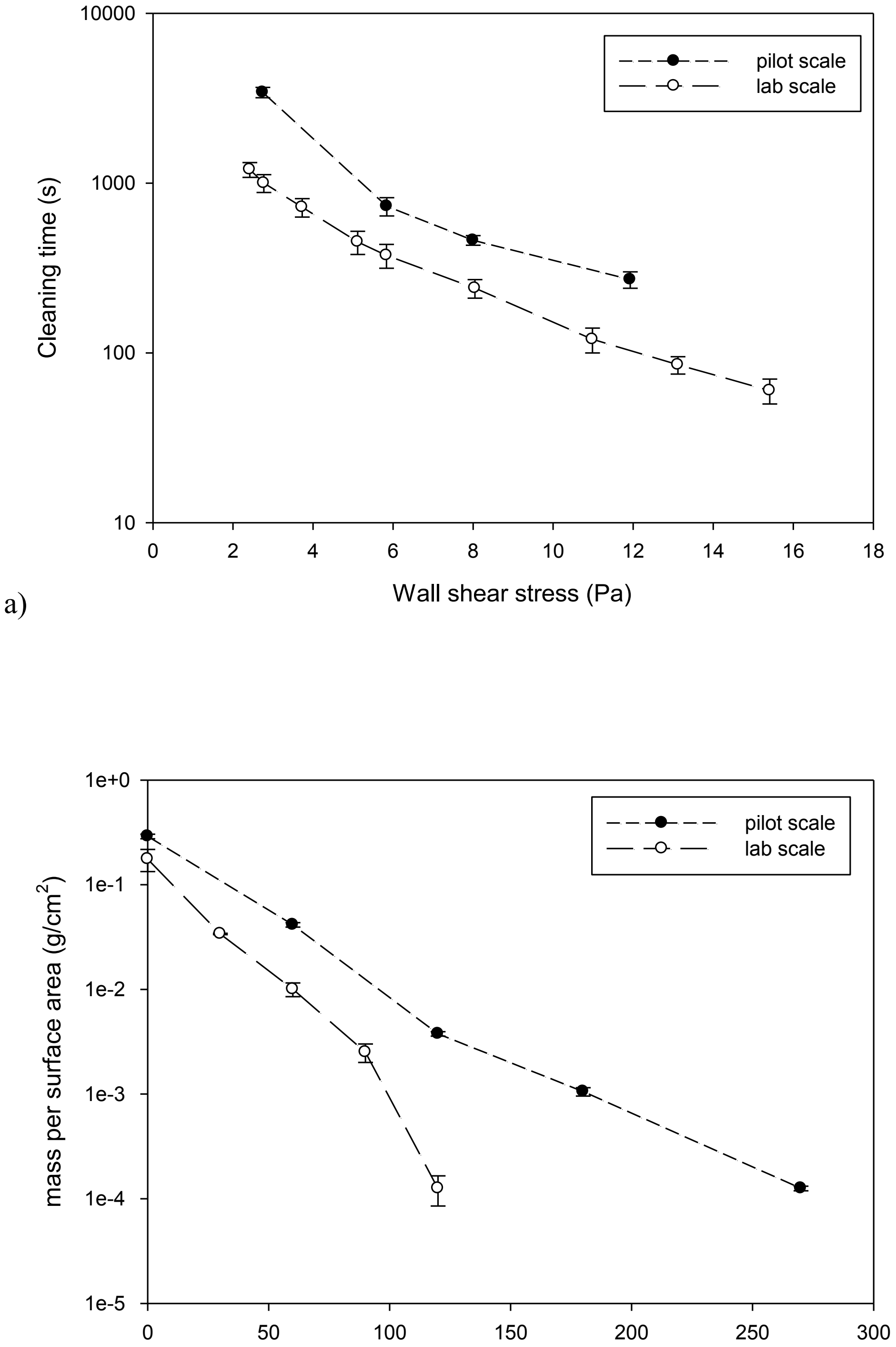


Figure 7.3: (a) Graph of cleaning time of hand cream vs. wall shear stress including the data in both scales. (b) Mass per unit surface area vs time at the same wall shear of 11.5 Pa in both scales at 20 °C. Pilot scale pipe - 0.5 m (ID 0.0477 m), lab scale pipe - 1 m (ID 0.0239 m).

