A NEW RISK ANALYSIS OF CLEAN-IN-PLACE (CIP) MILK PROCESSING

by

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Addendum

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EXECUTIVE SUMMARY

The food and pharmaceutical industry are generally a nation's largest manufacturing sector – and importantly one of the most stable. *Clean-In-Place* (CIP)[2] is a ubiquitous process in milk processing as thorough cleaning of wet surfaces of equipment is an essential part of daily operations. Faulty cleaning can have serious consequences as milk acts as an excellent substrate in which unwanted micro-organisms can grow and multiply rapidly.

Davey & Cerf (2003) introduced the notion of *Friday 13th Syndrome*[3] i.e. the unexpected failure of a well-operated process plant by novel application of *Uncertainty Failure Modelling* (Davey, 2010; 2011). They showed that failure cannot always be put down to human error or faulty fittings but could be as a result of stochastic changes inside the system itself.

In this study a novel CIP failure model based on the methodology of Davey and co-workers is developed using the published models of Bird & Fryer (1991); Bird (1992) and Xin (2003); Xin, Chen & Ozkan (2004) for the first time. The aim was to gain insight into conditions that may lead to unexpected failure of an otherwise well-operated CIP plant. CIP failure is defined as failure to remove proteinaceous deposits on wet surfaces in the auto-set cleaning time.

The simplified two-stage model of Bird & Fryer (1991) and Bird (1992) was initially investigated. This model requires input of the thickness of the deposit (δ = 0.00015 m) and the temperature and Re of the cleaning solution (1.0-wt% NaOH). The deposit is considered as two layers: an upper layer of swelled deposit which can be removed ($x\delta$) by the shear from the circulating cleaning solution and a lower layer ($y\delta$) that is not yet removable. The output parameters of particular interest are the rate of deposit removal (R) and total cleaning time (t_T) needed to remove the deposit.

The more elaborate three-stage model of Xin (2003) and Xin, Chen & Ozkan (2004) is based on a polymer dissolution process. This model requires input values of temperature of

[2] *see* Appendix A for a definition of some important terms used in this research.

[3] Unexpected (unanticipated) failure in plant or product of a well-operated, well-regulated unit-operation.

the cleaning solution (T), critical mass of the deposit (m_c) and cleaning rate (R_m). The output parameters of particular interest are the rate of removal during swelling and uniform stage (R_{SU}), the rate of removal during decay stage (R_D) and the total cleaning time needed to remove the deposit (t_T). The two CIP models are appropriately formatted and simulations used to validate them as a unit-operation.

A risk factor (p) together with a practical process tolerance is defined in terms of the auto-set CIP time to remove a specified deposit and the actual cleaning time as affected by stochastic changes within the system (t_T'). This is computationally convenient as it can be articulated so that all values $p > 0$ highlight an unwanted outcome i.e. a CIP failure.

Simulations for the continuous CIP unit-operation are carried out using Microsoft Excel™ spreadsheet with an add-in @Risk™ (pronounced 'at risk') version 5.7 (Palisade Corporation) with some 100,000[4] iterations from Monte Carlo sampling of input parameters. A refined Latin Hypercube sampling is used because 'pure' Monte Carlo samplings can both over- and under-sample from various parts of a distribution. Values of the input parameters took one of the two forms. The first was the traditional *Single Value Assessment* (SVA) as defined by Davey (2011) in which a single, 'best guess' or mean value of the parameter is used. The output therefore is a single value. The alternate form was a *Monte Carlo Assessment* (MCA) (Davey, 2011) in which the 'best guess' values take the form of a probability distribution around the mean value. Many thousands of randomly sampled values for each input parameter are obtained using Monte Carlo sampling. Generally, in QRA the input parameters take the form of a distribution of values. The output therefore is a distribution of values with each assigned a probability of actually occurring.

The values of all inputs are carefully chosen for a realistic simulation of CIP.

Results reveal that a continuous CIP unit-operation is actually a mix of successful cleaning operations along with unsuccessful ones, and that these can tip unexpectedly. For example for the unit-operations model of Bird & Fryer (1991) and Bird (1992) failure to remove a

[4] Experience with the models highlighted that stable output values would be obtained with 100,000 iterations (or CIP 'scenarios').

proteinaceous milk deposit (δ = 0.00015 m) can occur unexpectedly in 1.0% of all operations when a tolerance of 6% is allowed on the specified auto-set cleaning time (t_T = 914 s) with a cleaning solution temperature of 60 ^{O}C. Using Xin, Chen & Ozkan (2004) model as the underlying unit-operation some 1.9% of operations at a nominal mid-range cleaning solution temperature of 75 ^{O}C could fail with a tolerance of 2% on the auto-set CIP time (t_T = 448 s).

Extensive analyses of comparisons of the effect of structure of the two CIP unit-operations models on predictions at similar operating conditions i.e. 2% tolerance on the auto-set clean time (~ 656 s) and 1%-sd in the nominal mean temperature of the NaOH cleaning solution at 65 ^{O}C, highlighted that the underlying vulnerability to failure of the simplified model of Bird & Fryer (1991) and Bird (1992) was 1.8 times that of the more elaborate model of Xin (2003) and Xin, Chen & Ozkan (2004).

The failure analysis presented in this thesis represents a significant advance over traditional analysis in that all possible practical scenarios that could exist operationally are computed and rigorous quantitative evidence is produced to show that a continuous CIP plant is actually a mix of failed cleaning operations together with successful ones. This insight is not available from traditional methods (with or without sensitivity analysis). Better design and operating decisions can therefore be made because the engineer has a picture of all possible outcomes.

The quantitative approach and insight presented here can be used to test re-designs to reduce cleaning failure through changes to the plant including improved temperature and auto-set time control methods.

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I trust that the results of my research work justify the expectations and confidence of all the people concerned, and the interest and encouragement of my family, friends and colleagues.

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CHAPTER ONE

INTRODUCTION

The food and pharmaceutical industries are the nation's largest manufacturing sector, and importantly one of the most stable (Davey, 2001). These industries are especially important to Australia as a major food exporter.

Clean-In-Place (CIP) is a technique that was developed in 1950's as a manual process using a balance tank, a centrifugal pump and a connection to the system to be cleaned. Today automated CIP is widely used and is the standard globally for cleaning of process equipment, especially in the dairy industry.

Of particular research interest is the idea that no matter how good the design and operation of plant, there will occasionally be an unexpected failure. Failure in CIP can be a serious risk to public health (with or without fatalities). Failure however cannot always be put down to human error or faulty fittings (Cerf & Davey, 2001; Davey & Cerf, 2003, Roach, 2009; Suddath, 2009; Davey, 2010; 2011). In recent years Davey and co-workers have pioneered new mathematical approaches that can offer insight into process operation and process plant failure (Cerf & Davey, 2001; Davey & Cerf, 2003; Patil, Davey & Daughtry, 2005; Patil, 2006; Davey, 2010; 2011). These approaches are based on a new risk analysis of the effects of uncertainty on vulnerability to process failure.

The principal aim of this research is to assess this new approach to develop a new risk analysis of CIP in milk processing. CIP failure is defined as the failure to remove a proteinaceous deposit in the auto-set cleaning time on wet process surfaces.

The justification for this research is that it will aid a greater understanding of the effects of uncertainty on CIP failures and contribute to improved design and operating conditions for processing.

A logical and stepwise approach that was developed by Davey and co-workers was adapted to this research.

The relevant literature is reviewed in Chapter 2. This chapter first reviews CIP and two theoretical CIP unit-operations models are selected that are amenable to testing. The key notion of uncertainty is introduced and defined.

In Chapters 3 and 4, respectively, the selected models of Bird & Fryer (1991), and, Bird (1992), and; Xin (2003), and, Xin, Chen & Ozkan (2004) are appropriately formatted and validated as CIP unit-operations. Both the traditional and the proposed new, alternative solution are presented.

In Chapter 5 a comparison is made between the selected two models at different values of temperature.

Chapter 6 presents a summary of findings from this research.

Chapter 7 presents the conclusions from this investigation together with suggestions for further research work.

The more important terms used in this study are discussed in Appendix A, and all Nomenclature used in this study are carefully listed separately.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

The food and pharmaceutical industries are some of the most important industries world-wide. CIP is a unit-operation used widely in these industries. Failure in CIP can have enduring effect on public health, with or without fatalities.

The process of cleaning is a complex one. The mechanism of cleaning is not thoroughly understood. Proposals for models to simulate the unit-operation CIP have only lately appeared in the literature. This late development is undoubtedly due to the multidisciplinary nature of cleaning. In the mathematical modelling of CIP process, parameters characterizing the hydrodynamics of flow, temperature and kinetic constants of the cleaning solution (1.0-wt% NaOH) used must be combined with the nature of the 'dirt' deposits. Each of these aspects has been the subject of separate investigations. A comprehensive experimental evaluation and testing of CIP models has only recently been reported in the literature.

The factors that characterize the CIP process are temperature, flow rate, Reynolds number and rate constants. The two main parameters to be determined are temperature and total cleaning time (t_T). Generally, as the value of temperature increases, the value of t_T decreases.

In this chapter the relevant literature is reviewed and CIP unit-operation models are evaluated. Two of these models are selected for further investigation.

2.2 Clean-In-Place (CIP)

During processing, the interior, wet surfaces of process equipment can become fouled with deposits of food and other particles. These deposits can hide microbiological growth and can endanger sterility of the product (Belmar-Beiny, Schreier & Fryer, 1994).

Cleaning is necessary for both microbiological hygiene but also to restore the heat transfer and pressure drop characteristics. In CIP cleaning is generally done using both acid and alkali (sodium hydroxide and nitric or phosphoric acid) as a two-stage process, or, using specially formulated detergents as a single-stage process, with automatic CIP techniques

that have been developed empirically. In the dairy industry the time of non-production dedicated to CIP is excessive and ranges from 4 to 6 h per day (Alvarez, Daufin & Gesan-Guiziou, 2009; Gallot-Lavallee, Lalande & Corrieu, 1983).

CIP is done without disassembly of process equipments (Timperley, 1981; 1989) i.e. *in situ* in the dairy industry.

CIP was first developed in 1950's. Up to then closed systems were disassembled and cleaned manually. The advent of CIP was advantageous because closed systems need not be disassembled. In addition to the dairy industry, industries that rely on CIP include beverage, brewing, processed foods, pharmaceutical and some cosmetic industries (Wilson, 2005).

The aim with CIP is to eliminate all organic deposits, such as precipitated proteins, carbohydrates, fats, minerals and others, which form the nutritional base for the growth of bacteria from wet surfaces (Caroline, Yahaya & Christine, 2010).

CIP is a chemical process dependent on the type and concentration of solutions used and also on solution temperature, time in contact with the substance of deposits (Bird & Bartlett, 1995; Bird, 1994; Bird, Milford & Tucker, 1994). The deposits are dissolved chemically, and the cleaning solution flow velocity must always be sufficient to carry away the dissolved and dislodged particles from the wet surfaces.

Current auto-set CIP cleaning takes about 35 mins. Generally it is carried out in 5-steps (Bird & Espig, 1994):

1. Pre-rinse - The surfaces are first rinsed with water in order to remove any soil deposits. This step takes about 5 mins
2. *Alkali-clean* (detergent wash) - The alkali used (usually, 1.0-wt% sodium hydroxide (NaOH)), dissolves the deposited proteins. The NaOH used also importantly acts as bactericide. This step takes about 15 mins
3. Water rinse - The alkali solution is then flushed using water in this step. This step takes about 3 mins

4. Acid rinse - After alkali removal the wet surfaces are treated with acid (commonly nitric (HNO_3) or phosphoric acid (H_3PO_4)). The acid removes incrusted salts that may have formed. This step takes about 5 mins
5. Final rinse - A final rinse using water is used to remove the acid solution. This step takes about 7 mins.

This 5-step operation is shown schematically in Fig. 2-1. The figure proceeds from left to right on a time-axis.

Following the 5-step CIP operation, disinfection is usually carried out. Disinfection is used to make micro-organisms non-viable (i.e. kill them). Typical disinfecting agents are chlorine (Cl_2) and ozone (O_3). These agents are added through a dosing pump to the cleaning water. Ozone is more powerful compared with chlorine, and is more widely used in industries because of the fact that ozone does not leave behind any residues (Holah, 2003).

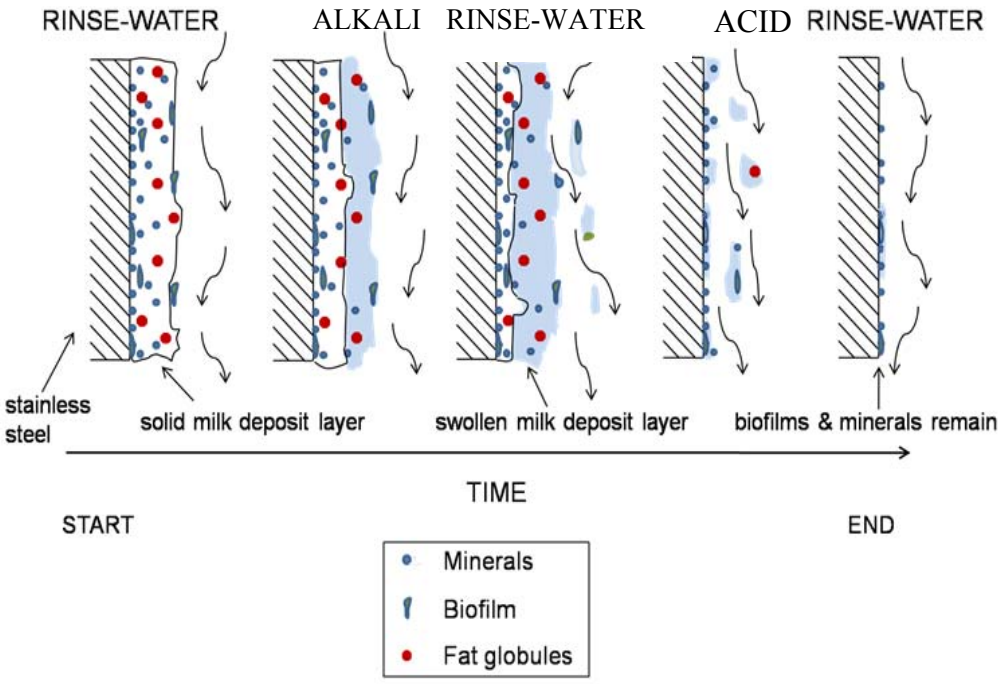

Fig. 2-1: Schematic of wet surface in 5-step CIP operation

2.3 Factors influencing CIP cleaning

The removal rate of deposits is affected by temperature and concentration of the cleaning solution, contact time, pressure and general flow hydrodynamics (Lorenzen & Tuchenhagen, 2005):

1. Temperature - Effective CIP cleaning can be achieved when the temperature of the cleaning solution is around 55 ^{O}C to 70 ^{O}C (Lloyd, 2008; Majoor, 2003).
2. Concentration - The concentration of caustic widely used is 1.0-wt% NaOH (Lloyd, 2008; Majoor, 2003)
3. Pressure - The pressure used with CIP inside pipelines is typically around 4.1 x 10^5 N m^{-2} (60 psig) to 6.9 x 10^5 N m^{-2} (100 psig); and in the case of tanks the pressure should be less than 4.1 x 10^5 N m^{-2} (60 psig) (Lloyd, 2008; Majoor, 2003)
4. Flow - Because CIP relies on shear force removal of material turbulent flow conditions are preferred, generally with Re > 4000 (Lloyd, 2008; Majoor, 2003).

2.4 Types of CIP systems

There are three types of CIP systems:

1. Single-use system – These use the cleaning solution only once. These are small units that are located adjacent to the system that has to be cleaned and sanitized. Heavily soiled equipment is suited to a single-use system as the cleaning solution and rinse water will be used only once
2. Re-use system – In the re-use system the detergent or acid is recovered and is used as many times as possible
3. Multi-use system – Multi-use systems combine the features of both single-use and re-use systems and are designed for cleaning the wet surfaces.

2.4.1 Comparison between different CIP systems

A summary comparison of the three different CIP systems is provided in Table 2-1. It can be seen that the Multi-use system is advantageous and is therefore widely used in the dairy industries.

Table 2-1: Comparison of different CIP systems (*Adapted* from Majoor, 2003)

NOTE:
This table is included on page 9 of the print copy of the thesis held in the University of Adelaide Library.

2.5 Equipment design types

According to the European Hygienic Equipment Design Group (EHEDG), hygienic design of equipment can be divided into two types (Fryer & Asteriadou, 2009; Fryer, Robbins & Asteriadou, 2011):

1. Hard to Reach Places (HTRP)
2. Equipment design that must be dismantled to permit cleaning.

HTRP's refer to places where the flow of cleaning solution is too low, or there is no flow, to remove deposits in these regions. In extreme cases these deposits cannot be removed. Examples include a tee-joint; hole or crevice on a surface; imperfect welds; and improper alignment of sections (often after maintenance procedures).

The second types of equipment are those that can be cleaned only after dismantling. These equipments are certified as ‘Class 2’ by the EHEDG.

