

Copyright is owned by the Author of the thesis. Permission is given for a copy to be downloaded by an individual for the purpose of research and private study only. The thesis may not be reproduced elsewhere without the permission of the Author.

The Effect of Milk Fat Globule Membrane Damage in the Absence of Air on Fouling in Heat Exchangers

A Thesis Presented in Partial Fulfilment of the Requirements for
the Degree of Master of Technology in Food Technology
of Massey University

Le Fang (Leon Fung)

February 1998

乳脂球膜的破损在无空气介入时
对换热器中牛乳结垢的影响

该论文是新西兰梅西大学食品技术专业
硕士学位的必要组成部分

方砾

一九九八年二月

To my dear wife and daughter

Thanks for their understanding and moral support!

Abstract

The fouling by whole milk of processing plant surfaces, especially in heat exchangers, is a serious problem, but is incompletely understood despite extensive past investigations. While milk fat has generally been thought to play a minor role in fouling, the results of some previous work suggest that this is not always the case. The state and form of the fat, as well as processing conditions, may have effects on milk fouling behaviour.

Careless mechanical handling of whole milk is known to cause fat damage. The present study set out to investigate the effect on fouling of damage to the milk fat globule membrane (MFGM) by mechanical stresses in the absence of air.

Pasteurised non-homogenised whole milk was deliberately stressed to differing degrees by passing it through a cavitating pump a variable number of times. The extent of damage was measured using four different techniques: a free fat (FF) test (a modified extraction method), a lipolysable free fat (LFF) test (free fatty acid determination after incubation of the sample with pig pancreatic lipase, a technique developed during this work), particle size distribution (PSD) measurement by laser light scattering, and confocal laser scanning microscopy (CLSM).

The fouling behaviour of both damaged and undamaged milk was investigated by heating the milk from about 4°C to about 94°C in a custom built double pipe heat exchanger, which could be disassembled easily to access the fouling layer. Milk flowed in the annulus, with a Reynolds number range of about 220-310. The fouling rate was measured and expressed as the rate of increase of the overall resistance to heat transfer, normalized using the overall heat transfer coefficient determined at the start of a run.

The fouling rate exhibited by damaged milk (normalized by the rate for undamaged milk, to account for batch-to-batch variation) was found to increase significantly with the extent of cavitation treatment. There was also a clear positive relationship between both the FF and LFF contents of milk and the extent of cavitation treatment, suggesting strongly that the observed increases in fouling rate were the result of increased MFGM damage.

PSD measurement and CLSM both showed that cavitation caused the appearance in the milk of some large, irregularly shaped fat globules, presumably the result of coalescence. The FF results, and observation by CLSM, indicated that only a small proportion (< 6%) of the total milk fat had to be measurably damaged to cause extensive fouling.

The fat contents of the fouling layers were found to be very high (>45% on a dry weight basis). Although some of the experimental conditions, especially the low Reynolds numbers, may have contributed to this result, other fouling investigations made in New Zealand have produced similar results.

It is hypothesised that large globules formed by the coalescence of native globules whose membrane have been damaged could migrate more easily to the stainless steel heating surface. There, they could act as anchor points for the build-up of a fouling layer with a continuous protein phase. This hypothesis is supported by CLS micrographs of the fouling layer. Further investigation is warranted.

Recommendations are made for improving the methods used to measure MFGM damage, fouling and fouling rate, and the structure of the fouling layer.

ACKNOWLEDGEMENTS

Today, my dream of studying for the Master's Degree in the Department of Food Technology at Massey University many years ago comes true. I had happy and, of course, hard-working times here which will be a beautiful memory in my life.

During my study, I met so many warm-hearted people who had given me friendly supports. First, I would like to thank my supervisors Dr. Tuoc K. Trinh and Dr. Owen J. McCarthy for their kind supervision and encouragement throughout this project. For me, they are real rigorous scholars and helpful friends.