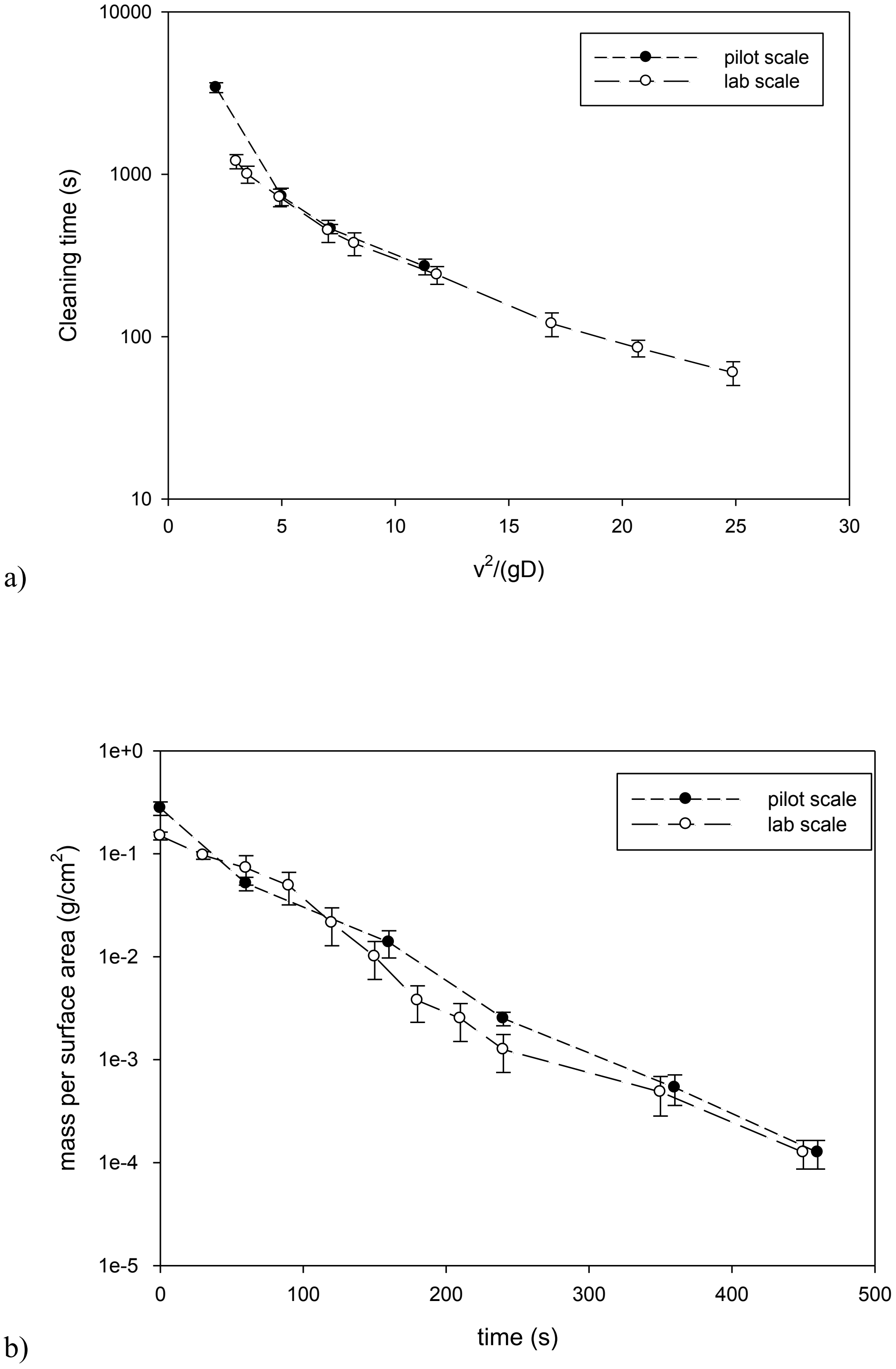
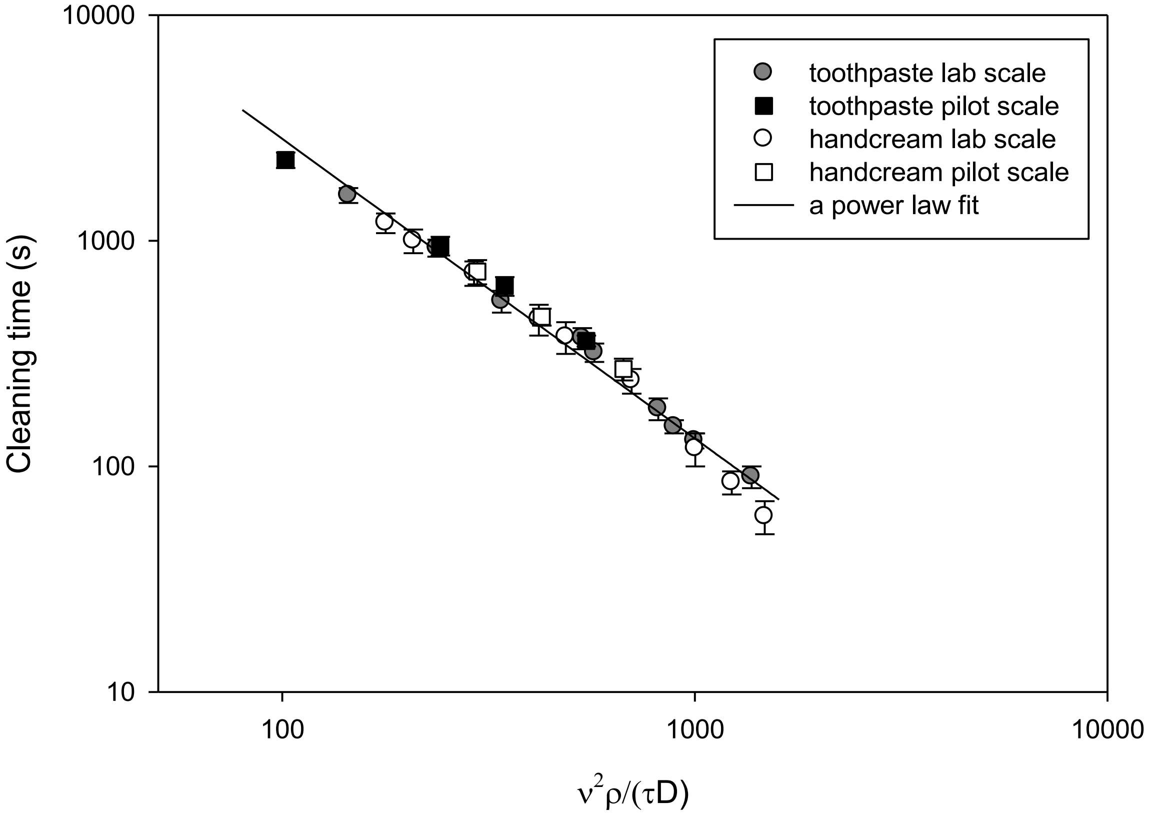