2.6 CIP failure seen from food process engineering and hygiene viewpoint

As fouling on equipment wet surfaces increases it in-turn reduces the heat and mass transfer characteristics and pressure drop. Proper cleaning must be done in order to retain the original characteristics of the process equipment. The aim with CIP is to remove all fouled substances (proteins, minerals or micro-organisms) inside the process equipment and to maintain performance of the plant equipment and hygiene (Gillham et al., 1999).

CIP failure can be caused when the cleaning solution is not of design concentration, or due to low temperature, insufficient time or inefficient temperature/time combinations. This type of failure is known as ‘Type-1 failure’ (Brigitte Carpentier, *Laboratoire de securite sanitaire de Maisons-Alfort*, France, *pers*. *comm*.).

From food hygiene view point the target of CIP is to reduce the concentration of potentially pathogenic or food spoiler micro-organisms to an acceptable level. This type of failure due to unwanted micro-organisms is called as ‘Type-2 failure’.

It has to be noted that ‘Type-2 failure’ is caused by ‘Type-1 failure’. ‘Type-2 failure’ also occurs when CIP although properly designed to avoid ‘Type-1 failure’ is ineffective against certain species of micro-organisms in the food product.

If micro-organisms are known to be present, the equipment should be sanitized before the start of processing. A third type of failure is the germination of spores during the time of heat treatment. This type of failure is called ‘Type-3 failure’.

2.6.1 Causes of failure

Some factors that can be responsible for these types of failures are: low flow rate, low temperature, insufficient time and diluted concentration of cleaning solution.

2.7 Advantages of CIP

The main advantages of CIP over other cleaning methods (manual scrubbing) are:

1. Reduced water usage
2. Reduced sanitiser consumption
3. Reduced operating cost
4. Reduced waste water costs
5. Increased efficiency
6. Reduced cycle times
7. Increased process time.

2.8 Milk Fouling

Fouling refers to the accumulation of unwanted materials on process surfaces, i.e. those of the wet environment.

Deposits formed during fouling are of two types:

1. Proteinaceous deposits
2. Mineral deposits.

Usually the first layer will be followed by proteinaceous deposits followed by mineral deposits (Belmar-Beiny & Fryer, 1993; Bansal & Chen, 2006; Visser & Jeurnink, 1997).
The fouling material of the deposit can consist of either living micro-organisms (bio-fouling) (Chmielewski & Frank, 2003) or a non living substance (organic or inorganic fouling). The process by which micro-organisms get attached to the surface layer of the equipment is called ‘soiling’.

Process equipment that commonly gets affected by fouling includes heat exchangers and pipelines (Lelieveld, 2003). When these are fouled it leads to reduction of the thermal efficiency of the exchanger (i.e. increase in hot side temperature, decrease in cold side temperature, corrosion and increase in use of cooling water). When pipelines foul it leads to an unwanted increase in pressure drop, upstream temperature, energy expenditure, slugging, cavitation and blockage (Lalande, Rene & Tissier, 1989).

Because of the widespread processing of milk, milk fouling is of particular importance. Milk fouling takes place by two methods namely, Type A fouling and Type B fouling (Changani, Belmar-Beiny & Fryer, 1997; Fryer, 1997; Fryer & Christian, 2005; Yoo, Chen & Bansal, 2005; Fryer, Christian & Liu, 2006).

Type A fouling takes place when the temperature of milk is around 75°C to 110°C. The proteinaceous deposits are white, soft and spongy. The deposit consists of 50 % to 70 % proteins, 30 % to 40 % minerals, 4 % to 8 % fat. Whey proteins have 5 % of milk solids and account for more than 50 % of the fouling deposits in Type A fouling. β-Lactoglobulin and α-Lactalbumin are the two major whey proteins. The most dominant is β-Lactoglobulin.

Type B fouling takes place when the temperature of the milk is above 110°C. The deposits are gray, hard, compact and granular in structure. The deposit consists of 70 % to 80 % minerals (mainly calcium phosphate) 15 % to 20 % proteins and 4 % to 8 % fat.

The time that is required for the formation of protein aggregates or insoluble mineral complexes before noticeable amounts of deposits are formed is called as *induction time*.

Fouling also takes place due to Maillard reaction (also called browning) and caramelization (Perez-Locas, 2008).

Maillard reaction is a chemical reaction between amino acid and a reducing sugar, usually requiring heat. This reaction was first discovered in the year 1910 by Louis Camille Maillard, and was named after him. Caramelization is the oxidation of sugar, a process used extensively in cooking for the resulting nutty flavor and brown color. As the process occurs, volatile chemicals are released, producing the characteristic caramel flavor. Both Maillard and caramelization are types of non enzymatic browning. However, unlike the Maillard reaction caramelization is pyrolysis, as opposed to reaction with amino acids.

2.9 Biofilms

A *biofilm* is a collection of micro-organisms surrounded by the slime they secrete, attached to either an inert or living surface (Carpentier & Cerf, 1993; 2011; Sharma & Anand, 2002;

Carpentier, 2009). It can also be defined as: 'In order to survive hostile environmental factors such as heat and chemicals microbes in micro colonies have a tendency to form protective extracellular matrices which mainly consists of polysaccharides and glycoprotein and are called 'biofilms' (Wirtanen, 1995; Wirtanen & Salo, 2005).

The micro-organism gets attached to the surface and leads to the formation of biofilms. The first stage in the formation of biofilms is the formation of micro-colonies which occur under suitable conditions both inert and living (Speers & Gilmour, 1985). Formation of biofilms starts with the attachment of free floating micro-organisms to the surface. They attach to the surface of the equipment by means of Vander-Waals forces (Flint, Bremer & Brooks, 1997; Gentil, Sylla & Faille, 2009). Micro-organisms can start up this formation when there is moisture or water available (Bryer, 2000). It has also been found that temperatures below 50 ^{O}C promote the growth of biofilms (Miller & Bott, 1982).

Some common micro-organisms that lead to the formation of biofilms are *Pseudomonas aeruginosa*, *Streptococcus thermophilus* and *Bacillus subtilus* (Klijn et al., 1997; Lelievre et al., 2002; Hornstra et al., 2007). It is reported by Somers, Schoeni & Wong (1994); Griffiths (2003) and Stopforth et al. (2003) that pathogens such as *Escherichia coli*, *Listeria monocytogenes*, *Salmonella* Typhimurium, *Campylobacter jejuni* and *Yersinia enterocolitica* can easily produce biofilms or be a part of biofilm community that cause severe cleaning and disinfection problems on surfaces in food industry. Furukawa et al. (2010) studied the removal of *Staphylococcus aureus* and *Escherichia coli* micro-organisms from stainless steel surfaces using cleaning agents.

There are two types of bacteria, Gram-positive and Gram-negative bacteria. Pasteurized milk is commonly contaminated with Gram-negative psychotropic bacteria (Meynell & Meynell, 1970; Bailey & Ollis, 1986; Austin & Bergeron, 1995; Bremer, Fillery & McQuillan, 2006).

The most concerning issue is that the micro-organisms should not form endospores (Aiba, Humphrey & Millis, 1973). Spores are the dormant forms of the cell, capable of resisting heat, radiation and poisonous chemicals. When spores are returned to the surrounding suitable for cell function, they can germinate to give normal functioning cells. This

biologically active state is often called the vegetative form, which is different from the spore.

2.10 Growth of micro-organisms

Micro-organisms can grow on process equipment surfaces wherever there is humidity (Brown, 1996). Micro-organisms can survive from a temperature range of a few degrees below zero ^{O}C up to 65 ^{O}C. If these micro-organisms are not removed properly they adhere, colonize the surface (Mittelmann, 1998).

Importantly, the term biofilm means a continuous layer of bacterial cells with one or more superposed layers attached to it. It can however be a confusing term because, in reality, bacteria do not exist as a biofilm over the equipment surface, rather they will be present as spots throughout the equipment surface (Brigitte Carpentier, *Laboratoire de securite sanitaire de Maisons-Alfort*, France*, pers. comm*.). Throughout therefore in this thesis the term biofilm is not used.

2.11 CIP process models

Fouling problems associated with milk processing have been reported for more than half a century. Fouling occurs in milk processing plants mainly due to denaturation of proteinaceous deposits and the decrease in solubility of milk salts with increasing temperature. The CIP models developed so far have a common origin; the process of cleaning will be a function of the system thermo-hydraulics (temperature and flow rate) and the system chemistry (cleaning solution formulation and concentration).

Table 2-2 presents a chronological listing of nine (9) process models proposed to date in the referred literature for simulation of CIP unit-operation. The contributions made by the various investigators are reviewed in this section in the order tabulated.

Jennings (1965) was the first to develop an analytical CIP cleaning model that can be thought of as a traditional unit-operation. Jennings used a rate law that is first-order in both deposit mass per unit area and the concentration of cleaning solution. Jennings (1965) found that the rate of reaction is zero-order deposit concentration and first-order for

cleaning solution concentration whilst studying the removal of whole milk deposits from stainless steel surfaces using NaOH as a cleaning solution. The model can be summarized by:

$$\frac{dm}{dt} = -k_2 cm \tag{2.1}$$

Equation (2.1) is of first-order where m is the mass per unit area of the deposit and c is the concentration of the cleaning solution used. Factors governing the mechanism of removal are therefore deposit mass and cleaning solution concentration.

Table 2-2: Summary and chronological listing of models available for CIP unit-operation

Author(s)	Proposal
Jennings (1965)	First-order model for CIP removal milk deposits
Harper (1972)	Proposed first multi-stage cleaning mechanism for removal of proteinaceous milk deposits from hard surfaces using chemical cleaning solutions
Schlussler (1976)	Zero-order model for dried milk deposits removal
Gallot-Lavallee, Lalande & Corrieu (1984); Gallot-Lavallee & Lalande (1985)	Turbulent flow model for hard surfaces soiled with whole milk deposits; 2-wt% NaOH as cleaning solution
Perlat (1986)	4-stage cleaning model similar to Gallot-Lavallee & Lalande (1985)
Bird & Fryer (1991); Bird (1992)	2-stage (swelling & removal stage) CIP mechanism for whey and whole milk protein deposits; extensive experimental testing for *Alkali-clean* step (1.0-wt% NaOH and 2.0-wt% NaOH); 30 ^{O}C – 70 ^{O}C
Gillham et al. (1999)	First to highlight that the process of cleaning takes place in 3-stages (swelling, uniform and removal stage) under turbulent flow (Re > 4000); 0.5-wt% NaOH as cleaning solution
Xin (2003); Xin, Chen & Ozkan (2004)	3-stage (swelling, uniform and removal stage) cleaning mechanism for removal of whey protein concentrate (WPC) gel as model foulant; extensive experimental testing for *Alkali-clean* (0.5-wt% NaOH) step
Popovic, Tekic & Djuric (2009)	5-parameter model for alkali cleaning and 6-parameter model for detergent cleaning; 0.2-wt% and 1.0-wt% NaOH; not directly relevant to milk fouling on wet food-grade steel surfaces

Harper (1972) was the first to postulate a multi-stage cleaning mechanism. This mechanism is very general and has not permitted simple kinetic equations to be obtained. It is a useful model as it breaks down the problem into a series of more manageable pieces that can be modelled readily.

The process of cleaning suggested by Harper (1972) takes place in the following four steps:

1. Bringing the cleaning solution into intimate contact with the deposit by good wetting and penetrating properties
2. Displacement of solid and liquid soils from the surface by saponifying fats, peptizing proteins and dissolving minerals
3. Dispersion of deposit in the solvent by dispersion, deflocculation or emulsification
4. Prevention of re-deposition of the dispersed deposit back onto the cleaned surface by providing good rinsing properties.

Schlussler (1976) studied the removal of milk proteinaceous deposits from stainless steel and glass surfaces using NaOH as cleaning solution, and found that the removal rates were zero-order with respect to both the amount of deposit and the cleaning solution concentration for caustic solutions above 1-wt%. Schlussler (1976) modelled deposit removal in terms of deposit per unit surface area and developed a zero-order model for milk deposit removal. The model can be explained by the use of following equation:

$$\frac{dm}{dt} = -k_0 \qquad (2.2)$$

Equation (2.2) is of zero-order where *m* stands for the mass per unit area of the deposit. Deposit mass was the only parameter used governing the mechanism of cleaning. If the process of removal is zero-order in deposit concentration it suggests that the deposits are removed uniformly from the solid-liquid interface; this is only possible if the deposit is continuous and of a uniform concentration. In this case the deposit thickness has no effect on the rate of removal from hard surfaces.

The multi-stage cleaning model of Harper (1972) was greatly improved by Gallot-Lavallee, Lalande & Corrieu (1984); Gallot-Lavallee & Lalande (1985) and Perlat (1986). Their models are the most complex in the literature to describe the process of cleaning prior to the current work. Their models consist of 4-differential equations that have to be solved manually.

Gallot-Lavallee, Lalande & Corrieu (1984) and Gallot-Lavallee & Lalande (1985) developed a 4-stage turbulent flow CIP model for hard surfaces. Diffusion is the rate controlling step in this model. Whole milk proteinaceous deposits were used as model foulant, containing 60% proteins, 30% fats and 10% minerals (i.e. Type A deposits). These models are based on the assumption the deposit is changed by the concentration of hydroxyl (OH^-) ions from an initial state to an intermediate state prior to removal.

The cleaning process comprises four stages:

1. Mass transfer of OH^- ions from the bulk to the deposit
2. Diffusion of OH^- ions through swelled deposit
3. Reaction with unswelled deposits
4. Removal of swelled deposit.

The cleaning solution used was 2-wt% NaOH. Extensive experiments were carried out over a temperature range of 55 ^{O}C to 95 ^{O}C. Turbulent flow conditions (Re > 4000) and a good flow velocity (0.3 m s^{-1} to 3.0 m s^{-1}) were used. In this CIP model the cleaning rate (R) increased rapidly at the beginning of cleaning and reaches a maximum from which it gradually decreases to zero. Factors governing the mechanism of cleaning in this model are deposit mass, cleaning solution concentration, cleaning solution temperature and flow rate. This is the first cleaning model that takes the effect of temperature into consideration (temperature dependent); this is the main advantage of this model.

Perlat (1986) developed a 4-stage turbulent flow cleaning model for removal of whole milk proteinaceous deposits from stainless steel surfaces. This model is much similar to the model proposed by Gallot-Lavallee, Lalande & Corrieu (1984) and Gallot-Lavallee & Lalande (1985).

Bird & Fryer (1991) and Bird (1992) developed a 2-stage CIP model to remove whey and whole milk proteinaceous deposits from stainless steel surfaces. This model is diffusion controlled. According to this model cleaning takes place in 2 stages:

1. Swelling stage - the stage in which the cleaning solution converts the deposits into a form by which it can be removed
2. Removal stage - the stage in which the swelled deposits are removed.

The factors that govern this model are cleaning solution temperature, flow and removal rate constants. Extensive experiments were carried out at over a temperature range of 30 ^{O}C to 70 ^{O}C. These authors used 1.0-wt% NaOH as the cleaning solution.

This model is well-suited in terms of analysis and reproducible experimental testing of the *Alkali-clean* (detergent wash) step[5] on a uniform proteinaceous deposit from milk. It can also be appropriately formatted as a CIP unit-operation. This simplified model was therefore selected for an initial new risk analysis using uncertainty modelling techniques (Chapter 3).

A drawback however with this CIP model of Bird & Fryer (1991) and Bird (1992) is that the turbulent flow (Re > 4000) conditions cannot generally be achieved and cleaning solution flow velocity is limited to around 0.174 m s^{-1}.

This drawback can be overcome through the CIP unit-operation model developed by Xin (2003) and Xin, Chen & Ozkan (2004). Xin (2003) and Xin, Chen & Ozkan (2004) developed a 3-stage cleaning model. These authors used Whey Protein Concentrate (WPC) gel as a model foulant. Diffusion is the rate controlling step. The cleaning solution used is 0.5-wt% NaOH solution. According to this model the process of cleaning takes place in 3 stages:

1. Swelling stage - stage in which cleaning liquid converts the deposits into a form by which it can be removed
2. Uniform stage - stage in which the rate of removal remains constant
3. Removal stage - stage in which the swelled deposits are removed.

[5] *see* Section 2.2.

This model is similar to the Bird & Fryer (1991) and Bird (1992) model except that there is a uniform stage[6] present. This CIP model is temperature dependent and extensive experiments were carried out at a temperature range reported as 303 K (35 ^{O}C) to 373 K (100 ^{O}C). The absolute temperature values are used for the purpose of unit-consistency in solving this CIP model. The governing factors are temperature, cleaning solution flow velocity, deposit mass, rate constants and reptation time.

The advantage of this model when compared with Bird & Fryer (1991) and Bird (1992) unit-operation model is that using this model as an underlying unit-operation the authors were able to achieve turbulent flow conditions (Re > 4000), but with a cleaning solution flow velocity in the lower range 0.07 m s^{-1} to 0.62 m s^{-1}.

This more complex model was therefore also selected for an advanced risk analysis. This is reported in detail in Chapter 4. A comparison with that of the CIP model of Bird & Fryer (1991) and Bird (1992) at different temperature values is presented in Chapter 5.

Popovic, Tekic & Djuric (2009) proposed a 5-parameter cleaning model for alkali cleaning and a 6-parameter cleaning model for detergent cleaning for removal of whey proteinaceous deposits. This model estimates how pore diameter and related thickness of the in-pore deposit change with time. This model is not temperature dependent.

