

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.

Characterising Stickiness of Dairy Powders

A thesis presented in partial fulfilment of the
requirements for the degree of
Master of Technology in Bioprocess Engineering

At Institute of Technology and Engineering
Massey University, Palmerston North, New Zealand

Rajesh Chatterjee

2004

ACKNOWLEDGEMENT

My quest for knowledge in the dairy processing industry put me into the safe hands of Tony Paterson, my principle supervisor. Thank you for everything including all the minor tweaking rendered, particularly in difficult times, to achieve the project targets as well as my accomplishments. I consider myself privileged to get you as my mentor.

The research underlying this thesis has been sponsored by the *Fonterra Research Centre, Palmerston North, New Zealand*. Many thanks to David Pearce, my sponsor cum supervisor, for all the support, faith and time.

Thanks to John Bronlund, my secondary supervisor, for all his guidance and contribution into this project. I am especially grateful to Kylie Foster and Jeremy Mcleod, my fellow researchers, who helped me in all aspects of research at Massey University.

I would also like to thank Kenneth Bidlake, Tuan Troung, Greg Crofskey, Tony Foskett and Shane Harvey of Fonterra and Rod Bennett, Bruce Collins, John Edwards and Don McLean of Massey and many others for all the support and general helpfulness.

I wish to thank Foundation for Research Science and Technology (Tech New Zealand) for funding this project.

Finally sincere thanks to my wife - Bonny, my daughter - Hiya and our new-born son - Dhusar, who kept on smiling against all odds and sailing with me in little bumpy but blue sea of New Zealand. Thanks to our parents, relatives and friends, Shantanu in particular, for supporting this dream voyage.

Thank you, all.

ABSTRACT

The stickiness phenomenon, one of the major operational problems, in the spray drying process is strongly related to changes in the powder particle surface. During the course of drying, powder particles with intermediate moisture pass through a very cohesive and adhesive 'plastic' phase. This phase has shown to be influenced by surface composition, moisture content, particle size, manufacturing method, surrounding air humidity and temperature.

During spray drying, the powder particle experiences varied temperature and humidity conditions, which were replicated under controlled dynamic conditions to some extent in a 'Bench-top-scale Fluid Bed Rig' or in a 'Particle Gun Rig'. In these two set-ups, stickiness-end-point or deposition rates at a particular temperature and humidity combination were plotted to develop 'Stickiness Curves' after testing different dairy-based powders. Further improvements in the 'Particle Gun Rig' has been identified to minimise heat loss for future experimentation.

It has been demonstrated that the stickiness property is a surface phenomenon. This is governed by the composition of a particular powder, manufacturing methods and the temperature / humidity conditions surrounding the powder particles. The low fat powders (<42%) tested followed a single step 'Lactose based stickiness model' and high fat powders (>42%) followed a combined ' Fat and lactose based stickiness model'. The 'lactose based model' followed the predicted glass transition (T_g) trend of amorphous lactose, shifted by some degree (X) upwards, depending on the product composition or the amount of amorphous lactose present – to be specific.

These quick and easy methods to identify a safe and non-sticky operating window to minimise product adhesion to the equipment wall would be of huge benefit to the dairy industry in process optimization, as fore knowledge of likely difficulties and specified operating conditions will help efficient and economic operation. Attempts have been made to rectify the humidity tracking system in a spray drier and relate the 'stickiness curves' with its drying parameters. Further work should be done by taking commercial

trial runs at recommended or allowable operating conditions with reference to 'Stickiness Curves', in order to maximise the throughput and to minimise the drying cost without compromising the product quality. Looking into the effects of other variables like air velocity, angle of impact, different impact surface materials and particle size on powder stickiness would be of much interest to the dairy industry.

TABLE OF CONTENTS

ACKNOWLEDGEMENT	ii
ABSTRACT	iii
TABLE OF CONTENTS	v
LIST OF FIGURES	ix
LIST OF TABLES	xi

CHAPTER 1 Project Overview

1.1	INTRODUCTION	1
1.2	STICKINESS DURING SPRAY DRYING	2
1.3	PROJECT OBJECTIVES	3
1.4	EXPECTED OUTCOME	3

CHAPTER 2 Literature Review

2.1	STICKING AND CAKING PHENOMENA	5
2.1.1	MECHANISM OF ADHESION & COHESION	6
2.1.1.1	Interparticle Attraction	9
2.1.1.1.1	Intermolecular forces	9
2.1.1.1.2	Electrostatic forces (non-conductor)	10
2.1.1.1.3	Electrostatic forces (conductor)	10
2.1.1.2	Liquid bridges	11
2.1.1.3	Solid bridges	14
2.1.1.4	Wetting and Thermodynamic Adsorption	14
2.1.1.5	Tack and sample rheology	15
2.2	FACTORS AFFECTING STICKINESS	15
2.2.1	WATER	16
2.2.2	TEMPERATURE	17
2.2.3	VISCOSITY	19