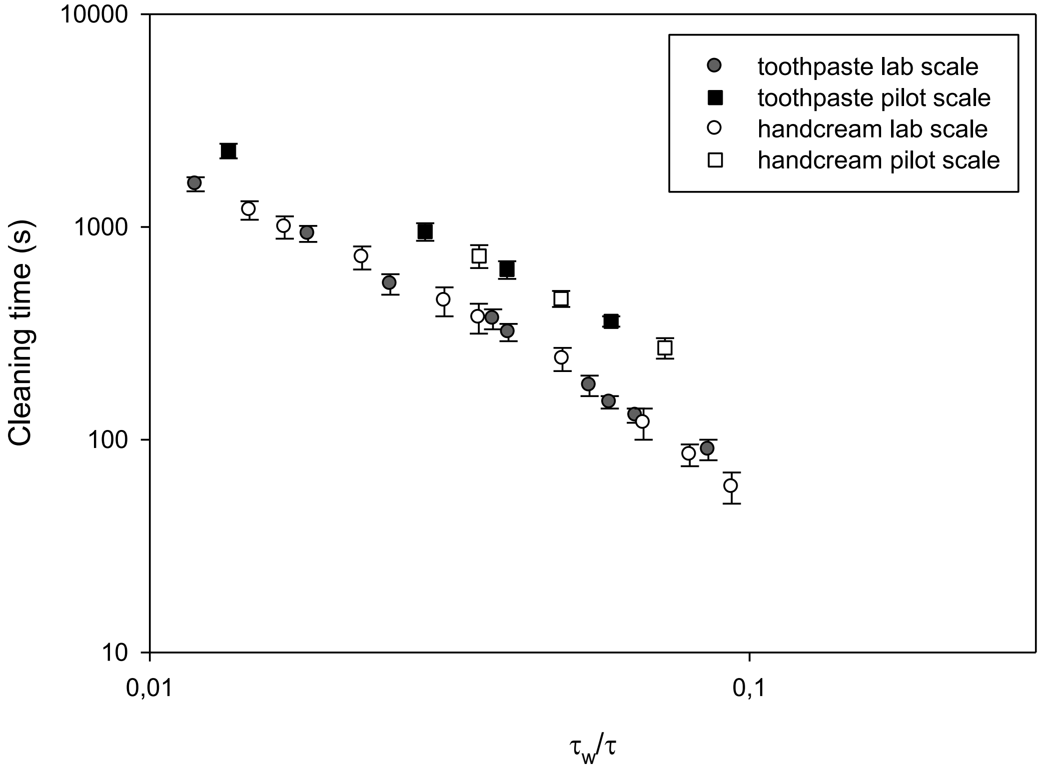