These authors used a ceramic membrane fouled with whey proteins as a model. The cleaning solution used was 0.2-wt % and 1.0-wt % NaOH solution and a detergent solution (Ultrasil®). This model is not discussed further because it is not directly relevant to milk fouling on wet food-grade steel surfaces.

A direct comparison can be made of the experimental conditions used in the models developed by Gallot-Lavallee, Lalande & Corrieu (1984), Gallot-Lavallee & Lalande (1985); Bird & Fryer (1991) and Bird (1992); 3. Gillham et al. (1999), and; Xin (2003) and Xin, Chen & Ozkan (2004), Table 2-3.

[6] The stage during which the rate of removal of proteinaceous deposits remains constant.

It can be seen from the table that the experimental conditions for these CIP models are very similar. Because of this a direct comparison can be made, especially with the selected models of Bird & Fryer (1991) and Bird (1992), and Xin, Chen & Ozkan (2004).

Solution of these CIP models can be carried out in two ways.

The first is through traditional Single Value Assessment (SVA) modelling (Davey & Cerf, 2003). This is the most widespread method in the literature where a single 'best guess' estimate or a mean value of an input parameter is used. The output is a single value.

Table 2-3: Comparison of experimental conditions of selected CIP models

CIP Model	Gallot-Lavallee Lalande & Corrieu (1984)	Bird & Fryer (1991) and Bird (1992)	Gillham et al. (1999)	Xin, Chen & Ozkan (2004)
Cleaning tube	L = 250 mm d_i = 16 mm	L = 150 mm d_i = 6.35 mm	L = 100 mm d_i = 6 mm	L = 150 mm d_i =16 mm
Foulant	Raw whole milk	Whole milk and WPC*	WPC 35% proteins	WPC
Formation conditions	T = 85 ^{O}C u = 0.6 m s^{-1} t = 15 mins	T = 70 ^{O}C u = 0.175 m s^{-1} t = 8 mins	T = 50 ^{O}C Re = 1520 u = 0.5 m s^{-1} t = 22 mins	0.5-wt% NaOH
Composition of deposit	60-wt% proteins 30-wt% fats 10-wt% minerals	90% proteins	35% proteins	25-wt% WPC solid
Cleaning conditions	T = 55-95 ^{O}C Re = 10000 - 70000 u = 0.3 – 1.9 m s^{-1} 2-wt% NaOH	T = 30-70 ^{O}C Re = 1270 – 2500 u = 0.6 m s^{-1} 0.5-wt% and 1.0-wt% NaOH	T = 20-80 ^{O}C Re = 500 - 5000 0.5-wt% NaOH	T = 35-85 ^{O}C Re = 2400 – 21000 u = 0.07 – 0.62 m s^{-1}
Method	Optical sensor	Bradford Protein Assay	Bradford Protein Assay	UV spectrophotometric method

* WPC = whey protein concentrate

The second is the Monte Carlo Assessment (MCA) modelling. In this approach, the input parameter values take the form of a distribution (e.g. Cerf & Davey, 2001; Davey, 2011). The output is a distribution of values.

Traditional Single Value Assessment (SVA) modelling is reviewed first in the following section.

2.12 Single value assessment (SVA) modelling

The traditional solution to the CIP unit-operation model taken by biochemical engineers (and others) is a single point, stochastic and deterministic approach. Cerf & Davey (2001) defined this approach as a Single Value Assessment (SVA).

SVA involves using a single value or 'best guess' estimate of the value of each parameter such as temperature, removal constant or velocity, to obtain a single value predictive outcome for the output parameter such as deposit removal rate, maximum removal rate or time to reach the maximum removal rate.

It can be seen that this is another way of saying the usual approach is using a sensitivity analysis for each input parameter – in which a small amount of variability (say $\pm$ 1-5 %) is introduced around the mean value.

In the SVA, the model input parameters are linked with each other as well as with the output parameters via the usual mathematical functions e.g. multiplication, addition, subtraction, exponentiation etc. The equations are then solved. This can be done using software for mathematical modelling for e.g. Microsoft Excel^{TM} spreadsheet.

Almost all unit-operations used in chemical engineering can be solved using this traditional SVA approach (Sinnott, 2005).

2.13 Monte Carlo Assessment (MCA) modelling

In contrast to the SVA is the Monte Carlo Assessment (MCA). This takes into account all possible values that the input parameters may take (Vose, 2008). Input parameters for the MCA are a distribution therefore of possible values (with the probability of each occurring). These are linked via the usual mathematical functions as in the case in SVA. This is because a distribution of values together with the probability of each occurring is given. The mean value of MCA will often be the SVA.

Cerf & Davey (2001) used MCA sampling in a new risk analysis of a UHT milk *sterilisation* unit-operation. Davey & Cerf (2003) pioneered its application to understanding of vulnerability to failure of otherwise well-maintained, well–operated plant (*see* Section 2.16).

An MCA can be defined as 'A stepwise analysis of hazards that may be associated with a particular type of food product, permitting an estimation of the probability of occurrence of adverse effects on health from consuming the product in question' (Notermans & Mead, 1996; Giaccone & Ferri, 2005).

MCA is a relatively new field – and one that is almost certain to grow rapidly in chemical and biochemical engineering (Davey *pers. comm.*). MCA was tried out in the year 1960 – but because of limitations in available software and computer programming, and necessary hardware, it all died out. However, it re-emerged in the mid 1990's when computing became more widespread and available (Vose, 2008).

Importantly for engineers and applied researchers, QRA is not to be considered as the same thing as *HAZOP* studies or *HACCP*. The fundamental principle of QRA is that Uncertainty and Variability are the two components that will enable precise prediction of future events.

Uncertainty - This is defined as a lack of knowledge, or level of ignorance, about the parameters that characterize the physical (process) system. Uncertainty is sometimes reducible through further measurement or careful study, or through consulting more experts. Uncertainty is essentially a statement with which any logical person should agree given the same information (Vose, 2008).

Variability - This is the effect of Chance – and is a function of the system. Variability is not reducible through further study or careful measurement, and can only be reduced by changing the physical system (Vose, 2008).

Total uncertainty - This is a combination of the two ideas i.e. Uncertainty and Variability, which influence the ability to predict future events.

Uncertainty and Variability might be called Fact and Chance respectively (Vose, 2008).

Uncertainty and Variability are separated in risk modelling so as to observe how both contribute to the risk model (Vose, 2008; 1998). Separating Fact and Chance is therefore important to understand process behavior – and to avoid large errors that could easily result in unexpected process failure.

2.14 Various risk assessment techniques

There are four other main risk assessment techniques used in the food and pharmaceutical industries (Davey, 2010). These are:

1. Microbiological risk assessment (MRA)
2. HAZard and Operability (HAZOP) studies
3. Hazard Analysis Critical Control Point (HACCP)
4. Reliability Engineering (RE).

Risk assessment is defined in Codex Alimentarius Commission (CAC, 1998) as 'A scientifically based process consisting of the following steps: (i) hazard identification (ii) hazard characterization (iii) exposure assessment and (iv) risk characterization' (Jaykus, 1996). There is no difference between the hazard characterization step and the risk characterization step. The term 'risk' here actually represents the term 'hazard' (Davey, 2010).

Hazard and Operability Studies (HAZOP) is a 'systematic, structured approach to questioning the sequential stages of a proposed operation in order to optimize the efficiency and the management of risk' (Swann & Preston, 1995). However, Swann & Preston (1995) underscored the problem with HAZOP actions is that they are created at a stage when detailed design is under way, and to make a number of changes at this stage is inevitably expensive and causes potential delay, and these changes could be expensive, they have also stated that HAZOPs are impracticable.

Whiting & Buchanan (1997) highlighted that HACCP is the more widely recognized. It focuses on identifying and controlling the key process steps that most significantly affect the safety of production. HACCP, Hazard Analysis Critical Control Point is a systematic approach to produce acceptable, safe product based on identification and management of

critical control points[7] (Notermans & Mead, 1996; Notermans et al., 1995). HACCP was apparently developed by National Aeronautics and Space Administration (NASA) in the 1960's to help prevent food poisoning with astronauts. However, Whiting & Buchanan (1997) pointed out that as HACCP has become more widely adopted, it has become evident that there are areas within this approach that could be strengthened if researchers were able to quantitatively link product attributes with public health concerns.

Reliability Engineering is a widely used capability to predict something to 'fail-well' that is, to fail expectedly without catastrophic consequences (O'Connor, Newton & Bromley, 2002).

These currently used risk assessment techniques however have many drawbacks: they do not deal with quantitative assessment of a process; and, do not provide insight into unanticipated (un-expected) plant failure which can have catastrophic impacts. Whenever such failures happen it is 'human error' or 'faulty fittings' is often blamed (Cerf & Davey, 2001; Davey & Cerf, 2003). This reasoning is not convincing as these are un-testable hypotheses (Davey, 2011).

2.15 Insight offered by MCA

The key insight offered through MCA modelling for any unit-operation over current widely used methods is the idea that (Davey & Cerf, 2003; Davey, 2011):

'*the combined effect of a series of small changes in input parameters can unexpectedly accumulate in one direction to leverage significant and unexpected change in process output'*

MCA attaches a practical likelihood, or probability, of any event occurring. It accounts for Uncertainty and Variability in the model input parameters by using repeated sampling from a distribution of values of an input parameter, and provides a framework to evaluate the influence of a variety of input parameters on the process efficacy.

[7] A critical control point is defined as any point or procedure in a specific food system where loss of control may result in an unacceptable health risk; it is a point where loss of control may result in failure to meet (non-critical) quality specifications.

MCA modelling has been applied to a unit-operation for a simplified fermenter (Patil, 2006; Patil, Davey & Daughtry, 2005) and UHT steriliser (Cerf & Davey, 2001; Davey & Cerf, 2003). This work has been summarized by Davey (2011).

It has been suggested that MCA modelling could be developed as a novel analysis to be used widely in the discipline of chemical engineering (Davey, 2010).

Models that account for Uncertainty and Variability in a system are referred to as *stochastic*[8] models. In *microbiological process modelling* (Davey & Cerf, 2003), the stochastic predictive models are used to define the growth kinetics of the micro-organism and predict behavior of the micro-organism under various environmental conditions.

The different components and stages of risk assessment are linked together by the usual mathematical relationships and Variability in inputs at each stage is propagated throughout to the final output.

The output is also expressed in the form of a probability distribution. This may give a better and more practical representation of the risk being assessed than is the current use of a SVA or 'best guess' estimate.

Repeated sampling of values of the distribution of input parameters is carried out using the Monte Carlo sampling method.

2.16 Uncertainty modelling using refined Monte Carlo sampling techniques

Within MCA, the Monte Carlo approach is used as a random sampling technique for solving deterministic equations. MCA replaces single value inputs with probability distributions of the input parameters. This involves random sampling of each probability distribution within a parameter to produce 100's or even 1000's of iterations. Each probability distribution is sampled in a manner that reproduces the shape of the distribution. The distribution of the values calculated for the parameter outcome therefore reflects the probability of the values that could occur practically in plant operation. The

[8] *see* Appendix A.

effect of distributions of the values in each of the model parameters is therefore highlighted through MCA with a consequent distribution of practical values (Vose, 2008). The characterization of Uncertainty using MCA allows the decision makers to choose whether to actively reduce an exposure or to conduct an additional research to study the impact of Variability in the risk factors on the output. The main advantage of using MCA is that the simulations are carried out in a repeated manner. This yields important insight into the sensitivity of the model to the variations in the model input parameters, as well as into the likelihood of occurrence of any particular outcome. It is therefore possible to represent Uncertainty in the output of a model by generating sample values for the model inputs, and running the model in a repetitive manner.

An uncertainty model was first applied to a unit-operation in the chemical – biochemical engineering literature by Cerf & Davey (2001) to explain the unexpected failure of a well-operated UHT milk process plant. Failure was defined as a non-sterile milk pack. *Bacillus stearothermophilus* and *Bacillus thermodurans* were used as contaminant spores. Failure of sterlisation with these micro-organisms could be a serious risk to public health. The concentration of contaminant spores, thermal resistance of the spores, heating temperature and residence time of the milk in the steriliser were identified as the process input parameters. Davey & Cerf (2003) illustrated the effect of distribution of values in each of those in the UHT process. This was highlighted with a distribution of practical values o the process input parameters.

In 2003, Davey & Cerf described unexpected failure as *Friday 13th Syndrome* (*Fr 13*) or *uncertainty modelling*. By this, it was meant that there will be failures despite all efforts in a well-operated, well-maintained plant due to Chance. Davey & Cerf (2003) noted that one reason that these *Fr 13* events are rare is that most commercial sterilisations involve over treatment, which is not only wasteful in terms of cost and energy, but also diminishes the nutritional and sensory qualities of the product (*see* Davey & Cerf , 1996).

Clearly, the idea of *Fr 13* modelling has been accepted in the literature but more traditional chemical engineers however do not like this descriptor as it underscore the effect of Chance, preferring a more deterministic and traditional SVA approach. *Vulnerability to failure* or *uncertainty modelling* has been suggested (Davey K R *pers*. *comm*.) and will be used in future sections of this thesis.

A practical upshot of vulnerability to failure modelling used by Davey & Cerf (2003) was that predictions showed a higher proportion of the number of UHT milk packs would be non-sterile than was predicted by the traditional SVA. This number of non-sterile packs was more or less the number that was anecdotally known to be found non-sterile in well-operated UHT process plant, between 1 and 4 in 10^4. Davey & Cerf (2003) concluded that the occurrence of a fixed number of non sterile milk operations associated worldwide with the UHT process plant, and greater than that predicted by the SVA, is the failure to take into account a distribution of values for each of the process parameters.

In 2006, Patil, Davey & Daughtry applied the technique of *Fr 13* modelling to fermentation, another widely used unit operation in chemical – biochemical engineering. The findings showed that a practical fermenter could exhibit vulnerability to failure, as unexpected fermenter washout, as a result of small, naturally occurring uncertainty and variability in the microbiological input parameters for the micro-organism. This practical insight into an otherwise well-operated continuous fermenter contrasted sharply with the more traditionally used SVA analysis in which the natural variability in microbiological parameters was simply not accounted for.

Davey (2010) developed a 5-step algorithm for uncertainty modelling. This is thought to be applicable to any unit-operation, although not tested for CIP.

2.17 Methodology for vulnerability to failure modelling

The 5-step algorithm pioneered by Davey (2010) adapted to CIP is:

1. Structure CIP cleaning as an identifiable hygienic unit-operation and establish a clear definition(s) of failure
2. Identify key parameters on failure(s) using traditional engineering SVA approaches
3. Derive, investigate and test plausible probability distributions for key CIP parameters
4. Simulate CIP and likely failure(s) using uncertainty modelling together with a refined Monte-Carlo sampling (Cerf & Davey, 2001)
5. Distil insights into advice / intervention strategies for minimizing failure and improving CIP unit- operation.

In uncertainty modelling, simulation of a unit-operation the process parameters is defined by a distribution of values, the mean of which generally agrees with the single point, or SVA value. A refined Monte Carlo sampling of the probability distributions is used to take account of the effect of uncertainty on the value of the risk factor (*p*).

A refined Monte Carlo sampling is used because 'pure' Monte Carlo samplings can both over- and under-sample from various parts of the distribution. It therefore cannot be relied on to replicate the key parameter distribution. Latin Hypercube sampling is used to ensure the random sampling of each probability distribution covers the entire range of the distribution. The method uses a random number generator (Vose, 2008). These simulations are used to identify scenarios of practical process events that give rise to CIP failure.

For the model output parameter distributions to be sufficiently normal a minimum number of random samples from parameter distributions are necessary; this usually means some 1,000 to 50,000 random samples (Davey K R - *unpublished data*) (Vose, 2008). This minimum number of samples can however be readily established visually.

Importantly, the uncertainty risk model is identical to the traditional model in that all mathematical manipulations (multiplications, additions, exponentiations, integrations and differentiations etc.) that link each process parameter are the same, except that each parameter is represented by a probability function instead of a single value with error estimate. This permits the calculation of the combined impact of the variability (fact) and uncertainty (chance) (Vose, 2008; Davey, 2010; Davey, 2011) in process parameters to determine a probability distribution of possible process outcomes, including those determined by chance changes 'with in' system.

2.18 Summary

From a critical review of the literature, the following important factors emerge which are relevant to this research:

1. CIP is a widely used unit-operation globally in the milk process industries.

2. Fouling is a major problem in milk processing. If fouled deposits are not removed they may contaminate end products which can have an enduring impact on public health.

3. The CIP unit-operations models developed by Bird & Fryer (1991) and Bird (1992) and Xin, Chen & Ozkan (2004) are particularly well-suited for assessment of a new uncertainty risk analysis of vulnerability to failure. CIP failure can be defined as failure to remove a defined proteinaceous milk deposit in the auto-set CIP cleaning time.

4. Unit-operations in chemical engineering are usually solved via a Single Value Assessment (SVA) in which 'best guess' estimates or a mean value for input parameters are used. The output is a single value. In contrast to SVA is the Monte Carlo Assessment (MCA) or vulnerability to failure model. In vulnerability to failure modelling both Uncertainty and Variability in the input parameters are accounted for using Monte Carlo sampling or Monte Carlo assessment (MCA). The output prediction is a probability distribution of values in which the mean is nearly always equal to the SVA value. The mathematics for both approaches are similar i.e. all mathematical formulations (addition, multiplication, exponentiation, etc) remain the same except in MCA the inputs are defined by a distribution of values. The vulnerability to failure model output is a distribution of values, in contrast to a single, or mean, value from SVA.