2.2.4	INGREDIENTS	20
2.2.5	PARTICLE SIZE	22
2.3	ROLE OF AMORPHOUS SUGARS	22
2.3.1	GLASS TRANSITION TEMPERATURE	23
2.3.2	MEASUREMENT OF GLASS-TRANSITION	26
2.3.2.1	Calorimetric Measurement Techniques	27
2.3.2.2	Mechanical Properties based	33
2.3.2.3	Molecular Mobility based	34
2.3.3	PREDICTION	34
2.4	ROLE OF FAT	35
2.4.1	MELTING RANGES	36
2.4.2	ROLE OF SURFACE FAT	37
2.5	STICKY POINT TEMPERATURE	38
2.5.1	MEASUREMENT	38
2.6	SPRAY DRYING – OVER VIEW	40
2.6.1	DYNAMICS OF PARTICLE DEPOSITION	42
2.6.1.1	Characterising Depositions	43
2.6.2	MANOEUVERS FOR REDUCING STICKINESS	44
2.6.3	UTILITY OF SIMULATION MODEL	46
2.7	EXPERIMENTAL PLAN	48

CHAPTER 3 Bench-top Scale Fluid-bed Stickiness Assessment Rig

3.1	INTRODUCTION	49
3.2	OBJECTIVES	50
3.3	BASIC APPARATUS	50
3.4	CHOICE OF METHODS	53
3.4.1	DATA LOGGING	53
3.4.2	TEMPERATURE	53
3.4.3	SAMPLE SIZE	55
3.4.4	AIR	55

3.4.5	EFFECT OF VIBRATION	56
3.5	EXPERIMENTAL RESULTS AND DISCUSSION	56
3.5.1	AMORPHOUS LACTOSE	57
3.5.2	ALPHA AND BETA LACTOSE	60
3.5.3	LACTOSE SUPERTAB	62
3.5.4	MILK PROTEIN CONCENTRATE 70	64
3.5.5	MILK PRTEIN CONCENTRATE 85	65
3.5.5.1	Isotherm Measurement of MPC 85	66
3.5.5.2	Thermal Analysis of MPC 85 as measured by DSC	68
3.5.6	WHOLE MILK POWDER 8051	71
3.5.7	AGGLOMERATED WHOLE MILK POWDER	73
3.5.8	INSTANT WHOLE MILK POWDER	74
3.5.9	HIGH FAT POWDERS	75
3.6	CLOSURE	76

CHAPTER 4 Measuring Stickiness of Dairy Powders in a Particle Gun Rig

4.1	INTRODUCTION	79
4.2	AIM AND OBJECTIVES	80
4.3	BASIC APPARATUS	81
4.4	EXPERIMENTAL METHODS	84
4.4.1	OPERATING PROCEDURE OF THE BUBBLE COLUMN HUMIDITY GENERATOR	84
4.4.2	TEST METHOD	88
4.5	CORRECTION OF EXPERIMENTAL DATA	90
4.5.1	NEED FOR CORRECTION	90
4.5.2	DEVELOPING THE CORRECTION MODEL USING STATISTICAL TOOLS	91
4.6	EXPERIMENTAL RESULTS AND DISCUSSION	93
4.6.1	AMORPHOUS LACTOSE	94
4.6.2	NUTRITIONAL POWDER – ANDEC 8811	97
4.6.3	CHEESE SNACK POWDER (COLOURED) – 3180	99

4.6.4	CHEESE POWDER (WHITE) – 3190	101
4.6.5	HIGH FAT CREAM POWDER (CP 70)	104
4.6.6	SKIM MILK POWDER – 6440	108
4.6.7	WHOLE MILK POWDER – 8051	111
4.6.8	STICKINESS – A SURFACE PHENOMENA	113
4.6.9	COMBINED $T - T_g$ TREND	114
4.7	CLOSURE	116

CHAPTER 5 Plant Study

5.1	INTRODUCTION	119
5.2	EXPERIMENTATION ON LONGBURN SPRAY DRIER	119
5.2.1	HUMIDITY CALCULATIONS	121
5.3	RESULTS AND DISCUSSION	126
5.4	CLOSURE	129