Figure 7.4: (a) Cleaning time of hand cream vs. head loss per unit length parameter including the data in both scales. (b) Mass per unit surface area vs time at the same head loss parameter of 7 in both scales at 20 °C. Pilot scale pipe - 0.5 m (ID 0.0477 m), lab scale pipe - 1 m (ID 0.0239 m).



a)

Figure 7.5 (a): Cleaning time vs Eq. 7-2 plot at 20 °C including all experimental data for toothpaste and hand cream. A power law fit of all cleaning time data gives: Cleaning time = $13e^5\ (v^2\rho/(D\tau))^{-1.328}$ ($R^2 = 0.985$).



b)

Figure 7.5 (b): Cleaning time vs dimensionless wall shear stress over yield stress of the deposit plot including all experimental data at two scales for toothpaste and hand cream.

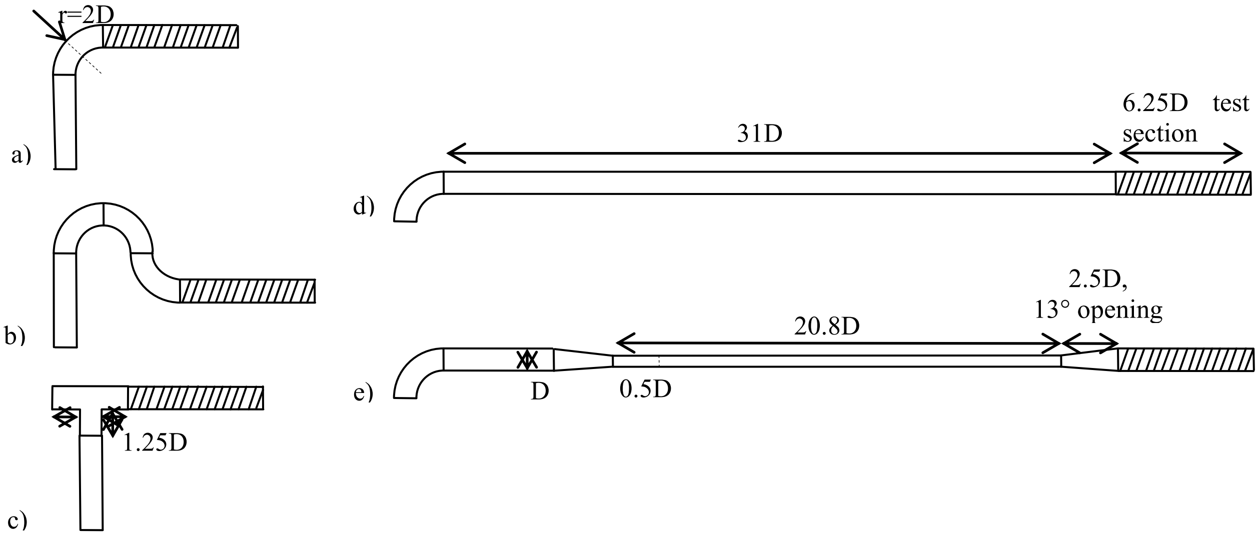


Figure 7.6: Schematic representation of the studied configurations. Striped area represents the test section. The test section is fully filled with toothpaste. Product recovery is only done using configuration (d) to get an even layer of material for each experiment (velocity of water was 2.45 m s^{-1} at 20 °C). For cleaning experiments following 'coring', the set-up is changed to study each configuration.

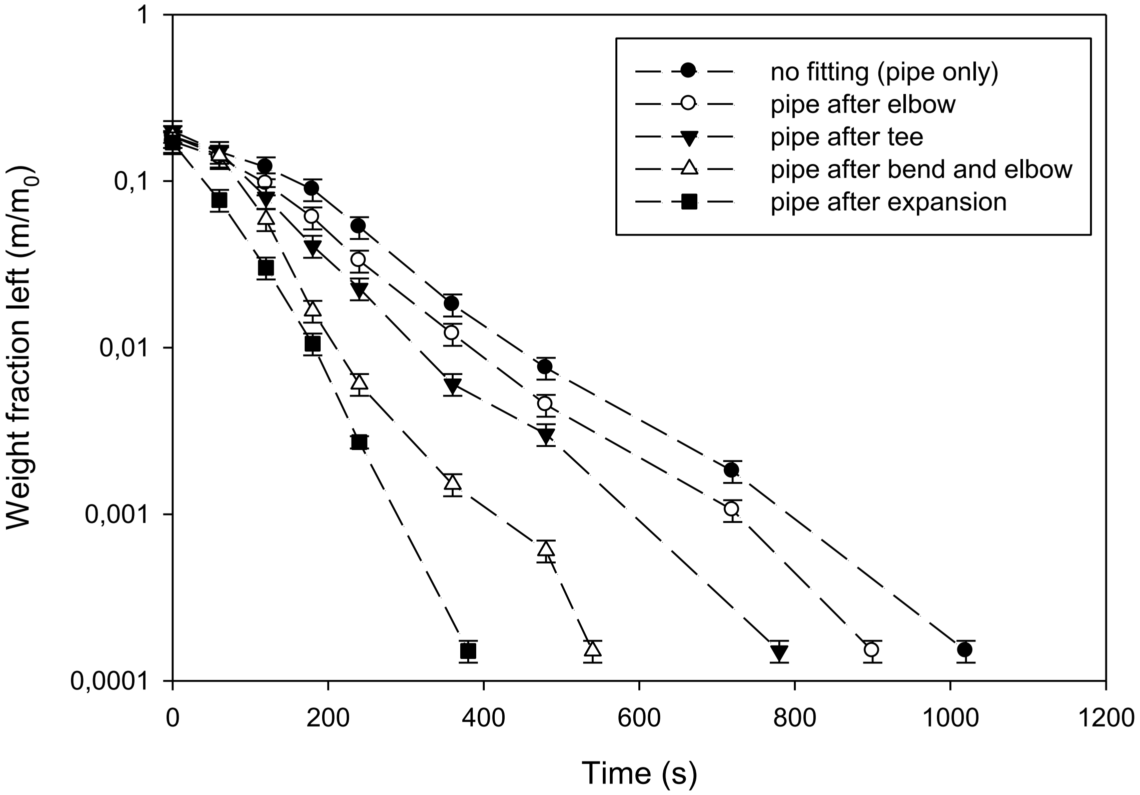


Figure 7.7: Weight fraction left in the straight pipe placed after various fittings with time (for set ups see Figure 7.6). m is mass of the toothpaste left in the pipe with time and m_0 is the original mass in the fully filled pipe. Test section was 30 cm (6.25D) straight pipe. Cleaning was done at 1.5 m s^{-1} at 20 °C.