5. Despite the apparent utility of a vulnerability to failure model for gaining greater insight into practical operation of CIP, none has been reported.

6. The methodology developed by Davey and co-workers for uncertainty modelling appears applicable for a new risk analysis of CIP milk processing.

In the next chapter, a new uncertainty model is developed based on the simplified CIP unit-operations model of Bird & Fryer (1991) and Bird (1992). The importance of Uncertainty and Variability in temperature of the cleaning solutions in failure to remove a proteinaceous milk deposit in an auto-set CIP cleaning time is highlighted.

CHAPTER THREE

A NEW UNCERTAINTY MODEL FOR CLEAN-IN-PLACE (CIP) MILK PROCESING BASED ON A BIRD & FRYER (1991) AND BIRD (1992) UNIT-OPERATIONS MODEL

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3.1 Introduction

A review of the literature (Chapter 2) showed that CIP is an important unit-operation used worldwide; failure can be a serious public health concern with potential enduring effects. A number of attempts at modelling CIP have been made with varying degrees of sophistication.

Of particular interest however is why apparently well-operated, well-maintained process plant fails unexpectedly. This widely recognized failure cannot be attributed to 'human error' or 'faulty fittings' (Cerf & Davey, 2001). Davey & Cerf (2003) called this 'Friday 13th syndrome' an expression that is meant to conjure the reality of the unexpected accumulation of small within system changes in the value of process parameters that accumulate in one direction to leverage change across a safe-unsafe (or sterile-non sterile) boundary.

From a chemical / bio-chemical engineering viewpoint the structured, two-stage model of Bird & Fryer (1991) and Bird (1992) can be said to be a milestone contribution to studies of CIP. The main aim of their model was to determine temperature and velocity effects on removal of proteinaceous milk deposits.

In this chapter this simplified model for the *Alkali-clean* step (with 1-wt% NaOH) on a uniform proteinaceous deposit from milk is first formatted into an appropriate unit-operations model which is then tested against the extensive experimental and predictive data of Bird (1992). The 5-step algorithm and methodology of Davey and co-workers (*see* Davey, 2010) is carefully applied to develop a new risk analysis of CIP for the first time and a comparison is made with the traditional single point (SVA) solution. A new quantitative definition of CIP failure is developed based on the introduction of a risk factor (p) such that for all $p > 0$ CIP will have failed to remove the deposit in the auto-set cleaning time. The aim was to gain new insight into conditions that could lead to failure of CIP and to identify scenarios that are probable.

3.2 CIP model of Bird & Fryer (1991) and Bird (1992)

The model developed by Bird (1992) and presented by Bird & Fryer (1991) is well-suited both in terms of analysis and reproducible experimental testing. In this model cleaning is postulated to take place in two-stages:

1. Transformation of the deposit by alkali into a form that can be removed
2. Removal of the transformed deposit in aggregates by fluid shear.

3.2.1 A unit-operations model

In the model of Bird & Fryer (1991 a, b; Bird 1992) a deposit of initial thickness δ, is considered as two layers: an upper layer of swelled deposit which can be removed, of thickness $x\delta$, and a lower layer of thickness $y\delta$ of deposit which is not yet removable. The equations governing the rate of change of thickness of the two layers are simply expressed as:

$$\frac{dy\delta}{dt} = -k_y \tag{3.1}$$

$$\frac{dx\delta}{dt} = k_y - k_x x\delta \tag{3.2}$$

Equations (3.1) and (3.2) express, respectively, that the rate of change of initial non-removable deposit y to removable deposit x is constant, and the rate of loss of removable deposit is proportional to the thickness of that deposit. Equation (3.2) can be integrated to give:

$$x = \frac{k_y}{\delta k_x}(1 - \exp(-k_x t)) \tag{3.3}$$

The first-stage is the break-up of the swelled x-layer by the alkali in which the removal rate ($R_{S\text{-}1}$) and the rate of removal ($R_{S\text{-}1}$) can be written as:

$$R_{\text{S-1}} = k_x \delta x = k_y (1 - \exp(-k_x t)) \text{ for } 0 < t < t^* \tag{3.4}$$

Equation (3.4) will apply until all the deposit has changed into a removable form by the alkali at time:

$$t^* = \frac{\delta}{k_y} \tag{3.5}$$

The maximum rate of removal occurs at $t = t^*$

$$R_{\max} = k_x \delta x_{\max} = k_y (1 - \exp[-\frac{\delta k_x}{k_y}]) \tag{3.6}$$

The second-stage applies for all $t > t^*$ in which the rate of removal falls with x such that:

$$x = x_{\max} \exp(-k_x (t - t^*)) \tag{3.7}$$

and

$$R_{S-2} = R_{\max} \exp(-k_x (t - t^*)) \, t > t^* \tag{3.8}$$

The combination of Equations (3.4), (3.7) and (3.8) gives the cleaning curve.

It is important to note that the time taken to reach the maximum cleaning rate (t^*) is dependent on k_y (and not both k_y and k_x) (Equation (3.5)), that is, the time to convert all the deposit to a removable form.

The time taken for the removal rate to drop half the maximum is given by the following equation ($t_{1/2}$):

$$t_{1/2} = \frac{\ln(2)}{k_x} \tag{3.9}$$

The total cleaning time (t_T) for the *Alkali-clean* step is defined as the time for removal rate to fall 0.02 R_{max}. Therefore the total cleaning time taken for this process can be calculated by rearranging Equation (3.9) and is given by the following equation:

$$t_T = t^* - \frac{\ln(0.02)}{k_x} \tag{3.10}$$

The unit-operations model of Bird & Fryer model is defined by Equations (3.1) through (3.10).

Solution is to solve Equation (3.5) for the time taken to reach the maximum removal rate, Equation (3.4) for rate of removal during decay stage, Equation (3.6) for maximum removal rate, Equation (3.8) for the rate of removal during removal stage. Solving Equation (3.9) gives the value of time taken for removal rate to drop half the maximum. The total cleaning time is the sum of time taken to reach maximum removal rate and time taken for removal rate to drop half the maximum and is given by Equation (3.10).

3.2.2 Data from Bird (1992) for model constants

Model constants for the CIP unit-operations model of Bird & Fryer (1991) were extracted from Bird (1992) for the reported experimental conditions by Davey, Chandrakash & O'Neill (2011) and are presented as Table 3-1.

Table 3-1: Constants for the unit-operations model of Bird & Fryer (1991) extracted from the experimental data of Bird (1992) for the *Alkali-clean* step (1-wt % NaOH) as a cleaning solution on proteinaceous milk deposit

T (^{O}C)	Re	k_x x 10^{-3} (s^{-1})	k_y x 10^{-7} (m s^{-1})
30	1270	1.155	1.34
40	1450	1.44	2.42
50	1770	1.65	3.85
60	2120	7.70	6.25
70	2500	0.115	0.10

$Re = 742.79 \exp(0.0173\ T)$ $R^2 = 0.9969$
$k_x = 1 \times 10^{-4} \exp(0.0680\ T)$ $R^2 = 0.8783$
$k_y = 3 \times 10^{-8} \exp(0.0497\ T)$ $R^2 = 0.9979$

3.3 Traditional Single Value Solution (SVA)

The method traditionally used for solution is the traditional single point assessment (SVA) calculation. This type of approach is called as the traditional single point or Single Value Assessment (SVA) (Davey & Cerf, 2003; Davey, 2011). For example, at a cleaning solution temperature of $T = 60\ ^{O}$C, Re = 2097; $k_x = 5.9 \times 10^{-3}\ s^{-1}$ and $k_y = 5.92 \times 10^{-7}$ m s^{-1}. Substitution and simplification yields for an initial proteinaceous deposit of thickness 0.00015 m (Bird & Fryer, 1991): $t^* = 253.46$ s from Equation (3.5); $t_T = 914.88$ s from Equation (3.9); $R_{S-1} = 5.89 \times 10^{-7}$ m s^{-1} from Equation (3.4); $R_{max} = 4.60 \times 10^{-7}$ m s^{-1} from Equation (3.6) and $R_{S-2} = 9.19 \times 10^{-9}$ m s^{-1} from Equation (3.8).

The resulting CIP curve can be simulated and shows agreement with that of Bird (1992) and Bird & Fryer (1991) and Fryer (1997). An example of CIP cleaning curve obtained using this unit-operations model at a cleaning solution temperature of 60 ^{O}C is shown as Fig. 3-1. The figure shows the presence of two stages: removal and decay. This CIP model of Bird & Fryer (1991) and Bird (1992) applies for a range of temperatures of the cleaning solution from 30 ^{O}C to 70 ^{O}C; a mid-range cleaning solution temperature of 60 ^{O}C is selected for this simulation and validation of the model.

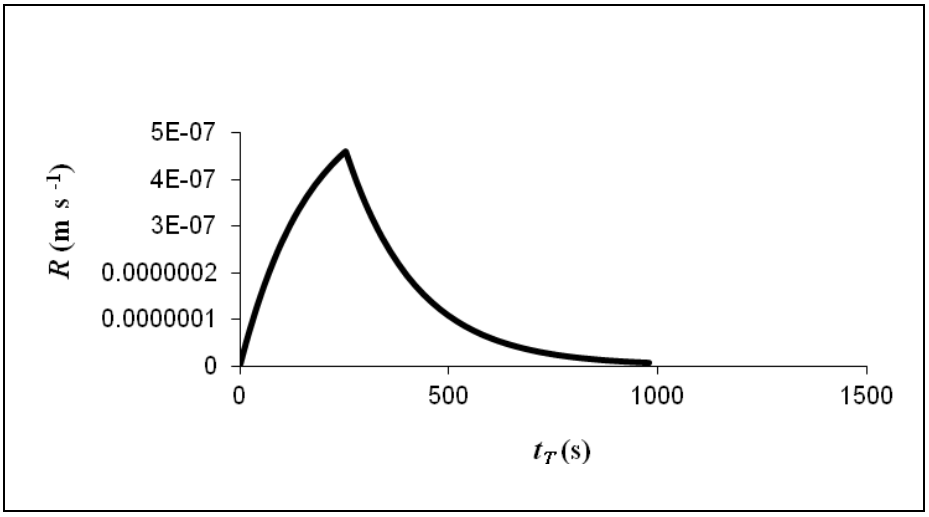

Fig. 3-1: Predicted CIP curve obtained using Bird & Fryer (1991) and Bird (1992) unit-operations model for a cleaning solution (1-wt % NaOH) temperature of 60 ^{O}C on a proteinaceous milk deposit of thickness 0.00015 m

3.4 Vulnerability to failure model solution

In vulnerability to failure simulation the process parameters are defined by a distribution of values, the mean of which generally agrees with the SVA value (Cerf & Davey, 2001; Patil, Davey & Daughtry, 2005; Patil, 2006; Davey, 2011). A refined Monte Carlo sampling of the probability distributions is used to take account of the effect of uncertainty on the value of the risk factor (p). A refined Monte Carlo sampling is used because 'pure' Monte Carlo samplings can over- and under-sample from various parts of the distribution (Davey, 2010). It therefore cannot be relied on to replicate the key parameter distribution (Vose, 2008). Latin Hypercube sampling is used to ensure the random sampling of each probability distribution covers the entire range of distribution. The method uses a random number generator (Vose, 2008).

3.4.1 Defining CIP failure (risk factor)

Generally, a process cannot be accepted below a tolerance on the design performance.

CIP times are usually specified in an auto-cycle. A suitable risk factor p can be defined as a cleaning time needed to remove the proteinaceous milk deposit (t_T') that is greater than the specified time (t_T) together with an acceptable tolerance value. That is:

$$p = -\%\,\text{tolerance} + 100\left(\frac{t_T'}{t_T} - 1\right) \tag{3.11}$$

Equation (3.11) is computationally convenient because it shows failure of CIP occurs for all values of $p > 0$. For a specified lower tolerance, for example, of 6% in the total cleaning time, we have from Equation (3.11):

$$p = -6 + 100\left(\frac{t_T'}{t_T} - 1\right) \tag{3.12}$$

From Equation (3.12) it can be understood that, if the removal of the deposit requires more than the specified cleaning time plus a tolerance value of 6% then the CIP process has failed. A tolerance value of 6% was selected based on discussions with Brigitte Carpentier (*Laboratoire de securite sanitaire de Maisons-Alfort*, France, *pers. comm*.).

3.4.2 Simulation of vulnerability to failure

This simulation methodology can be used to identify scenarios of practical process events that may give rise to product failure. To ensure that the output parameter distribution is sufficiently normal a minimum number of random samples from the key parameter distributions are necessary (Vose, 2008). This usually means 1,000 to 50,000 samples will be needed (Davey, K R – *unpublished data*). In most commercial software it is a simple matter to establish this minimum number of samples visually.

Calculations were performed in Microsoft Excel™ with a commercially available 'add-on' *@Risk* (pronounced *at risk*) version 4.5 (Palisade Corporation). This is computationally

convenient because Excel has nearly universal use, and therefore makes communication of results streamlined.

3.5 Results

The results obtained using uncertainty modelling and that of the traditional approach (SVA) can be studied with the aid of Table 3-2. Column 2 shows the value used in the SVA for each of the key process parameters. Column 3 gives the value of each of these for the uncertainty failure model. The values given in column 3 are for one only of 1,000 scenarios. The bolded values in column 4 give the selected distribution of the cleaning temperature. The distribution is defined as: **RiskNormal** (mean, standard deviation, **RiskTruncate** (minimum, maximum)). Failure is defined for all values of the risk factor $p > 0$.

Table 3-2: Summary comparison of uncertainty model with traditional SVA for the CIP unit-operations model of Bird (1992) and Bird & Fryer (1991a, b) for an *Alkali-clean* step with a 1-wt% NaOH cleaning solution at a mid-range temperature of $T = 60\ ^{O}C$ with a tolerance of 6% on total cleaning time (t_T)

Process Parameter	**SVA***	**Uncertainty Risk Model****	
T (^{O}C)	60	**59.04388**	**RiskNormal**(60,0.5, **RiskTruncate**(59, 61))
Re (dimensionless)	2097	2062.909	Correlation
k_x (m s^{-1})	0.0059	0.005542	Correlation
k_y (s^{-1})	5.92 x 10^{-7}	5.64 x 10^{-7}	Correlation
δ (m)	0.00015	0.00015	Constant
t^* (s)	253	265.7916	Equation (3.5)
t_T (s)	914	971.6478	Equation (3.9)
$R_{S\text{-}1}$ (m s^{-1})	5.89 x 10^{-7}	5.62 x 10^{-7}	Equation (3.4)
R_{max} (m s^{-1})	4.60 x 10^{-7}	4.35 x 10^{-7}	Equation (3.6)
$R_{S\text{-}2}$ (m s^{-1})	9.19 x 10^{-9}	8.70 x 10^{-9}	Equation (3.8)
p		0.20486	Equation (3.11)

* SVA = Single Value Assessment, or, Single Point Value
** With Latin Hypercube sampling

3.6 Discussion

1,000 random Latin Hypercube samples of the cleaning solution temperature were simulated. The data for the uncertainty risk predictions in Column 3 of Table 5-1

represents one-only scenario of these 1,000. For the scenario presented it can be seen that $p > 0$ (Equation (3.11)). This reveals a failure of specified total cleaning time required in the CIP unit-operations model of Bird & Fryer. With the 1,000 simulations of the uncertainty risk model practically all combinations of CIP operational scenarios that could occur will have been calculated in cleaning the initial proteinaceous deposit of thickness $\delta = 0.00015$ m.

The number of CIP failures would be expected to increase with a decreasing %-tolerance on the total cleaning time. Figure 3-2 presents a summary of repeat calculations of Table 3-2 for a range of values of the %-tolerance from 1 to 10. It can be seen from Figure 3-2 that there is an apparent exponential dependence of the number of predicted failures per 1,000 operational scenarios with %-tolerance. At a %-tolerance of 7 onwards there would not be any unexpected failures in CIP total cleaning time as predicted from the unit-operations model. Figure 3-2 suggests that better and improved temperature control could be readily justified.

Fig. 3-2: Predicted effect of %-tolerance on CIP failures with a 1-wt% NaOH cleaning solution at a mid-range temperature of $T = 60\ ^{O}C$ with a tolerance of 6% on total cleaning time (t_T)

A total of 10 predicted failures were identified in the 1,000 scenarios with a tolerance of 6% on total cleaning time required from the CIP unit-operations model of Bird & Fryer and are shown below in Table 3-3, it can be seen all have a value of $p > 0$. The bolded values in Table 3-3 represent the scenario reported in Table 3-2 using uncertainty modelling technique

Table 3-3: 10 failures per 1,000 CIP operations at T = 60 ^{O}C and %-tolerance = 6

T (^{O}C)	t_T (s)	p
59.00565	973.9904	0.46091
59.00908	973.7796	0.43788
59.01865	973.1927	0.37372
59.02732	972.6618	0.31570
59.03878	971.9599	0.23898
59.04388	**971.6478**	**0.20486**
59.04929	971.3165	0.16865
59.05837	970.7608	0.10791
59.06850	970.1414	0.040214
59.07063	970.0114	0.025997

The 10 predicted failures in CIP cleaning time of Table 3-3 (failure fraction = 0.10) reveal that a failure, which cannot be attributed to human or operator error or faulty fittings, will occur without warning, on average, once about every three months (i.e. 365.25 day/year x 1/(100 day) ~ 3.65/year) at a nominal cleaning solution temperature of 60 ^{O}C. These predicted failures are caused by chance changes in the cleaning solution temperature and will therefore not be equally-spaced in time.