I also like to give my special thanks to Mr. Byron Mckillop, who was always patient with my questions and helped me in building the experimental rig, and Mr. Alistair Young, who helped me in obtaining the milk for the experiments. I will remember his kindness and humour.

Professional advice from Mr. Jaap Evers, Mr. Kevin Palfrayman and Dr. Alistair MacGibbon at the New Zealand Dairy Research Institute was valuable in the understanding of free fat and free fatty acids. Mr. Tuan Truong gave me much helpful advice and information on the topic of milk fouling. Dr. Mike Taylor of Massey University gave me guidance related to milk fat.

I appreciate those Massey staff who gave me technical support during this research, especially Ms. June Latham, Mr. Steve Glasgow, Mr. Mark Dorsey, Ms. Elizabeth Nickless, Dr. Al Rowland, Mr. Garry Radford and others.

Financial support from the New Zealand Dairy Board was highly appreciated. The New Zealand Rennet Company kindly supplied me some calf lipase.

Table of Contents

	Page
Abstract	iv
Acknowledgements	vi
Table of Contents	vii
List of Figures	x
List of Tables	xiii
List of Symbols	xiv
Chapter 1: Introduction	1
Chapter 2: Literature Review	4
2.1 Milk Fouling	4
2.2 Foulant Composition	6
2.3 Fouling Mechanisms	8
2.3.1 Phases of Fouling	8
2.3.2 Outline of Fouling Mechanisms	10
2.3.3 Protein Denaturation and Aggregation	13
2.3.4 Mineral Precipitation	14
2.3.5 Effect of Milk Fat	14
2.3.6 Other Parameters Affecting Fouling	15
2.4 Fouling Monitoring (Measurement)	18
2.5 Milk Fouling and Air Incorporation	19
2.5.1 Air Incorporation	19
2.5.2 Effect of Air Incorporation on Milk Fouling	21
2.6 Damage to Milk Fat Globule Membrane During Handling	23
2.6.1 Milk Fat Globule Membrane Composition	24
2.6.2 Consequences of Damage to the Milk Fat Globule Membrane	26
2.7 Milk Fat Globule Membrane Damage Determination	29
2.7.1 Measurements of Free Fat (FF)	29
2.7.2 Measurements of Free Fatty Acid (FFA)	30
2.7.3 Skim Milk Fat Test	33
2.7.4 Fat Globule Size Distribution Measurement	33
2.7.5 Fat Globule Appearance Observed by Confocal Laser Scanning Microscopy	35

2.7.6 Released Enzyme Test	38
Chapter 3: Materials and Methods	39
3.1 Raw Material	40
3.2 Fouling Rig	40
3.3 Thermocouple Calibration and Locations	47
3.3.1 Thermocouple Calibration	47
3.3.2 Thermocouple Locations	49
3.4 Method to Damage the Milk Fat Globule Membrane	49
3.4.1 Cavitation	50
3.4.2 Cavitation Rig	51
3.5 Methods of Measuring Milk Fat Globule Membrane Damage	53
3.5.1 Confocal Laser Scanning Microscopy	53
3.5.2 Free Fat (FF) Content	55
3.5.3 Lipolysable Free Fat (LFF) Content, Measured as Free Fatty Acid (FFA) Content	55
3.5.4 Modifications of Free Fat Test and Free Fatty Acid Test	56
3.6 Chemical Analysis of Liquid Milk Samples and Fouling Deposits	61
3.6.1 Moisture Test	61
3.6.2 Ash Test	62
3.6.3 Total Fat Content	62
3.6.4 Total Protein Content	62
3.7 Sampling Method	62
3.8 Fouling Trials	63
Chapter 4: Results and Discussion	67
4.1 Properties of Raw Materials	67
4.2 Result of Measurements of Milk Fat Globule Membrane Damage	67
4.3 Extent of Fouling	81
4.3.1 Observations of Fouling Layers	81
4.3.2 Fouling Rate	84
4.3.3 Experimental Constrains	92
4.3.4 Performance of the Fouling Rig	93
4.4 Fouling Deposit Analyses	94
4.4.1 Chemical Analysis	94
4.4.2 Observation of the Fouling Layer by Confocal Laser Scanning Microscopy	96
4.5 The Effect of Milk Fat Globule Membrane Damage on Fouling	97
4.6 Mechanism of Fouling --- Hypothesis	101
Chapter 5: Conclusions and Recommendations	103
5.1 Conclusions	103
5.2 Recommendations for Further Work	104