CHAPTER 6 Conclusions and Recommendations for Future Work

6.1	CONCLUSIONS	131
	APPENDIX I PRODUCT COMPOSITION	135
	NOMENCLATURE	137
	REFERENCES	139

LIST OF FIGURES

FIGURE 2.1	Schematic diagram of liquid bridges. P, particle, LB, liquid bridges, A, air (Peleg, 1977)	12
FIGURE 2.2	T_g of WPC hydrolysate at different water activities analysed by DSC (Kim <i>et al.</i> , 2002)	32
FIGURE 2.3	CFD analysis of particle deposit concentration in a spray drier (Fry, 2001b)	47
FIGURE 3.1	Schematic diagram of FRC Bench-top-scale Fluid Bed Rig	51
FIGURE 3.2	Photo of FRC Bench-top-scale Fluid Bed Rig	52
FIGURE 3.3	Alpha Lactose crystals (A) and Amorphous Lactose powders (B) as observed under polarising microscope	57
FIGURE 3.4	Amorphous Lactose under polarising microscope after the stickiness run in the fluid-bed rig	58
FIGURE 3.5	Stickiness end points of amorphous lactose tested on fluid-bed rig	60
FIGURE 3.6	Stickiness curve of α and β lactose tested on Fluid-bed rig	61
FIGURE 3.7	Stickiness curve of <i>Supertab</i> lactose tested on Fluid-bed rig	63
FIGURE 3.8	Stickiness curve of MPC 70 tested on Fluid-bed rig	64
FIGURE 3.9	Stickiness curve of MPC 85 tested on Fluid-bed rig	65
FIGURE 3.10	Moisture sorption isotherm for MPC85 at 20°C	67
FIGURE 3.11	Glass Transition Temperature (T_g) of MPC85, measured by DSC	70
FIGURE 3.12	Stickiness curve of WMP 8051 tested on Fluid-bed rig	72
FIGURE 3.13	Stickiness curve of AWMP 8490 tested on Fluid-bed rig	73
FIGURE 3.14	Stickiness curve of IWMP tested on Fluid-bed rig	75
FIGURE 3.15	Stickiness curve of all powders tested on Fluid-bed rig	76
FIGURE 4.1	Photo of Particle Gun Rig	82
FIGURE 4.2	Schematic diagram of Particle Gun Rig	83
FIGURE 4.3	Deposition of amorphous lactose tested on Particle Gun	95
FIGURE 4.4	Stickiness Curve of amorphous lactose tested on Particle Gun	96
FIGURE 4.5	Deposition of ANDEC powder tested on Particle Gun	97
FIGURE 4.6	Stickiness Curve of ANDEC powder tested on Particle Gun	98
FIGURE 4.7	Deposition of Cheese Snack (Coloured) powder - 3180 tested on Particle Gun	99

FIGURE 4.8	Stickiness Curve of Cheese Snack (Coloured) powder	100
FIGURE 4.9	Deposition of Cheese (White) powder - 3190 tested on Particle Gun	101
FIGURE 4.10	Deposition due to fat in Cheese powder – 3190 (White) tested on Particle Gun	103
FIGURE 4.11	Stickiness Curve of Cheese (White) powder - 3190 tested on Particle Gun	104
FIGURE 4.12	Deposition of CP 70 tested on Particle Gun	105
FIGURE 4.13	Deposition due to fat in CP 70 tested on Particle Gun	106
FIGURE 4.14	Stickiness Curve of CP 70 tested on Particle Gun	107
FIGURE 4.15	Deposition of SMP 6440 tested on Particle Gun	108
FIGURE 4.16	Stickiness Curve of SMP 6440 tested on Particle Gun	109
FIGURE 4.17	Stickiness Curve of SMP 6440 tested on Particle Gun including new experimental data	110
FIGURE 4.18	Deposition of WMP 8051 tested on Particle Gun	111
FIGURE 4.19	Stickiness Curve of WMP 8051 tested on Particle Gun and Fluid Bed	112
FIGURE 4.20	$T - T_g$ condition of Stickiness Curves of different powders Vs. their Amorphous Lactose content	115
FIGURE 5.1	Photo of coloured Cheese Powder deposition layer inside the powder conveying duct going out from the Fluidised Bed	120
FIGURE 5.2	Measured and predicted drying conditions for ANDEC powder on production	127
FIGURE 5.3	Predicted drying conditions for Snack Cheese Powder (3180) on production	128
FIGURE 5.4	Measured and predicted drying conditions for Cream Powder 70 on production	128
FIGURE 5.5	Predicted drying conditions for White Cheese Powder (3190) on production	129

LIST OF TABLES

TABLE 2.1	Glass transition temperature of anhydrous sugars, carbohydrate polymers and some foods (Bhandari and Howes, 2000)	21
TABLE 2.2	Methods for measurement of glass-transitions (Schenz, 1995)	27
TABLE 2.3	Transition temperatures of different lactose forms corresponding to physical transformation analyzed by DSC (Drapier-Beche <i>et al.</i> , 1997)	28
TABLE 2.4	Comparison of various test methods currently used to assess food stickiness, adapted from (Adhikari <i>et al.</i> , 2001)	39