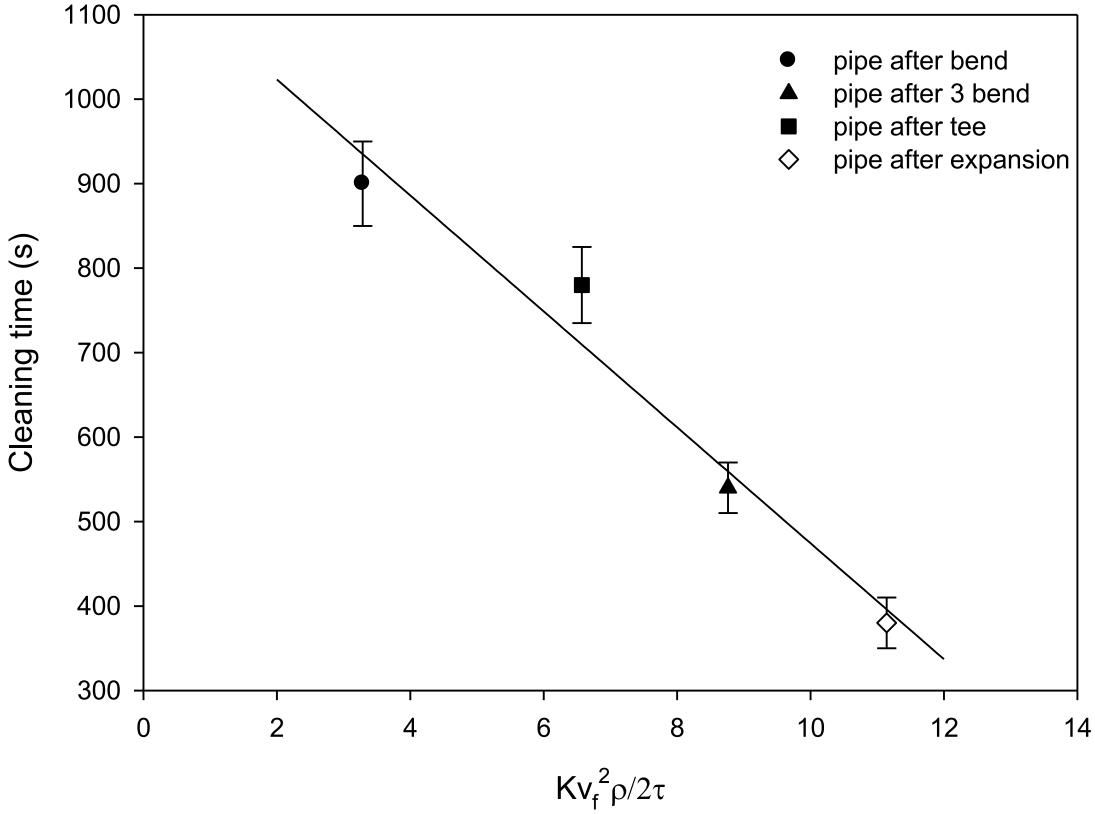


Figure 7.8 Cleaning time of toothpaste from straight pipe placed after various fittings (see Figure 7.6) vs. the dimensionless ratio of pressure loss (m) due to fittings to yield stress of the material. A linear fit gives: Cleaning time = -68.62 (P_{loss}/yield stress) + 1161 ($R^2 = 0.96$).

Chapter 8: CONCLUSIONS

Chapter 1 has highlighted the importance and significant impact of cleaning in the process industries. Therefore, the main aim of cleaning research is to minimise significant process costs associated with cleaning. The most important stage in minimising the impact of cleaning is to understand the cleaning process thoroughly. Chapter 2 has highlighted the importance of studying the below strands to gain a more fundamental understanding of the cleaning process:

- simple cleaning problems,
- cleaning of a simple hydrodynamic system,
- cleaning stages thoroughly,
- cleaning of different deposits and,
- different length scales

Therefore, this thesis has focused on understanding the fluid mechanical removal in the cleaning process by investigating the cleaning of fully filled pipes with different deposits at laboratory and pilot scale. The deposits selected in this thesis can all be removed by water alone to simplify the cleaning problem.

8.1 The effect of product recovery stage on the cleaning of pipes

Product recovery stage is the first stage in cleaning of a process material from a fully filled pipe. It removes the bulk of the process material and leaves a thin layer of deposit on the pipe wall. The product recovery stage is generally overlooked in cleaning of fully filled pipes as it is just initial few seconds of the overall cleaning process. Thus, the effect of product recovery stage on cleaning time and on the amount of product recovered is studied. The main conclusions obtained from this study are:

- The investigation has showed that the fully filled pipes with toothpaste are cleaned at different times under the same cleaning conditions when the product recovery stage is conducted at different conditions. This suggests that if any study in the future investigates cleaning of fully filled pipes, the product recovery stage should be taken into consideration and this stage should be conducted at the same conditions in all experiments to study the effect of concerned variables alone on cleaning.
- Doing the product recovery stage with water at cold (ambient) temperature increases the amount of product recovered. 12 wt % more toothpaste can be recovered by decreasing the product recovery temperature from 40 °C to 15 °C at the pilot scale. Similarly, the amount of product recovered increases by applying water at higher velocity at the product recovery stage. The same trend is observed between the results of lab and pilot scale experiments. 25 % increase is observed in the weight fraction recovered by increasing the flow velocity from 0.28 m s^{-1} to 1.7 m s^{-1} at the product recovery stage.
- The process conditions applied at the product recovery stage affect the shape of the annular film and this itself influences the cleaning time; the wavier film enhances the cleaning performance. Up to 40 % decrease in cleaning time is observed at the pilot scale. Experiments suggest that the aim of the product recovery stage should be to develop a wavy three-dimensional film which can be rapidly removed.
- Product recovery at cold (ambient) temperature and high velocity is generally observed to result in wavier film, thus causes shorter cleaning times.