5.2.1 Lipolysable Free Fat Test	104
5.2.2 Fouling Rig	106
5.2.3 Fouling Layer	107
References	108
Appendices	113
1. Modified Free Fat Test for Whole Milk	113
2. Lipolysable Free Fat Test	115
3. Technical Characteristics of Calf Lipase	117
4. Technical Characteristics of Pancreatic Lipase	118
5. Moisture Test	119
6. Ash Test	120
7. Mojonnier Method for Crude Fat Content	121
8. Kjeldahl Method	123
9. Chronology of Experimental Work	125
10. Curves of Temperature Versus Time in the Fouling Rig Heat Exchanger	128
11. Results of Chemical Analyses to Fouling Deposits	137

List of Figures

	Page
Fig. 2.1 Fouling deposit on heating surface	5
Fig. 2.2 A typical fouling resistance curve, showing the three phases of fouling	8
Fig. 2.3 Schematic representation of the fouling mechanisms during heating of whey and milk	10
Fig. 2.4 Structure of fouling deposit from fresh whole milk on heating surface	11
Fig. 2.5 Milk transport within conventional milking machines	20
Fig. 2.6 Schematic representation of the participation of an air bubble in the fouling by milk of a hot stainless steel surface	22
Fig. 2.7 Average size frequency distribution of the fat globules in milk of a Friesian cow	34
Fig. 2.8 Principle of confocal laser scanning microscope	37
Fig. 3.1 Schematic of the custom built fouling rig	42
Fig. 3.2 Photograph of the fouling rig	42
Fig. 3.3 The small “window” cut in the outer tube of the rig (a) and the method of connecting the tubes (b)	44
Fig. 3.4 Structural features of the heat exchanger	45
Fig. 3.5 The hot water supply system for fouling rig	46
Fig. 3.6 Locations of thermocouples on the counter-current flow heat exchanger	49
Fig. 3.7 Damage of pump impeller blade owing to extremely high mechanical stress caused by cavitation	51
Fig. 3.8 Cavitation rig	52

Fig. 3.9 Photograph of the cavitation rig	52
Fig. 3.10 Picture of Leica confocal scanning laser microscope	54
Fig. 4.1 Fat particle size distribution - trial one	70
Fig. 4.2 Fat particle size distribution - trial two	71
Fig. 4.3 Fat particle size distribution - trial three	72
Fig. 4.4 Micrograph (63×) of fat globules in untreated milk. Trial one (run 1.1)	75
Fig. 4.5 Micrograph (63×) of fat globules in milk after one pass through the cavitating pump. Trial one (run 1.2)	75
Fig. 4.6 Micrograph (63×) of fat globules in milk after two passes through the cavitating pump. Trial one (run 1.3)	76
Fig. 4.7 Micrograph (63×) of fat globules in milk after one pass through the cavitating pump. Trial two (run 2.1)	76
Fig. 4.8 Micrograph (63×) of fat globules in untreated milk. Trial two (run 2.2)	77
Fig. 4.9 Micrograph (63×) of fat globules in milk after four passes through the cavitating pump. Trial two (run 2.3)	77
Fig. 4.10 Micrograph (63×) of fat globules in milk after five passes through the cavitating pump. Trial three (run 3.1)	78
Fig. 4.11 Micrograph (63×) of fat globules in milk after three passes through the cavitating pump. Trial three (run 3.2)	78
Fig. 4.12 Micrograph (63×) of fat globules in untreated milk. Trial three (run 3.3)	79
Fig. 4.13 Micrograph (63×) of fat and protein (protein in red and fat in green in milk after one pass through the cavitating pump. Trial two (run 2.1)	79
Fig. 4.14 FF and FFA content versus number of pass through the cavitating pump - Trial three	80
Fig. 4.15 Brown dots on the fouling layer from the hottest section (Trial 1, Run 1.1)	82