The solution to this newly developed uncertainty model using Bird & Fryer (1991) underlying unit-operations model can be conveniently shown schematically as a flow sheet by means of using a fish bone diagram (or Ishikawa diagram *see* Ishikawa, 1976) Fig. 3-3. This concept was first introduced by Kaoru Ishikawa in 1968. The figure is read from left to right.

In the figure the bones represent the input factors that lead to the process output. This fishbone is helpful in clearly identifying the relationship between the output result and the possible causes that relate to it. The combination of factors is underscored. This fish bone diagram can also be called as ‘The Cause and Effect Diagram’ or ‘Herringbone Diagram’. The format of the diagram is easy-to-read and for increasing levels of model sophistication puts the focus on the inputs and possible output effects.

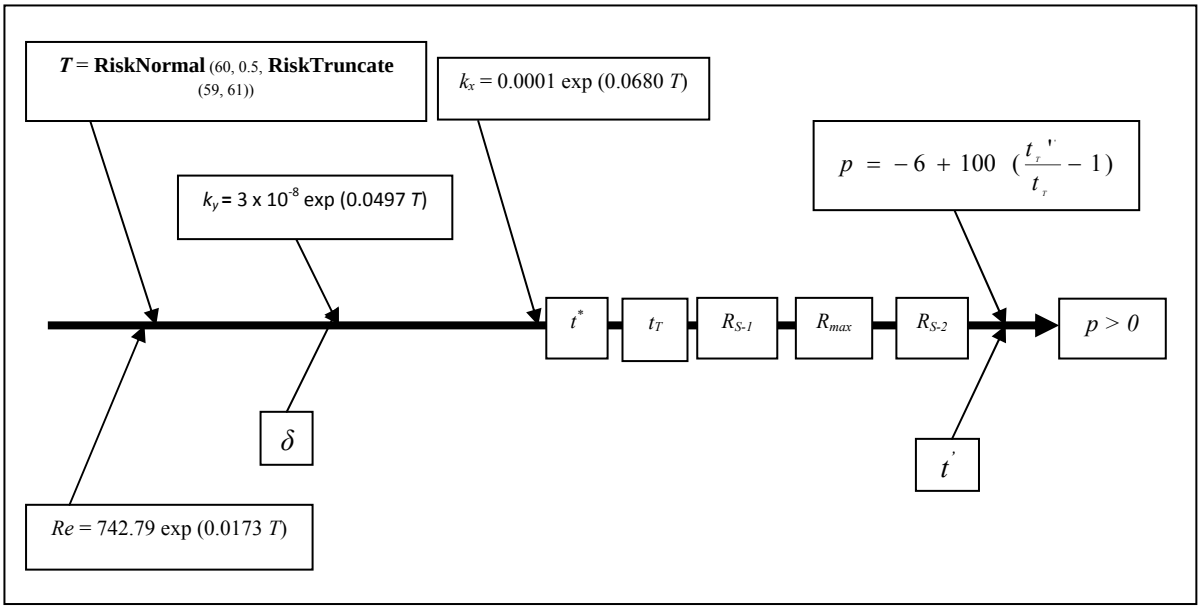

Fig. 3-3: Fish bone (Ishikawa) diagram to highlight uncertainty model solution of the unit-operations CIP model of Bird & Fryer (1991) and Bird (1992)

This type of representation highlights the mathematics and the mathematical interconnectedness of the inputs and outputs of the unit-operations model.

The utility of the diagram is realized as the underlying CIP unit-operations model increases in complexity and with the number and type of distributions used.

3.7 Summary and conclusions

1. A novel uncertainty model for auto-set CIP cleaning has been developed for the first time based on the extensive experimental data of the temperature dependent, two-stage removal model of Bird & Fryer (1991) and Bird (1992) for the *Alkali-clean* step on a uniform proteinaceous deposit from milk.

2. Findings highlight that with a practical mid-range temperature of 60 ^{O}C for the 1-wt% NaOH cleaning solution on a deposit thickness of 0.00015 m and a tolerance of 6% over the auto-set clean time, 10 out of every 1,000 continuous operations can fail unexpectedly. This equates to 1% vulnerability to failure, or, a failure to remove the deposit in the auto-set CIP time approximately every three months. These failures will not be spaced equally in time however.

3. The insight gained with this new model over traditional methods is to identify all scenarios that are probable. This new analysis of CIP failure represents a significant advance over traditional analyses in that all possible practical scenarios that could exist operationally are computed and rigorous quantitative evidence is produced to show that the CIP unit-operation is actually a mix of failed cleaning operations together with successful ones.

4. This insight cannot be obtained using the traditional approach (SVA) (with or without sensitivity analysis).

5. Improved design and operating decisions can be made with the new model because the engineer has a picture of all possible outcomes. The ‘golden rule’ however is that each of the outcome scenarios produced must be potentially observable in real life.

6. Because variability is the effect of chance, and a function of the system, it is not reducible through further study. This view contrasts in some areas of engineering with the widely-held view of determinism in which chance cannot be accepted to play a part, or even a significant part, in failure and cannot be minimised through more facts about the process. Vulnerability to failure can however be reduced through changing the physical system. This raises the possibility of intervention

strategies, such as improved temperature control. The uncertainty model can be used in second-tier simulations to reveal quantitatively the effect of any proposed physical changes.

7. A drawback with the new CIP model based on Bird & Fryer (1991) and Bird (1992) is that it is mathematically simple being only temperature dependent and turbulent flow conditions cannot be used (the value of cleaning solution flow velocity predicted is low as 0.174 m s^{-1}).

A potentially better cleaning model is that of Xin (2003) and Xin, Chen & Ozkan (2004). Although this model is temperature dependent it posits a third stage in CIP cleaning of milk and whey proteinaceous deposits and permits turbulent flow conditions to be used with increased cleaning solution velocities over that of Bird & Fryer (1991) and Bird (1992).

In the next chapter an uncertainty model is developed using the model of Xin (2003) and Xin, Chen & Ozkan (2004) as an underlying CIP unit-operation model. This more complex model is investigated for vulnerability to unexpected failure in continuous operation.

CHAPTER FOUR

A NEW UNCERTAINTY MODEL FOR CLEAN-IN-PLACE (CIP) MILK PROCESSING BASED ON A XIN (2003) AND XIN, CHEN & OZKAN (2004) UNIT-OPERATIONS MODEL

Parts of this chapter have been published as:

Davey, K. R., Chandrakash, S. & O'Neill, B. K. 2012. Study of failure of clean-in-place (CIP) in a dairy plant – A case study using uncertainty failure modelling, *Journal of Food Protection* – in preparation.

4.1 Introduction

Findings from Chapter 3 highlighted that the simplified CIP model of Bird & Fryer (1991) and Bird (1992) could be formatted as a CIP unit-operations model that was amenable to new uncertainty analyses with Monte Carlo sampling, developed by Davey and co-workers. A drawback highlighted with this CIP model however was that it is mathematically simple being temperature dependent only with limited flow conditions of the 1-wt% NaOH cleaning solution.

The principal aim of this chapter is to develop and explore a novel uncertainty analysis of CIP based on the more sophisticated mathematical treatment developed by Xin (2003) and Xin, Chen & Ozkan (2004).

The principles and the mathematics involved in development of this CIP model are first discussed in detail and the 5-step algorithm and methodology of Davey and co-workers is carefully applied. A risk factor (p) is computationally defined such that for all $p > 0$ CIP will have failed to remove the deposit in the auto-set cleaning time. A comparison is made with the traditional single point (SVA) solution of the resulting CIP unit-operations model.

In the following chapter, Chapter 5, a comparison is made between these two selected CIP unit-operations models using the concept of vulnerability to failure modelling.

4.2 Xin (2003) and Xin, Chen & Ozkan (2004) model

In the Xin (2003) and Xin, Chen & Ozkan (2004) CIP model the process of cleaning is posited to take place in three stages, namely:

1. Swelling stage
2. Uniform stage
3. Decay stage.

The cleaning is based on a polymer dissolution principle. This is the process in which a solid (or liquid) forms a homogenous mixture with a solvent (solution) with consequent breakdown of a crystal-lattice into individual ions, atoms or molecules, and these are

transport into the solvent. The researchers used Whey Protein Concentrate (WPC) gel as a model foulant.

This CIP model of Xin (2003) and Xin, Chen & Ozkan (2004) is presented diagrammatically as Fig. 4-1.

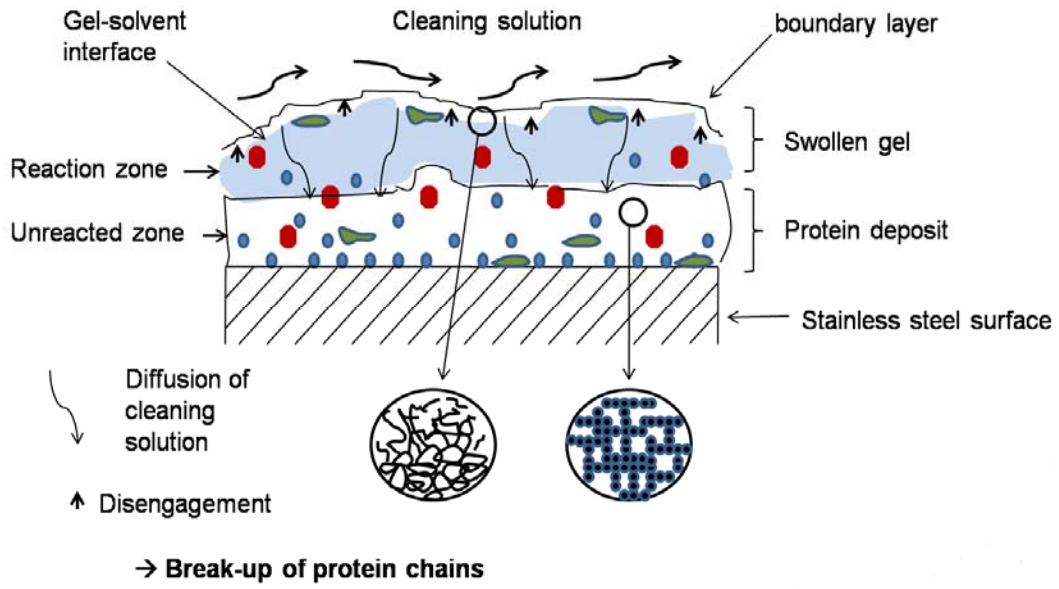

Fig. 4-1: Diagrammatic representation of Xin (2003) and Xin, Chen & Ozkan (2004) CIP model

The five steps involved in CIP cleaning and removal of the deposit are:

1. Cleaning solution transfers from its bulk to the surface of the deposit
2. A series of chemical reactions generating intermediate products
3. Further penetration into the deposit and build up of a reaction zone and a swollen gel layer
4. Disengagement of deposit particles
5. The disengaged particles are washed away by the cleaning solution leaving behind a layer of possible non removable deposit.

4.2.1 A unit-operations model

Based on the conventional concept of mass transfer (e.g. Foust et al., 1980; Wankat, 2007) the cleaning rate of the WPC gel molecules from the gel solution interface may be written as:

$$-R = \frac{1}{A}\frac{dm}{dt} = k_{\phi}(\phi - \phi_b) \tag{4.1}$$

where m is the mass removed, R the cleaning rate, k_{φ} the mass transfer coefficient based on volume fraction changes of materials, A the surface area, ϕ the volume fraction of disengaged protein molecule at the gel solution interface, and ϕ_b the volume fraction of disengaged protein molecule in the bulk cleaning solution.

The concentration of the disengaged protein molecule outside the boundary layer ϕ_b is negligible and can be taken as zero, thus Equation (4.1) becomes:

$$-R = \frac{1}{A}\frac{dm}{dt} = -k_{\phi}\phi \tag{4.2}$$

The change of the volume fraction of disengaged protein molecular chains accumulated at the gel solution interface at any time has been assumed to change according to first order reaction mechanism:

$$\frac{d\phi}{dt} = k_d\phi \tag{4.3}$$

where k_d is the disengagement rate constant. In this model it is assumed that the disengagement rate constant of the molecule chains (k_d) decreases with increasing ϕ, and approaches zero when the maximum value (ϕ_{m}) is reached. As a first estimation k_d takes the following form;

$$k_d = \xi(1 - \frac{\phi}{\phi_m}) \tag{4.4}$$

where ξ is a kinetic constant and ϕ_m is the maximum volume fraction taken up by the disengaged protein molecules.

The change of the volume fraction of the disengaged protein molecules at the gel solution interface is expressed by the following:

$$\frac{d\phi}{dt} = \xi(1 - \frac{\phi}{\phi_m})\phi \tag{4.5}$$

The polymer chain requires a finite induction time to disengage from the gel solution interface; the disengagement rate is initially zero. This minimum induction time required for first few chains to disengage is equivalent to the reptation time (t_r) (Narasimhan & Peppas, 1998). Therefore, it is assumed that the following initial condition exists at the gel solution interface:

$$\frac{d\phi}{dt} = 0 \qquad t < t_r \tag{4.6}$$

After the process of reptation, from $t = t_r$ to $t > t_r$, the volume fraction of the disengaged protein molecules at the gel solution interface can be calculated by integrating Equation (4.5):

$$\phi = \frac{\phi_m e^{\xi(t-t_r)}}{\frac{\phi_m}{\phi_0} - 1 + e^{\xi(t-t_r)}} \tag{4.7}$$

where ϕ_0 is the volume fraction of the tangling protein chains at the solution side of the gel solution interface at time $t = t_r$ ($\phi_0 \neq 0$). The equations above represent a self limiting process when the concentration of the protein molecules becomes high.

We define a dimensionless parameter ψ as:

$$\psi = \frac{\phi_m}{\phi_0} - 1 \tag{4.8}$$

Combining Equations (4.2) and (4.7) the cleaning rate can be rewritten as:

$$-R = \frac{1}{A}\frac{dm}{dt} = -\frac{R_m e^{\xi(t-t_r)}}{\psi + e^{\xi(t-t_r)}} \tag{4.9}$$

where R_m (constant cleaning rate during uniform stage) is defined as:

$$-R_m = -k_\phi \phi_m \tag{4.10}$$

The mass removed from the deposit as a function of time can then be calculated by integrating Equation (4.9):

$$m = \frac{AR_m}{\xi}\left(\ln\frac{(\psi + e^{\xi(t-t_r)})}{(\psi+1)}\right) \tag{4.11}$$

At the end of the uniform stage the continuous film of WPC deposit is broken up and only patches of the deposit film are left on the stainless steel surface. The film is no longer continuous and uniform, it is expected that the cleaning rate will depend on the amount of the gel film remaining on the surface, the cleaning rate will still be controlled by dissolution and mass transfer processes. In the study of removal of organic films in the decay stage, the change of the surface area of the remaining film has been modelled as a first order process, and adopting this approach, the protein removal in the decay stage is given as:

$$\frac{dA_L}{dt} = -k_A A_L \tag{4.12}$$

where A_L is the surface area covered by the protein film in the decay stage and k_A is the first order rate constant for the surface area reduction. This rate constant is expected to be dependent on temperature, mechanical properties of deposit, cleaning solution concentration, and flow velocity. The initial condition for Equation (4.12) is:

$$A_L = A_{L,0} \qquad \text{when } t \le t_{su}$$

where $A_{L,0}$ is the total surface area covered by the protein deposit. Integrating Equation (4.12) from $t \ge t_{su}$ to $t \le t_t$ (the total cleaning time) yields:

$$\frac{A_L}{A_{L,0}} = e^{[-k_A(t-t_{su})]} \tag{4.13}$$

Assuming that the cleaning rate during the decay stage depends on the remaining protein film area A_L, the cleaning rate during this stage can be expressed as:

$$R = R_m \frac{A_L}{A_{L,0}} \tag{4.14}$$

Combining Equations (4.13) and (4.14) gives:

$$R = R_m e^{[-k_A(t-t_{su})]} \tag{4.15}$$

To calculate t_{su} and the total cleaning time t_t, a critical protein mass remaining (m_c) at the start of the decay stage is identified as:

$$m_c = m_0 - m_{su} \tag{4.16}$$

where m_0 is the original mass of the deposit and m_{su} is the total mass removed during the swelling and uniform stages. m_{su} can be calculated using Equation (4.11) with the boundary condition at $t = t_{su}$.