8.2 Flow regimes in the cleaning of pipes

After the product recovery stage, removal of a thin film of deposit takes place in cleaning of pipes. The cleaning stages of the thin film of toothpaste from pipes is studied at lab and pilot scale. Toothpaste is chosen as a model deposit which can be a representative of a group of materials which have yield stress. The following conclusions can be drawn from this study:

- Interestingly, cleaning rate of toothpaste shows some similarities to that of whey protein deposits at the low cleaning flow velocities. This can be attributed to the same cohesive-adhesive behaviour of these deposits, that is, adhesive forces between deposits and cleaning surface are stronger than the cohesive forces between the elements of deposits.
- Two distinct cleaning stages are explored in cleaning of the thin film of toothpaste formed after the product recovery stage.
- Chunk removal is typically observed in the 1st cleaning stage, thus removal rates are very high and ca. 95 wt % of the annular film is removed in this stage. Statistical analysis has showed that the cleaning flow velocity is more significant variable, therefore, increasing the temperature of the cleaning water causes energy waste in this stage.
- However, very slow cleaning rate is observed in the 2nd cleaning stage where patches of toothpaste are cleaned by erosion. Although, only ca. 5 wt % of the annular film is left in the 2nd stage, cleaning of it usually takes half of the total cleaning time. Here temperature of the cleaning water is found as the dominating variable by the statistical analysis. This implies that the state or rheology of the deposit should be changed by increasing the temperature to remove the deposit by fluid flow easily at this stage.

- According to the results, a two step alternative cleaning protocol is proposed for the industrial CIP application. The proposed CIP protocol is compared with the traditional CIP protocols currently used in the industry. It reduces the energy consumption by 40 % compared to the hot traditional CIP and decreases the cleaning time by 55 % compared to the cold traditional CIP. The proposed CIP protocol is that; using cleaning water at ambient temperature (20 °C) during the 1st cleaning stage and then using 70 °C cleaning water during the 2nd cleaning stage at the same flow rate (16 m^3h^{-1}). The optek turbidity meter is used to monitor the beginning of the 2nd cleaning stage in the experiments.

8.3 The effect of deposit rheology on the cleaning behaviour and time

Cleaning of various food and personal care products is studied in an aim to understand the effect of deposit rheology on cleaning time and behaviour. The main conclusions obtained from this study are as follows:

- Cleaning behaviours of Newtonian, shear thinning and Herschel-Bulkley fluids are investigated. Yield stress is identified to be the key rheological parameter which mainly affects the cleaning behaviour of a soil. It is observed that yield stress fluids are removed in two distinct cleaning stages. Typical features of the cleaning stages are similar to the findings in Chapter 5, that is, yield stress fluids behave like a solid and are cleaned by fracturing in the 1st stage and slow erosion in the 2nd stage.
- The shear thinning deposit is cleaned faster than Newtonian deposit. However, it takes much longer time to clean the yield stress deposits because the patch removal stage (2nd stage) is not observed in the cleaning of shear thinning and Newtonian deposits.

- Yield stress of a deposit is also found to be the critical rheological parameter by which the cleaning time of the deposit can be predicted.
- It is observed that the effect of temperature on the cleaning of toothpaste is mainly due to change in rheology by increasing temperature. This suggests that the dissolution process has a negligible effect on the cleaning of this deposit and it is cleaned predominantly by fluid mechanical forces.
- A new dimensionless number is found which is a ratio of flow energy of a cleaning fluid to the yield stress of a deposit. It collapses all cleaning time data onto a one line with an R^2 of 0.955 for different deposits, which are cleaned at different velocities. It suggests that the effect of deposit rheology could be quantified by the found dimensionless number and cleaning time of simple soils from straight pipes could be predicted by knowing the yield stress of soils and flow velocity of cleaning water.

8.4 Scale up and characterisation of the flow effect on the cleaning

Cleaning of toothpaste and hand cream from pipes is studied at different scales with the aim of detecting the underlying processes which affect the cleaning process mechanically. Also, the straight pipe section is cleaned after various fittings to quantify the effect of turbulence caused by these fittings on the cleaning. The following conclusions are drawn from this study:

- Reynolds number, flow velocity and wall shear stress alone are proved to have an insufficient relation with cleaning time.
- The dimensionless head loss number is observed to be the best parameter to characterise the flow effect on cleaning, which suggests that head loss calculations in flow might be used to quantify the effect of fluid action on the cleaning process. It is found that straight pipes at different scales are cleaned at similar times as long as this

head loss number is the same in two scales. This also implies that scale up of cleaning could be achieved by calculating the magnitude of head losses in hydrodynamic systems.