Fig. 4.16 Fresh fouling layers on the upper and lower sides of the inner stainless tubes of the fouling rig heat exchanger	83
Fig. 4.17 Temperature drop by fouling of pasteurised whole milk with 5 passes by cavitating pump (Trial 3, Run 3.1)	85
Fig. 4.18 The thermal resistance to heat transfer versus time - trial one	89
Fig. 4.19 The thermal resistance to heat transfer versus time - trial two	90
Fig. 4.20 The thermal resistance to heat transfer versus time - trial three	91
Fig. 4.21 Confocal laser scanning micrograph (10×) shows fat particles (in red) and proteins (in green) in a dried fouling layer. Trial one (run 1.2)	97
Fig. 4.22 The thermal resistance to heat transfer versus time - Trials 1-3	98
Fig. 4.23 Normalised fouling rate versus number of passes through cavitating pump	100
Fig. 5.1 Effect of activator in milk lipolysis	104
Fig. 5.2 Effect of pH on the amount of lipolysable fat in untreated and mechanically treated raw milk	106

List of Tables

	Page
Table 2.1 Composition of bovine milk fat globule membrane	25
Table 3.1 Dimensions of the stainless steel tubes	41
Table 3.2 Thermocouples calibration	48
Table 3.3 FFA content without added lipase, milk samples incubated at 30°C for 48 hours after storage at 4°C for 6 days followed by cavitation treatment	59
Table 3.4 Comparison of pancreatic lipase and calf lipase	60
Table 3.5 Descriptions of three trials performed	66
Table 4.1 Some composition data for pasteurised whole milk	68
Table 4.2 The chemical analysis results for the milk samples	68
Table 4.3 Performance of the fouling rig heat exchanger	93
Table 4.4 Compositions of fouling layers	94
Table 4.5 Fouling rates and normalised fouling rates for all runs	100

List of Symbols

R_F :	heat transfer resistance of fouling deposit (m^2KW^{-1})
U :	heat transfer coefficient after fouling formation ($Wm^{-2}K^{-1}$)
U_o :	heat transfer coefficient prior to fouling formation ($Wm^{-2}K^{-1}$)
d_{vs} :	volume surface average diameter of fat globules (μm)
$N_i/\Delta d$:	number frequency ($ml^{-1}\mu m^{-1}$)
d_o :	outlet diameter of inside tube (mm)
d_i :	inlet diameter of inside tube (mm)
D_o :	outlet diameter of outside tube (mm)
D_i :	inlet diameter of outside tube (mm)
Q' :	flow rate of milk ($L\ hr^{-1}$)
t_m :	milk outlet temperature ($^{\circ}C$)
T_m :	milk inlet temperature ($^{\circ}C$)
t_w :	hot water outlet temperature ($^{\circ}C$)
T_w :	hot water inlet temperature ($^{\circ}C$)
ρ_{milk} :	milk density (Kgm^{-3})
μ_{milk} :	milk viscosity (Pa s)
c_p :	heat capacity ($KJKg^{-1}K^{-1}$)
S' :	area of milk cross section (m^2)
d_e :	equal diameter of milk cross section (mm)
V' :	milk velocity ($m\ s^{-1}$)
Re :	Reynolds number
ΔT_m :	logarithmic temperature difference ($^{\circ}C$)
$Q_1(Q_m)$:	energy needed to heat up milk (W)
$Q_2(Q_w)$:	energy given by the hot water (W)
A :	heat exchange area (m^2)
Q'_{water} :	flow rate of hot water ($L\ hr^{-1}$)
ρ_{water} :	water density (Kgm^{-3})
$R_F U_o$:	fouling rate (min^{-1})