Combining Equations (4.11) and (4.16), the mass removed decay stage can be determined as:

$$m_c = m_0 - \frac{AR_m}{\xi} \ln \frac{[\psi + e^{\xi(t_{su} - t_r)}]}{(\psi + 1)} \tag{4.17}$$

Rearranging Equation (4.17), t_{su} is given by the following:

$$t_{su} = \frac{1}{\xi} \ln[(\psi + 1)e^{(m_0 - m_c)\xi / R_m A} - \psi] + t_r \tag{4.18}$$

The mass loss of the deposit during the decay stage can also be expressed as:

$$\frac{1}{A_L} \frac{dm}{dt} = \frac{dm}{Ae^{[-k_A(t - t_{su})]} dt} = -R_m \tag{4.19}$$

Integrating with the boundary conditions:

$$m = 0 \text{ and } A_L = A_{L,0} \text{ when } t = t_{su} \tag{4.19a}$$

and;

$$m = m_c \text{ and } A_L = 0 \text{ when } t = t_t \tag{4.19b}$$

Then m_c can be expressed as:

$$m_c = \frac{R_m A}{k_A} \{1 - e^{[-k_A(t_t - t_{su})]}\} \tag{4.20}$$

By rearranging the Equation (4.20), t_t is given by the following:

$$t_t = -\frac{1}{k_A} \ln(1 - \frac{m_c k_A}{R_m A}) + t_{su} \tag{4.21}$$

By combining Equations (4.18) and (4.21), the total time t_t can be determined by using the following:

$$t_t = \frac{1}{\xi}\ln[(\psi+1)e^{(m_0-m_c)\xi/R_mA} - \psi] - \frac{1}{k_A}\ln(1-\frac{m_c k_A}{R_m A}) + t_r \qquad (4.22)$$

4.3 Effect of temperature and flow rate

This unit-operations model based on Xin (2003) and Xin, Chen & Ozkan (2004) is temperature dependent i.e. the effect of process temperature on other process parameters can be readily studied. The dependency of the model parameters on temperature and Reynolds number is defined through:

$$Y = f(\mathrm{Re})\exp(\frac{-E_a}{R_g T}) \qquad (4.23)$$

where Y represents the model parameters R_m, ξ, k_A, and $1/t_r$; E_a is the apparent activation energy (J mol^{-1}); R_g is the molar gas constant, and T is the value for absolute temperature (K). The value of cleaning solution temperature in this model was reported in Kelvin scale by Xin and co-workers and has been maintained for the purpose of unit consistency in the development of the CIP unit-operations model.

It is assumed that the linear relationship between the Reynolds number and Y within the Reynolds number range used in this reported experimental study is independent of temperature and is defined as:

$$f(\mathrm{Re}) = \alpha + \beta\,\mathrm{Re} \qquad (4.24)$$

where α and β are constants. The value of these constants (α and β) for different process parameters are be obtained from Table 4-1.

Table 4-1: Experimental parameters used in Xin (2003) and Xin, Chen & Ozkan (2004) model for *Alkali-clean* (0.5-wt% NaOH) step

Cleaning Stage	**Parameters (Units)**	***α***	***β***	***E_a*** **(kJ mol^{-1})**
Reptation	$1/t_r$ (s^{-1})	-2.7 x 10^{12}	1.0 x 10^{9}	85
Swelling	ξ (s^{-1})	6.5 x 10^{3}	1.4	33
Uniform	R_m (g m^{-2} s^{-1})	9.7 x 10^{5}	1.8 x 10^{2}	41
Decay	k_A (s^{-1})	-5.6 x 10^{2}	1.2	38

Using the values shown in Table 4-1 together with published data from these researchers the various process parameters involved in this CIP unit-operation can be calculated for a range of experimental conditions, Table 4-2.

4.4 Traditional Single Value Solution (SVA)

Using the correlations of Table 4-2 and the values of constants from Table 4-1 a traditional single value assessment (SVA) can be carried out. For example, at a cleaning solution temperature of T = 348 K (75 ^{O}C), Re = 5418; t_r = 2.54 s; ξ = 0.1865 s^{-1}; k_A = 0.0094 s^{-1}; R_m = 1.4736 g m^{-2} s^{-1}; m_0 = 573.17 g m^{-2} and u = 0.1579 m s^{-1}. The value of ψ (a dimensionless parameter) = 20 and critical mass m_c = 100 g m^{-2} remains constant throughout the CIP unit-operations model. Substitution of these values into the model equations yields: t_{su} = 340 s from Equation (4.18); t_T = 448 s using Equation (4.21); R_{su} = 1.4736 g m^{-2} s^{-1} from Equation (4.9) and R_d = 0.5326 g m^{-2} s^{-1} using Equation (4.15) and a CIP curve can be simulated, Figure 4-2.

Table 4-2: Temperature dependence of parameters in the CIP model based on Xin (2003) and Xin, Chen & Ozkan (2004) and reported by Davey, Chandrakash & O'Neill (2012)

T (K)	Re	t_r (s)	ξ (s^{-1})	R_m (g m^{-2} s^{-1})	k_A (s^{-1})	u (m s^{-1})	m_0 (g m^{-2})
308	9943	36	0.0604	0.3733	0.0036	0.29	310
318	8229	17	0.0796	0.5453	0.0048	0.24	652
328	7200	8	0.1066	0.8041	0.0064	0.21	354
338	6171	4	0.1386	1.1458	0.0082	0.18	621
348	5486	2	0.1578	1.6310	0.0107	0.16	532
358	4800	1	0.2024	2.2592	0.0134	0.14	619
368	4117	0.82	0.2891	3.0498	0.0160	0.12	679
373	3771	0.75	0.3204	3.5105	0.0172	0.11	711

Re = 813103 exp (-0.0144 T)	R^2 = 0.9976
t_r = 5 x 10^{-9} exp (-0.0615 T)	R^2 = 0.9774
ξ = 3 x 10^{-5} exp (0.0251 T)	R^2 = 0.9934
R_m = 9 x 10^{-6} exp (0.0345 T)	R^2 = 0.9977
k_A = 2 x 10^{-6} exp (0.0243 T)	R^2 = 0.9926
u = 23.702 exp (-0.0144 T)	R^2 = 0.9976
m_0 = 21.758 exp (0.0094 T)	R^2 = 0.4940

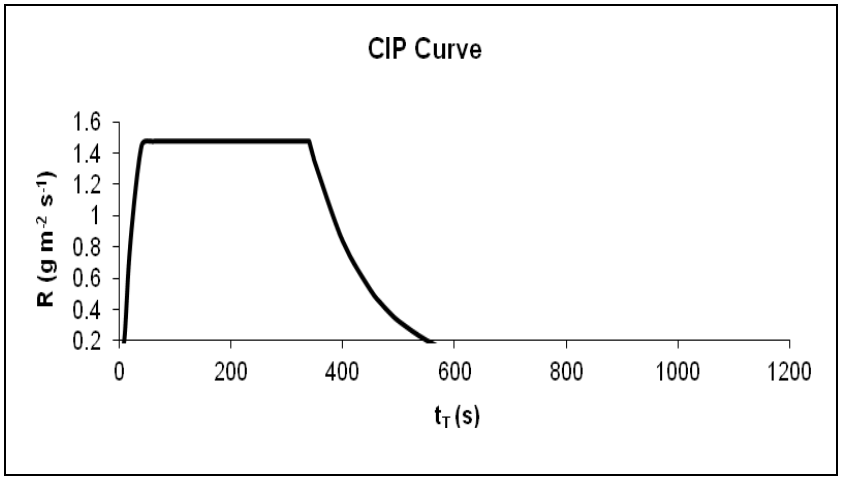

Fig.4-2: Predicted CIP curve based on Xin (2003) and Xin, Chen & Ozkan (2004) model for *Alkali-clean* step (with 0.5-wt% NaOH) at a temperature T = 348 K (75 ^{O}C) and Re = 5418

The predicted CIP cleaning curve of Figure 4-2 for a mid-range temperature of the cleaning solution of 348 K (75 ^{O}C) and Re = 5418 shows very good agreement with that of the experimental data of Xin, Chen & Ozkan (2004).

4.5 Vulnerability to failure modelling

Simulations were done in a similar way as done with the unit-operations model of Bird & Fryer (1991) and Bird (1992) in this section. A similar equation for CIP risk factor is devised as shown in the next section.

4.5.1 CIP failure (risk factor)

A suitable risk factor (p) based on an auto-cycle CIP cycle can be defined in similar fashion to that presented in Chapter 3 (Equation (3.11)) such that:

$$p = -\%\text{tolerance} + 100\left(\frac{t_T'}{t_T} - 1\right) \tag{4.25}$$

Equation (4.25) is again computationally convenient because it shows failure of CIP occurs for all values of $p > 0$. For an assumed lower tolerance of 2% in the total cleaning time (t_T), Equation (4.25) becomes:

$$p = -2 + 100\left(\frac{t_T'}{t_T} - 1\right) \tag{4.26}$$

From Equation (4.26) it can be understood that if the removal of the deposit requires more than the specified cleaning time plus a tolerance value of 2% then CIP has failed.

The resulting uncertainty model of Xin and co-workers can be conveniently depicted using a fish bone (Ishikawa) diagram. This is shown in Figure B-1 (Appendix B).

4.6 Results

The results of uncertainty modelling of the CIP unit-operations model of Xin (2003) and Xin, Chen & Ozkan (2004) using the method of Davey and co-workers, are summarized in Table 4-3.

The table presents a summary of comparison with the traditional SVA at a mid-range cleaning solution temperature of 348 K (75 ^{O}C) with a tolerance of 2% on the auto-set cleaning time (t_T). 1,000 random Latin Hypercube samples were used. Each simulation can be conveniently thought of corresponding to a daily end-of-process CIP plant clean.

Column 2 in Table 4-3 shows the value used in the SVA for each of the key process parameters. Column 3 gives the value for each of these for the uncertainty failure model. It is very important to note that the values given in column 3 are for only one of the 1,000 scenarios simulated. The bolded values in column 4 give the selected distribution of the cleaning solution temperature. The selected distribution is defined as: **RiskNormal** (mean, standard deviation, **RiskTruncate** (minimum, maximum)) i.e. a cleaning solution temperature of 348 K (75 ^{O}C) with a standard deviation of 0.348 K (standard deviation (sd) varies with the mean value of temperature i.e. if T = 308 K (35 ^{O}C) then sd = 0.308 and so on) and a minimum of 347 K (74 ^{O}C) and a maximum of 349 K (76 ^{O}C). Failure is defined for all values of the risk factor (p) greater than zero.

Table 4-3: Summary comparison of vulnerability to failure model with the traditional SVA for the CIP unit-operations model based on Xin (2003) and Xin, Chen & Ozkan (2004) for an *Alkali-clean* step (with 0.5%-wt NaOH) at a mid-range temperature of T = 348 K (75 ^{O}C) with a tolerance of 2% on total cleaning time (t_T)

Process Parameter	**SVA***	**Uncertainty Risk Model****	
T (K)	348	**347.25**	**RiskNormal**(348,0.348,**RiskTruncate**(347,349))
Re (dimensionless)	5418	5476	Correlation
u (m s^{-1})	0.1579	0.1596	Correlation
t_r (s)	2.5363	2.6554	Correlation
ξ (s^{-1})	0.1865	0.1830	Correlation
k_A (s^{-1})	0.0094	0.0092	Correlation
m_0 (g m^{-2})	573.17	569.16	Correlation
R_m (g m^{-2} s^{-1})	1.4736	1.4361	Correlation
ψ (dimensionless)	20	20	Constant
m_c (g m^{-2})	100	100	Constant
t_{su} (s)	339.96	345.97	Equation (4.18)
t_T (s)	448.11	**457.59**	Equation (4.21)
R_{su} (g m^{-2} s^{-1})	1.4736	1.4361	Equation (4.9)
R_d (g m^{-2} s^{-1})	0.5326	0.5120	Equation (4.15)
p		0.1147	Equation (4.26)

* SVA = Single Value Assessment
** With Latin Hypercube sampling

Analyses of the 1,000 simulations revealed that at a mid-range temperature of the cleaning solution of T = 348 K (75 ^{O}C) there were 19 cases that failed to meet the specified cleaning time i.e. $p > 0$. These were individually identified and are summarized in Table 4-4. The row with bolded values is the particular scenario reported in Table 4-3.

Table 4-4: 19 failures in total cleaning time (t_T) per 1,000 CIP operations with a cleaning solution (0.5-wt% NaOH) temperature of T = 348 K (75 ^{O}C) on a proteinaceous deposit

T (K)	t_T (s)	p
347.2859	457.1597	0.019445
347.2839	457.1859	0.025302
347.2713	457.3469	0.061221
347.2671	457.4016	0.073433
347.2584	457.5125	0.098171
347.2526	**457.5867**	**0.114738**
347.2400	457.7494	0.151049
347.2307	457.8684	0.177586
347.2165	458.0508	0.218299
347.2082	458.1569	0.241976
347.1998	458.2653	0.266161
347.1836	458.4737	0.312672
347.1666	458.6923	0.361465
347.1483	458.9276	0.413965
347.1376	459.0651	0.444656
347.1056	459.4771	0.536598
347.0935	459.6338	0.571560
347.0639	460.0151	0.656654
347.0067	460.7547	0.821693

4.7 Discussion

Results show that this unit-operation has a within system, stochastic, tendency to fail; for realistic values of the key parameters this is some 2 % of operations, averaged over the long term. If each simulated scenario is thought of as a processing day then the failure rate is expected to be about one every 19 days of continuous CIP operation (i.e. 365.25/19 = 19.2237 day). However this failure rate cannot be expected to be equally-spaced in time. This insight into a processing operation cannot be gained through the traditional single point approach (with or without sensitivity analysis) and is the major advantage of using uncertainty failure modelling technique over the traditional SVA.

The number of CIP failures would be expected to be influenced by the value of sd on the distribution used for the temperature of the cleaning solutions. A number of simulations were made to investigate this effect on a mid-range temperature of 348 K (75 ^{O}C) and the results are conveniently summarized in Table 4-5. It can be seen in the table that as sd is increased linearly the number of CIP failures to remove the proteinaceous deposit in the

auto-set time increases exponentially i.e. at sd = 0.348, n = 19 whereas at sd = 1.0, n = 119 per, 1,000 operations.

Table 4-5: Effect of standard deviation in the risk function for cleaning solution (0.5-wt% NaOH) temperature T = 348 K (75 ^{O}C) on the number of failures ($p > 0$) of CIP total cleaning time (t_T)

Temperature (K)	Number of failures
RiskNormal (348,0.1, RiskTruncate (347,349))	0
RiskNormal (348,0.348, RiskTruncate (347,349))	19
RiskNormal (348,0.5, RiskTruncate (347,349))	59
RiskNormal (348,1.0, RiskTruncate (347,349))	119
RiskNormal (348,2.0, RiskTruncate (347,349))	139

The implication of decreasing sd on the temperature of the cleaning solution is a practical improvement in control of the temperature. Better control will give fewer unexpected CIP failures but will have the disadvantage of increasing costs of the unit-operation.

It can be glimpsed therefore that the uncertainty modelling could be used in second-tier simulations to investigate the effect of improved control and design decisions aided in trading costs against resulting benefits (Davey, 2010).

4.8 Summary and conclusions

1. A novel uncertainty model for auto-set CIP has been developed for the first time from an underlying unit-operations model developed using the mathematical analyses of Xin (2003) and Xin, Chen & Ozkan (2004) for an *Alkali-clean* step on a proteinaceous whey deposit.

2. CIP is revealed quantitatively to actually be a continuous mix of successful and failed unit-operations. CIP failure is defined as the failure to remove all the deposit in the auto-set time plus 2%. For a nominal mid-range cleaning solution (0.5-wt% NaOH) temperature of 75 ^{O}C (348 K) and a tolerance on auto-set CIP time of 2%, some 19 failures per 1,000 (daily) operations on average could be expected to fail unexpectedly.

3. As the sd on the mean temperature of the cleaning solution is increased linearly the number of CIP failures to remove the proteinaceous deposit in the auto-set time increases exponentially. This implies better control of the temperature of the cleaning solution will give fewer unexpected CIP failures. A disadvantage however will be increasing costs of the CIP unit-operation.

4. It has been glimpsed that uncertainty modelling could be used in second-tier simulations to investigate the effect of improved control and other potential intervention strategies and changes to input parameters on costs and potential benefits of the unit-operation.

In the next chapter a comparison is made between the CIP unit-operations model developed in this chapter from Xin and co-workers, with that of Bird & Fryer (1991) and Bird (1992), presented in Chapter 3. Both unit-operations models are for the *Alkali-clean* step and are dependent on temperature and Re of the cleaning solution. Proteinaceous milk deposits on wet surfaces are postulated to be removed by shear from circulating cleaning solution. The output parameter of particular interest is the total cleaning time (t_T) needed to remove the deposit.

CHAPTER FIVE

A COMPARISON BETWEEN CIP UNIT-OPERATIONS MODELS DEVELOPED FROM XIN (2003) AND XIN, CHEN & OZKAN (2004) WITH THAT OF BIRD & FRYER (1991) AND BIRD (1992)

5.1 Introduction

In Chapters 3 and 4 an underlying CIP unit-operations model and quantitative predictions of vulnerability to failure was developed for the first time, respectively, from the simplified two-stage analysis of Bird & Fryer (1991) and Bird (1992), and; the more elaborate three-stage analysis of Xin (2003) and Xin, Chen & Ozkan (2004), using the methodology of Davey and co-workers.