- The effect of head loss on cleaning is also identified by studying the cleaning of straight pipe placed just after various fittings. Cleaning time of the test section is found inversely proportional to the magnitude of head loss caused by the fittings; the more the head loss caused by the fittings, the quicker the cleaning process.
- Also a new expression (Equation 7.2) is proposed which allows prediction of cleaning time of a deposit from straight pipes by knowing the following variables; yield stress of a deposit, velocity of the cleaning fluid and the diameter of the pipe. This expression is a ratio between pressure loss (energy loss) per unit length due to friction in straight pipes and yield stress of a deposit. It successfully collapses all cleaning time data onto a one master curve with an R^2 of 0.985 for different deposits which are cleaned at different scales at different velocities.
- The deposit rheology and the fluid mechanical parameters (which are responsible for energy losses) are found as the two main variables which determine cleaning time and behaviour of simple deposits. Therefore, quantification of these variables is valuable for scaling up of cleaning data or can be used to predict and compare cleanability of any system where fluid mechanical removal is the governing process.

8.5 Contributions and future work

The contributions of this study to its field of research include;

- The effect of product recovery stage on the cleaning of fully filled pipes is studied for the first time. The results reveal the considerable effects of this stage on cleaning time of pipes such that cleaning times can decrease by up to 40 % at pilot scale by conducting the product recovery stage at lower temperatures and higher flow velocities of cleaning fluid.

- The techniques and methods are illustrated to identify an alternative CIP protocol for the cleaning of a specific deposit to minimise the cleaning costs. For instance, a two step CIP protocol is proposed for the cleaning of toothpaste from pipes which has resulted in 40 % less energy consumption without significantly deteriorating the cleaning performance compared to the traditional CIP protocols used in the industry.
- A new dimensionless number is defined which allows to quantify the effect of deposit rheology on cleaning. This parameter can be used to predict cleaning time of a deposit by knowing its rheology in a specific hydrodynamic system. Predicting cleaning time by deposit rheology would be very beneficial especially for the multi-product plants where product changeovers require different cleaning strategies for the same equipment.
- Fluid mechanical removal is found as a complex process in the cleaning. Nevertheless, the effect of fluid mechanical actions on cleaning is able to be quantified by the empirical head loss calculations. This indicates that fluid mechanical removal of a deposit happens by the hydrodynamical parameters which cause head losses in flow. Hence, calculating the magnitude of head losses in systems might be used to scale up the cleaning process.

For the future work, waviness of the deposit film can be characterised during cleaning with openable pipes and a systematic study can be conducted to investigate the relation between deposit structure and cleaning process.

In this thesis, generally a Herschel-Bulkley type of deposits are studied and a relationship between cleaning time and yield stress values of deposits is defined in Equation 7.2. This equation may also be valid for the other type of deposits when the yield stress term is replaced with the magnitude of adhesive or cohesive forces of deposits. Therefore, AFM or micromanipulation methods can be used to find adhesive forces, and then the magnitude of these forces can be correlated with the cleaning time of deposits by using this equation. This may allow to predict the cleaning time of all types of deposits at a known cleaning fluid velocity. This can be an area which needs a further investigation.

By Equation 7.2, the relative effect of the dissolution process can also be assessed in the cleaning of complex soils such as milk deposits. After finding the magnitude of adhesive forces in milk deposit at a certain temperature and chemical concentration of the cleaning solution (by using micromanipulation method), the cleaning time can be estimated by the equation by replacing the yield stress term with the found adhesive force values. If the experimental cleaning time of milk deposit is less than the predicted time, it can be deduced that this difference in time is due to the contribution of the dissolution process to the cleaning process.

More experiments (pipes other than circular pipes, etc.) should be conducted along with the CFD studies to check the validity of Equation 7.2 in the other hydrodynamic systems to exploit the results of this thesis for industrial benefit.