The output parameter of particular interest was the total cleaning time (t_T) needed to remove the proteinaceous deposit: if the deposit was not removed in the auto-set CIP cleaning time, plus a practical tolerance, than the CIP operation was said to have failed i.e. all $p > 0$.

In this chapter a comparison is made between the structure of two CIP unit-operations models and the resultant predictions of underlying vulnerability to failure at a mid-range cleaning solution temperature of 65 ^{O}C. The general impact of temperature of the cleaning solution on each of the unit-operations models is then examined. The CIP model of Xin (2003) and Xin, Chen & Ozkan (2004) is demonstrated to have an overall lower vulnerability to failure as defined by the number of fails per 1,000 scenarios when compared to the unit-operations model of Bird & Fryer (1991) and Bird (1992).

5.2 CIP unit-operations model structure

A summary of the overall structure and a direct comparison of the unit-operations models is given in Table 5-1. Both unit-operations models are for the *Alkali-clean* step and are dependent on temperature and Re of the NaOH cleaning solution to remove proteinaceous milk deposits on wet surfaces by fluid shear due to forced circulation. Both are supported by extensive experimental validation.

Although the number of input parameters is similar at, respectively two and three (Table 5-1), both models are in practice temperature-dependent only. This limits investigation of the effect(s) of stochastic, within system, changes on vulnerability and likely failure to remove proteinaceous deposits on wet surfaces to this parameter. In practical terms however the temperature of any given cleaning solution will be overriding.

In contrast to the Xin, Chen & Ozkan model that of Bird & Fryer requires the thickness of the proteinaceous milk deposit be specified. This may not always be able to be accurately determined. Experience however might be used to fix the auto-set CIP clean time to remove deposits of the largest thickness observed. This thickness of the deposit is not explicitly required in the Xin, Chen & Ozkan model. The point has been made earlier that the deposit will almost certainly not be a continuous film in any event but will most likely be limited to small defined areas or pockets.

Table 5-1: Summary comparison of the two CIP unit-operations models for the *Alkali-clean* step

Parameter	**CIP unit-operations model**	
	Bird & Fryer (1991) and Bird (1992)	**Xin (2003) and Xin, Chen & Ozkan (2004)**
Input	2 (T, δ)	3 (T, ψ, m_c)
Cleaning solution	1-wt% NaOH	0.5-wt% NaOH
Experimental validation	Yes	Yes
Accuracy of prediction	Good	Good
Ease of use	Simple	More elaborate steps for simulation
Operational dependency	Cleaning solution temperature	Cleaning solution temperature
Realistic simulations	No	Yes Turbulent flow conditions (Re > 4,000) with cleaning solution velocity 0.1 m s^{-1} to 0.3 m s^{-1}

From the point of realistic simulations the model of Xin, Chen & Ozkan is advantageous because the cleaning solution velocity is explicitly used in the range 0.1 - 0.3 m s^{-1}. This covers that widely used in industry of about 2.5 m s^{-1}. With these velocities the flow is almost certainly turbulent (Re > 4,000) whereas with the Bird & Fryer model it cannot be ascertained that there will be turbulent flow of the cleaning solution and thereby effective removal rates.

Strictly, the utility of the models is fixed to respectively the 1-wt% or 0.5-wt% NaOH cleaning solution as this cannot be varied mathematically in the analyses. Both concentrations of cleaning solution are however typical in CIP cleaning operations.

5.3 Vulnerability to failure predictions

In vulnerability to failure or *Fr 13*, modelling the aim is to gain insight into conditions that may lead unexpectedly to failure of otherwise well-operated plant. The method pioneered by Davey and co-workers requires analysis of the effect of the accumulation of small stochastic changes (variances on inputs) that may trip operation from successful to failed states.

For both these CIP unit-operations models the key variance is the practical tolerance that can be used on the auto-set CIP cleaning time (t_T) and the sd (%-sd) in the **RiskNormal** distribution used to define the temperature of the NaOH cleaning solution.

A comparative summary of predictions for both unit-operations models at a mid-range cleaning solution temperature of T = 65 ^{O}C together with tolerance of 2% on t_T and a 1%-sd on temperature in the distribution risk function for the cleaning solution is presented in Table 5-2.

Surprisingly, both unit-operations models can be seen from the table to predict very nearly the same CIP total clean time of ~ 656 s for the mid-range cleaning solution temperature of 65 ^{O}C. The number of likely failures is apparently significantly different however with 287 for the Bird & Fryer and 160 for the Xin, Chen & Ozkan CIP unit-operations models. That is, the relative failure rate of Bird & Fryer/Xin, Chen & Ozkan averaged over time is ~ 1.8. This finding cannot lead to the conclusion that the Bird & Fryer model is worse in any sense than that of Xin, Chen & Ozkan. These failures are related to the inherent structure of the unit-operations models.

Table 5-2: Comparison of predictions of vulnerability to failure of the two unit-operations models at a mid-range cleaning solution temperature $T = 65\ ^{O}C$ together with a 2% tolerance on auto-set clean time (t_T) and a sd of 1% in the temperature distribution

Parameter	**CIP unit-operations model**	
	Bird & Fryer (1991) and Bird (1992)	**Xin (2003) and Xin, Chen & Ozkan (2004)**[§]
Input(s)	$\delta = 0.0015$ m	$\Psi = 20$ (dimensionless) $m_c = 100$ g m^{-2}
Cleaning solution	1-wt% NaOH	0.5-wt% NaOH
Temperature	**RiskNormal**(65, 0.65, **RiskTruncate**(64, 66))	**RiskNormal**(338, 3.38, **RiskTruncate**(337, 339))
Total clean time (t_T, s)	699.9	612.6
Failures per 1000 scenarios [§§]	287	160

[§] requires input as degree absolute

[§§] i.e. for all $p > 0$

It is important however to note that the three-stage, more elaborate analysis of Xin, Chen & Ozkan, is seen to be less vulnerable to unexpected CIP failure to remove proteinaceous milk deposits in the auto-set clean time.

The mid-range value of cleaning solution temperature of 65 ^{O}C used to compare the two unit-operations models is realistic for practical CIP operations. Temperatures below 50 ^{O}C are not used (Lloyd, 2008). This is because of the significant impact of temperature on the rates of deposit removal.

However, the impact of the nominal temperature of the NaOH cleaning solution on predicted CIP cleaning was investigated to better understand the structure of the two unit-operations models.

5.4 Effect of nominal cleaning solution temperature (*T*) on removal of deposits

5.4.1 Bird & Fryer (1991) and Bird (1992) model

For the Bird & Fryer model the effect of nominal temperature of the cleaning solution (1-wt % NaOH) on the maximum clean rate (R_{max}) of removal of proteinaceous milk deposit (δ = 0.00015 m) is summarized in Figure 5-1 for a range of values $30 < T < 80$ ^{O}C.

From the figure it can clearly be seen that the maximum rate of removal of the proteinaceous milk deposit increases (exponentially) significantly with increasing temperature. For example at a nominal temperature of 80 ^{O}C the removal rate is ~ 3.1 times that at 60 ^{O}C with the 1-wt% NaOH cleaning solution.

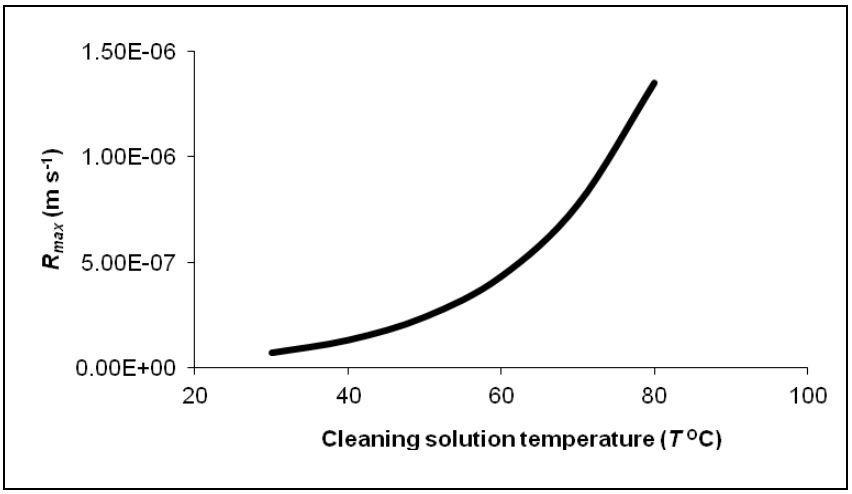

Fig. 5-1: Effect of nominal temperature of the (1-wt% NaOH) cleaning solution (*T*) on maximum removal rate (R_{max}) of proteinaceous milk deposit (δ = 0.00015 m) for the CIP unit-operations model of Bird & Fryer (1991) and Bird (1992) for $30 < T < 80$ ^{O}C

The effect of the nominal cleaning solution temperature on the predicted total cleaning time (t_T) required to remove the specified deposit (δ = 0.00015 m) of proteinaceous milk is summarized in Figure 5-2 for a range of values $30 < T < 80$ ^{O}C.

It can readily be seen in the figure that at the higher cleaning solution temperatures ($T > 50$ ^{O}C) the total cleaning time (t_T) reduces significantly; underscoring the use of temperatures of around 60 to 65 ^{O}C used in the food and allied industries. For example, at a nominal

cleaning solution temperature of 40 ^{O}C t_T = 3473 s, whilst at 60 ^{O}C, t_T = 971 s. This is a reduction in CIP time of ~ 2,500 s in each (daily) operation. The effect of temperature on cleaning time is therefore highly significant.

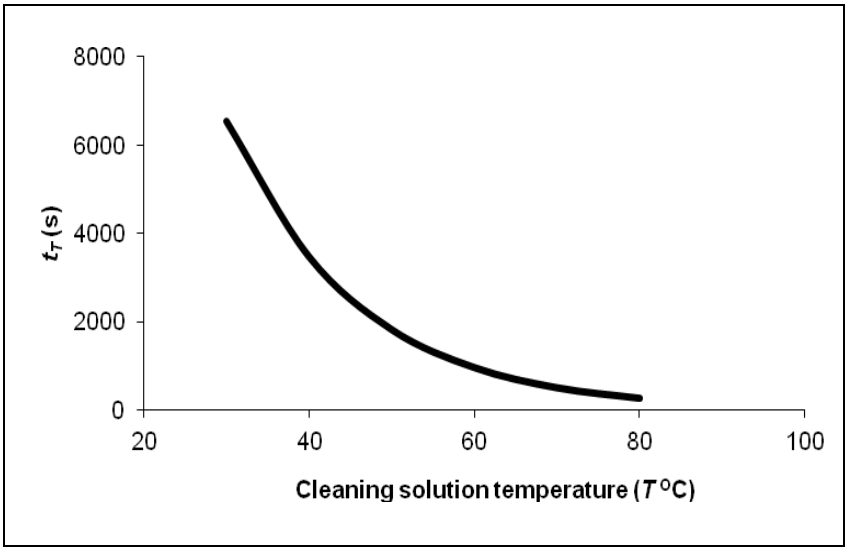

Fig. 5-2: Effect of nominal temperature of cleaning solution (1-wt% NaOH) (T) on total cleaning time (t_T) for removal of proteinaceous milk deposit (δ = 0.00015 m) for the CIP unit-operations model of Bird & Fryer (1991) and Bird (1992) for $30 < T < 80$ ^{O}C

The effect of nominal temperature of the cleaning solution ($30 < T < 80$ ^{O}C) on the vulnerability to failures of the CIP unit-operations model of Bird & Fryer (1991) and Bird (1992), at a constant value of 2% tolerance on auto-set clean time t_T and 1% sd of the nominal mean value of temperature, is summarized in Table 5-3. Failure is defined for all values $p > 0$. From the table it is seen that at constant %-tolerance and %-sd that increasing temperature increases (linearly) the number of unexpected failures.

Table 5-3: Comparison of predictions of vulnerability to failure of the unit-operations model of Bird & Fryer (1991) and Bird (1992) with a 2% tolerance on auto-set clean time (t_T) and a sd of 1% in the mean value of temperature for 30 ^{O}C < T < 80 ^{O}C

T (^{O}C)	Number of failure per 1000 scenarios
30	153
40	217
50	255
60	279
65	287
70	293
80	302

5.4.2 Xin (2003) and Xin, Chen & Ozkan (2004) model

In similar fashion the effect of cleaning solution temperature (T) on the CIP unit-operations model of Xin (2003) and Xin, Chen & Ozkan (2004) was investigated. This model is an elaboration on that of Bird & Fryer (1991) and Bird (1992) (*see* Chapter 2). It posits a stage in which the rate of removal remains constant for a particular period of time (uniform stage).

The effect of temperature of the 0.5-wt% NaOH cleaning solution (T) on reptation time (t_r) is presented in Figure 5-3 for a range of values 308 < T < 373 K. The temperature values used in this model are reported in degree absolute as Kelvin for the purpose of unit-consistency. Reptation time (t_r) is the minimum induction time required for the first few chains of the proteinaceous milk deposit to disengage[9].

Increasing the value of temperature of the cleaning solution can be seen in the figure to reduce the reptation time (t_r) significantly i.e. at higher temperature values the proteinaceous deposits disengage quicker. For example, at a nominal temperature of 348 K the reptation time is ~ 18 times lower than that at 308 K with the 0.5-wt% NaOH cleaning solution used.

[9] *see* Appendix A

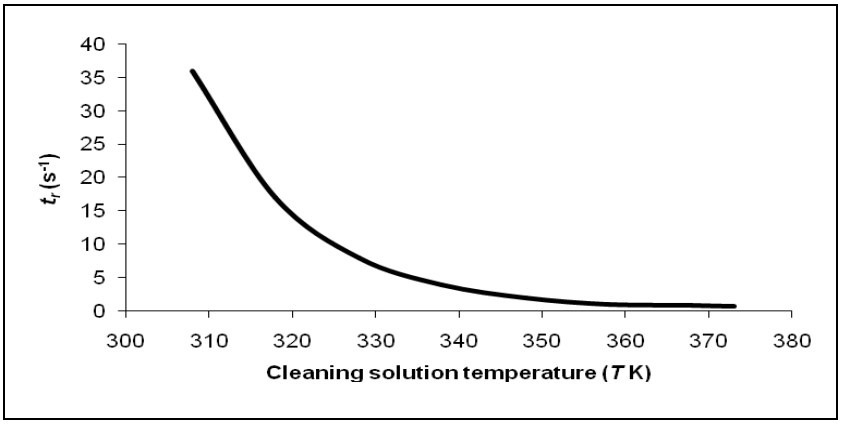

Fig. 5-3: Effect of nominal temperature of the (0.5-wt% NaOH) cleaning solution(T) on reptation time (t_r) of proteinaceous milk deposits for the CIP unit-operations model of Xin (2003) and Xin, Chen & Ozkan (2004) for $308 < T < 373$ K

The effect of cleaning solution temperature (T) on maximum rate of removal (R_m) of the deposits is summarized as Figure 5-4 for a range of values $308 < T < 373$ K.

From the figure it is seen that the maximum rate of removal of deposit increases with increasing cleaning solution temperature i.e. higher temperatures have higher rates of removal. For example, at a nominal cleaning solution temperature of 348 K the reptation time is ~ 4.4 times higher than that at 308 K.

The effect of cleaning solution temperature (T) on Reynolds number (Re) can be summarized in Figure 5-5 for a range of values $308 < T < 373$ K. As expected increasing temperature decreases the value of Reynolds number, this factor does not affect the turbulent flow nature of the cleaning solution (0.5-wt% NaOH) used. For example at a nominal temperature of 348 K Reynolds number is ~ 1.8 times lower than that at 308 K (but the flow is turbulent i.e. Re > 4000) with the 0.5-wt% NaOH cleaning solution used.