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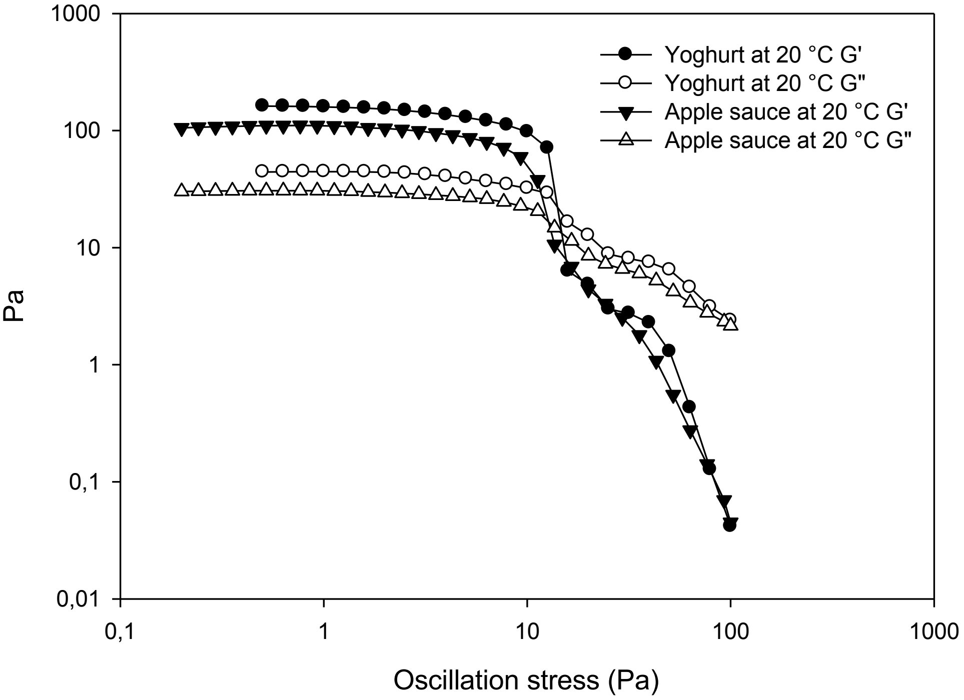
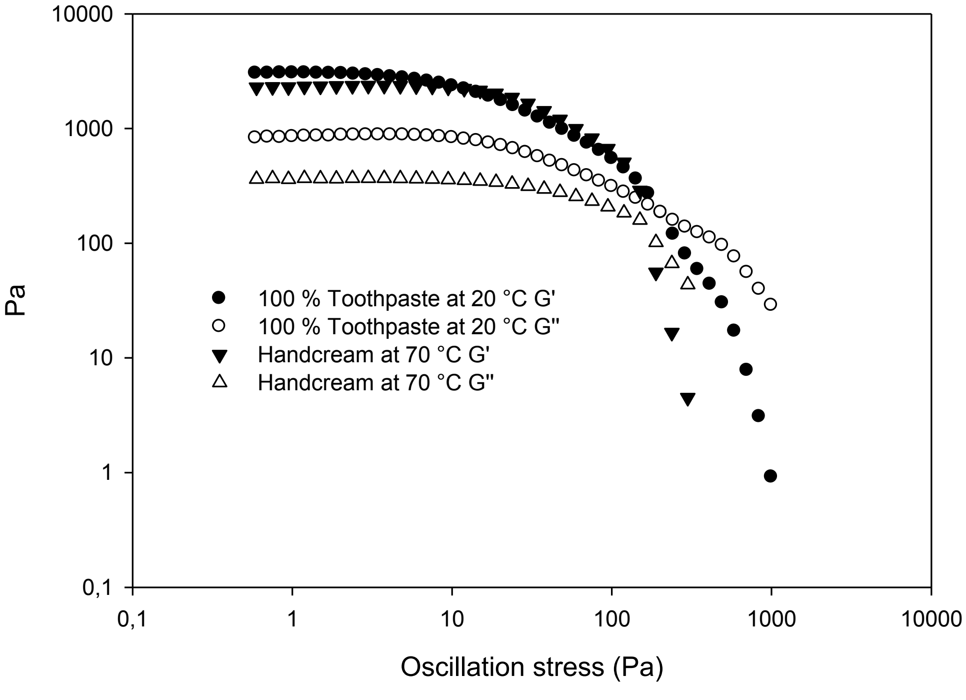
APPENDIX A



a)



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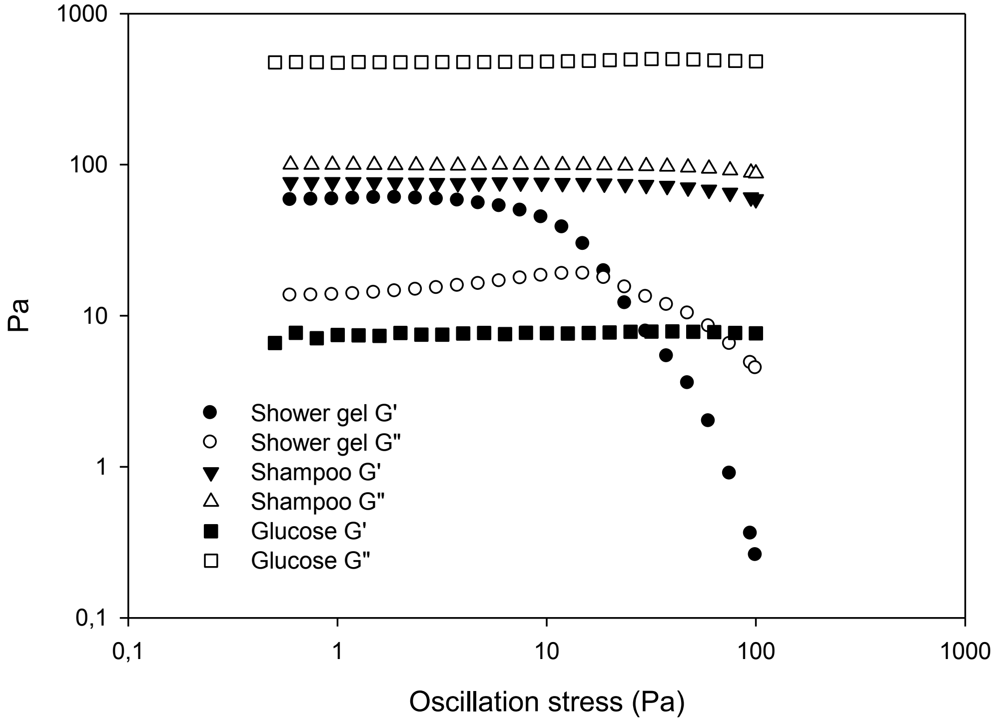

c)

Figure A.1 Oscillatory stress sweep tests to determine the rheological behaviour and yield stress of materials.