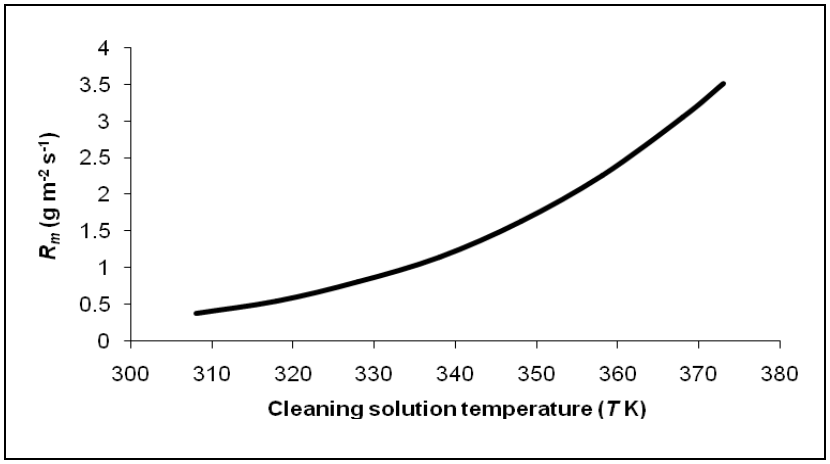

Fig. 5-4: Effect of nominal temperature of the (0.5-wt% NaOH) cleaning solution (T) on maximum rate of removal (R_m) of proteinaceous deposits for the CIP unit-operations model of Xin (2003) and Xin, Chen & Ozkan (2004) for $308 < T < 373$ K

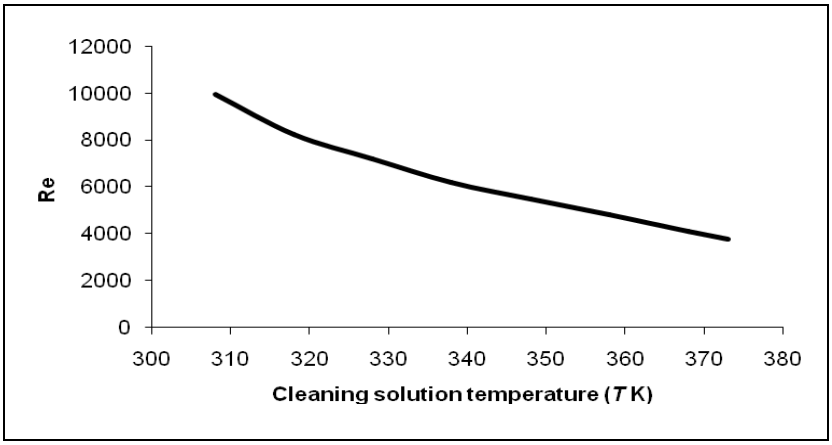

Fig. 5-5: Effect of nominal temperature of the (0.5-wt% NaOH) cleaning solution (T) on Reynolds number (Re) for removal of proteinaceous milk deposits in the CIP unit-operations model of Xin (2003) and Xin, Chen & Ozkan (2004) for $308 < T < 373$ K

The effect of nominal temperature of the cleaning solution ($308 < T < 373$ K) on the vulnerability to failure of the CIP unit-operations model of Xin (2003) and Xin, Chen & Ozkan (2004) at a constant value of 2% tolerance on auto-set clean time (t_T) and 1% sd on the nominal mean value of temperature, is summarized in Table 5-4.

Table 5-4: Comparison of predictions of vulnerability to failure of the unit-operations model of Xin (2003) and Xin, Chen & Ozkan (2004) with a 2% tolerance on auto-set clean time (t_T) and a sd of 1% in the mean value of temperature for 308 K < T < 373 K

T **(K)**	**Number of failure per 1000 scenarios**
308	344
318	229
328	184
338	160
348	144
358	134
368	126
373	124

From Table 5-4 it is evident that increasing temperature of the 0.5-wt% NaOH cleaning solution at a constant value of 2% tolerance on auto-set clean time and 1%-sd on mean value of temperature decreases the number of unexpected failures. The trend is seen to be actually opposite that for the more simplified structural model of Bird (1992) and Bird & Fryer (1991).

5.5 Overall effect of cleaning solution temperature (*T*) on removal

The results shown as Figures 5-3, 5-4 and 5-5 and Table 5-4 can be readily used to justify the selection of higher temperatures of the cleaning solution in practical operations with the more elaborate CIP unit-operations model of Xin (2003) and Xin, Chen & Ozkan (2004) since there is less vulnerability to underlying unexpected failure.

5.6 Summary and conclusions

1. Extensive analyses and comparison of the effect of structure of the two CIP unit-operations models of Bird & Fryer (1991) and Bird (1992), with that of Xin (2003) and Xin, Chen & Ozkan (2004), on predictions at similar operating conditions i.e. 2% tolerance on the auto-set clean time (t_T ~ 656 s) and 1%-sd in the nominal mean temperature of the NaOH cleaning solution at 65 ^{O}C, highlight that the underlying vulnerability to failure of the simplified two-stage model of Bird & Fryer is 1.8 times that of the more elaborate three-stage model of Xin, Chen & Ozkan.

2. The general need for use of higher values of the cleaning solution temperature i.e. $50 < T < 80$ ^{O}C for effective removal rates of proteinaceous milk deposits have been demonstrated to be best suited for CIP unit-operations on wet surfaces.

CHAPTER SIX

CONCLUSIONS

From this research the following can be concluded:

1. A new uncertainty risk analysis of a Clean-In-Place unit-operation can be carried out using the methodology of Davey and co-workers for milk processing. Failure of CIP is defined as the failure to remove a proteinaceous milk deposit from wet equipment surfaces in a prescribed auto-set cleaning time.

2. Predictions from a new unit-operations risk analysis developed from the simplified, two-stage model of Bird & Fryer (1991) and Bird (1992) highlight that for the *Alkali-clean* step of a uniform proteinaceous deposit from milk with a thickness of 0.00015 m and a practical, mid-range nominal temperature of 60 ^{O}C of the 1-wt% NaOH cleaning solution and tolerance of 6% over the auto-set clean time, 10 out of every 1,000 continuous operations can fail unexpectedly. This equates to 1% vulnerability to unexpected failure. If each operation is considered a daily CIP clean then there will be overall a failure to remove the deposit in the auto-set CIP time on average every three months.

3. Simulations from a new unit-operations risk analysis of CIP developed from the more elaborate three-stage model of Xin (2003) and Xin, Chen & Ozkan (2004) show that for the *Alkali-clean* step (0.5-wt% NaOH) of a proteinaceous whey deposit at a nominal mid-range cleaning solution temperature of 75 ^{O}C (348 K) and a tolerance on auto-set CIP time of 2%, some 19 failures per 1,000 (daily) operations on average could be expected to fail unexpectedly.

4. Extensive analyses and comparison of the effect of structure of the two CIP unit-operations models of Bird & Fryer (1991) and Bird (1992), and; Xin (2003) and Xin, Chen & Ozkan (2004), on predictions at similar operating conditions i.e. 2% tolerance on the auto-set clean time (t_T ~ 656 s) and 1%-sd in the nominal mean temperature of the NaOH cleaning solution at 65 ^{O}C, highlight that the underlying vulnerability to failure of the simplified model of Bird & Fryer is ~ 1.8 times that of the more elaborate three-stage model of Xin, Chen & Ozkan. The three-stage analysis is likely therefore to have included a better description of the mechanism of the clean step.

5. For both unit-operations risk models there is a need to use higher values of the cleaning solution temperature i.e. $50 < T < 80$ ^{O}C for effective removal rates of proteinaceous milk deposits. However if the value of temperature falls below the set value the process will not always fail. Importantly, if temperature falls below the set value the process will have increased vulnerability to fail. Improved control of the temperature of the cleaning solution will therefore result in fewer unexpected CIP failures. A potential disadvantage however will be increasing costs of the CIP unit-operation.

6. Quantitative results show that both CIP unit-operations are a mix of failed cleaning operations together with successful ones. This insight cannot be obtained using the traditional approach (SVA), with or without sensitivity analysis. Failures will not be spaced equally in time however.

7. The new uncertainty risk models could be used in second-tier simulations to investigate the effect of for e.g. improved temperature control, or other intervention strategies and changes to input parameters on costs and potential benefits of the unit-operation. Improved design and operating decisions can be made with the new model because the engineer has a picture of all possible outcomes.

8. The notion of the underlying vulnerability to failure of a global food process model can be glimpsed through the successful application of this risk methodology to CIP, and earlier UHT plant (Cerf & Davey, 2001; Davey & Cerf, 2003) and continuous fermenter (Patil, Davey & Daughtry, 2005). A global food process is defined as two or more unit-operations connected in a typical process flow (Davey, 2011; Davey, 2010).

6.1 Recommendations for future research

Because CIP is complex and multidisciplinary in nature better analyses may yet be developed to include the risk of re-growth of micro-organisms and a more realistic development of deposits and the effect of hydrodynamics of flow of the cleaning solution. Each of these aspects continues to be the subject of separate investigations. By using the

methodology demonstrated in this research it should be possible to gain insight into increasingly sophisticated analyses of CIP. As the CIP unit-operation becomes more sophisticated the use of the fishbone diagrams will prove useful to highlight the underlying mathematics and mathematical interconnectedness of the model parameters.

The success of this research strongly supports the notion that uncertainty modelling can, in principle, be applied to a wide range of single and possibly inter-connected food unit-operations. The benefits of uncertainty modelling will assist in evaluating the effects of Uncertainty and Variability on operational risk of unit-operations in chemical engineering.

APPENDICES

APPENDICES A - C

APPENDIX A – A definition of some important terms used in this research

Biofilm	A collection of micro-organisms surrounded by the slime they secrete, attached to either an inert or living surface. Importantly, the term means a continuous layer of cells with one or more superposed layers attached to it. In reality, bacteria do not exist as a biofilm over the equipment surface, rather they will be present as spots throughout the equipment surface
Biofouling	Accumulation of undesirable micro-organisms, algae and diatoms to the wet surface of process equipment
Caramelization	Oxidation of sugar; a type of pyrolysis reaction
Chance	*see* Variability
Clean-In-Place (CIP)	A standard method of cleaning the interior surface of process equipments and associated fittings without disassembly
Fact	*see* Uncertainty
Fouling	Process of accumulation of unwanted materials on solid surfaces, most often in an aquatic environment
Friday 13th Syndrome	Events defined by where just about all the bad in everything see to combine to make a failure of plans and opportunities despite all good design and operation (defined by Davey & Cerf 2003)
Gram test	Cells are first stained with crystal violet dye, and then treated with iodine solution, and then washed with alcohol
Induction time	Initial time that is required for the formations of protein aggregates of insoluble mineral complexes before noticeable amount of deposits are formed

Maillard reaction	A chemical reaction between amino acid and a reducing sugar, usually requiring heat
Probability	A numerical measure of the likelihood of a particular outcome of a stochastic process
Reptation time	The minimum induction time required for first few chains of proteinaceous deposits to disengage
Risk modelling	A structured science based process, used to estimate the likelihood of risk
Single Value Assessment	Process of a desired model output using a single value input (defined by Davey & Cerf, 2003)
Stochastic process	A system of countable events to a well-defined random process
Uncertainty	A lack of knowledge, or level of ignorance, about the parameters that characterize the physical system. It is also referred to as a *Fact*. Uncertainty is sometimes reducible through further measurement or careful study, or through consulting more experts (Vose, 2008)
Unit-operation	An operation in which chemical as well as physical changes takes place e.g. mixing, drying, heating, distillation, evaporation etc.
Variability	The effect of *Chance* on an outcome. It is a function of the system. Variability is not reducible through further study or careful measurement. It can be reduced through changing the physical system (Vose, 2008)

APPENDIX B – Fish bone diagram for simulation of CIP unit-operations model based on Xin (2003) and Xin, Chen & Ozkan (2004) analyses

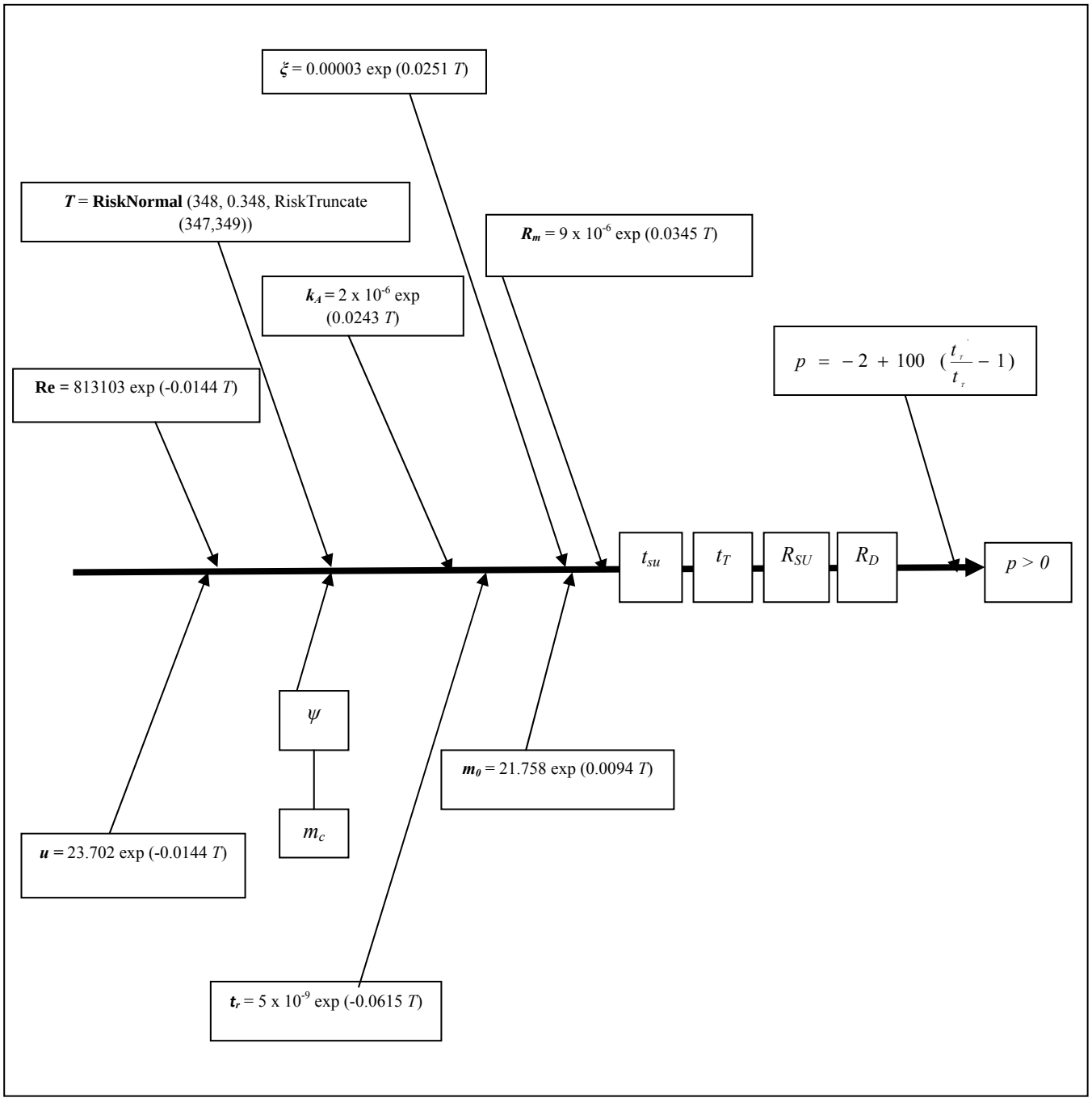

Fig. B-1: Fish bone (Ishikawa) diagram for Xin (2003) and Xin, Chen & Ozkan (2004) CIP model

APPENDIX C – Referred publications from this research

1. Davey, K. R., Chandrakash, S. & O'Neill, B. K., 2011. Friday 13th failure of Clean-In-Place operation in a milk process plant. In *Proc. 41st Australasian Chemical Engineering Conference (Engineering a Better World)*, CHEMECA 2011, September 18-21, Sydney, Australia, paper 150. ISBN: 9780858259225.

2. Davey, K. R., Chandrakash, S. & O'Neill, B. K., 2012. A new risk analysis of Clean-In-Place milk processing. *Food Control* – status date 30 March 2012.

3. Davey, K. R., Chandrakash, S. & O'Neill, B. K., 2012. Study of failure of Clean-In-Place (CIP) unit-operation in a dairy plant – A case study using Friday 13th failure modelling. *Journal of Food Protection* – in preparation.

Davey, K. R., Chandrakash, S. & O'Neill, B. K., (2011). Friday 13th failure of Clean-In-Place operation in a milk process plant.
In *Proceedings of the 41st Australasian Chemical Engineering Conference (Engineering a Better World)*, CHEMECA 2011, September 18-21, Sydney, Australia, paper 150.

NOMENCLATURE - Bird & Fryer (1991) and Bird (1992) model

k_x	First order rate constant, m s^{-1}
k_y	Zero order rate constant, s^{-1}
p	Risk factor (Equation (3.11)), dimensionless
Re	Reynolds number, dimensionless
R_{max}	Maximum removal rate, m s^{-1}
R_{S-1}	Rate of removal during stage-1, m s^{-1}
R_{S-2}	Rate of removal during stage-2, m s^{-1}
T	Cleaning solution (1-wt% NaOH) temperature, ^{O}C
t_T	Auto-cycle set cleaning time, s
t_T'	Actual or scenario value of t_T, s
t^*	Time taken to reach R_{max}, s

Greek Symbols

δ	Initial deposit thickness, 0.00015 m

Other

%-tolerance	Practical over treatment in design t_T

NOMENCLATURE - Xin (2003) and Xin, Chen & Ozkan (2004) model

m_c	Critical mass, g m^{-2}
m_0	Original deposit mass, g m^{-2}
p	Risk factor (Equation (4.25)), dimensionless
k_A	First order rate constant, s^{-1}
Re	Reynolds number, dimensionless
R	Cleaning rate, g m^{-2} s
R_m	Constant cleaning rate, g m^{-2} s
R_{SU}	Rate of removal during swelling and uniform stage, g m^{-2} s^{-1}
R_D	Rate of removal during decay stage, g m^{-2} s^{-1}
T	Cleaning solution (0.5-wt% NaOH) temperature, K
t_d	Cleaning time during decay stage, s
t_r	Reptation time, s
t_{su}	Sum of cleaning times during swelling and uniform stage, s
t_T	Total cleaning time, s
u	Cleaning solution flow velocity, m s^{-1}
Y	Symbol for model parameters (Equation (4.23))

Greek Symbols

α	Constant (Equation (4.24))
β	Constant (Equation (4.24))
ξ	Kinetic constant, s^{-1}

ψ	Dimensionless parameter

Other

%-tolerance	Practical over treatment in design t_T

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