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.

Massey Unive.s. / Library Palmerston North Turitea

THE MODELLING OF CAKING IN BULK LACTOSE

46

A thesis presented partial fulfilment of the requirements for the degree of Doctor of Philosophy in Process and Environmental Technology at Massey University

John Bronlund

B. Tech (Hons)

1997

J. Bronlund

THE MODELLING OF CAKING IN BULK LACTOSE

- 1 I give permission for my thesis to be made available to readers in Massey University Library under conditions determined by the Librarian.
- 2 I agree that my thesis, or a copy, may be sent to another institution under conditions determined by the Librarian.
- 3 I agree that my thesis may be copied for Library use.

Frankunt. Signed

5

1.1.1

Date 22nd October 1997

The copyright of this thesis belongs to the author. Readers must sign their name in the space below to show that they recognise this. They are asked to add their permanent address.

Name and Address

Date

ABSTRACT

Caking during storage is a serious problem for manufacturers of bulk lactose. This study was carried out to investigate the causes of caking and identify solutions as to how such problems can be eliminated.

The mechanisms for caking in crystalline lactose powders were identified. Liquid bridging between adjacent particles was shown to occur in high relative humidity environments (>80% RH) These liquid bridges could form crystalline solid bridges if the material was subsequently dried out. The potential mechanism of amorphous lactose flow and bridging in conditions where the glass transition temperature is exceeded was shown to be insignificant in predominantly crystalline lactose powders (<5% amorphous lactose) The presence of amorphous lactose is still important as the amorphous matrix acts as a sink of moisture, which can be released upon crystallisation. This increases the moisture available in the system which can contribute to caking by the liquid bridging mechanism. Both of these mechanisms involve changes in the local temperature and moisture conditions within the bulk powder. Such changes were known to be caused by moisture migration under the influence of a temperature gradient.

A model which describes the transport of moisture in one dimension as a result of temperature gradients was developed and validated. The microscopic scale processes of liquid bridging and amorphous lactose moisture relations were included into this model. The model predictions agreed well with experimental trials for completely crystalline lactose powders. Comparison of model predictions for the case where amorphous lactose was present on the surface of the particles showed some inadequacies exist in the model. These were the rate of amorphous lactose crystallisation and the assumption of instantaneous equilibrium between the crystallising amorphous matrix and the air present in the interstices of the bulk lactose.

Using the model it was shown that for expected storage conditions, the product should be stored with a water activity below 0.57 a_w if no amorphous lactose is present and below 0.25 a_w if it is present. If these prescribed limits are met then the goal of producing caking free lactose powders can be achieved.

ACKNOWLEDGEMENTS

I always used wonder why research into industrial problems satisfies the requirements for a Doctorate in Philosophy.

Then I found a definition for Philosophy;

Philosophy, is like a blind man, in a dark room, looking for a black cat that isn't there.

Then I realised that this was the ideal qualification for this kind of research.

When it comes to lactose caking, I have been that blind man in the dark room. This is not to say that I didn't get any help during my search for the alleged black cat. Indeed there have been many people who have helped and encouraged me along the way.

Tony Paterson, my principal supervisor, has spent many hours over the last few years helping me ponder the riddles of what is lactose. My other supervisors, Dr Richard Archer, Dr Dong Chen and Professor Ray Winger were also of great help, particularly at the two ends of the whole exercise. Dr Jim Hargreaves's initial work into lactose has also been invaluable to this work. Also thanks to Aaron O'Donnell for all his work in searching for the same lactose cat.

John Alger, Bruce Collins and Don McLean have aided me in the design and manufacture of many cat searching devices. Thank you all very much.

I would like to thank Ma and Pa Bronlund, my family and friends who have listened and encouraged me to complete this thesis. This was mostly in the form of questions like "Is your thesis finished yet John?". The people responsible for asking such questions know who they are and many will be hearing similar questions from me in the near future. Special thanks to Jules, Kath, Dave, Kim, Lynley, Stacey, Mike, Clint, Ross, Silvia and Inge.

Thanks to Marcel Gesterkamp, Stephen Kellam, David Hall, and John Thomas at (or once with) The Lactose Company of New Zealand at Kaponga for all their valuable assistance and financial backing for this project.

Most thanks of all goes to Susie, without whose love and support, none of this would have been possible. With this thing now complete we can breath a combined sigh of relief.

At the end of this work there was still no sign of the Black Cat, but I did find a small hard deposit of a white powdery substance in one corner.

TABLE OF CONTENTS

BSTRACT	11
CKNOWLEDGEMENTS	v
ABLE OF CONTENTS	. VII
IST OF FIGURES	. XII
IST OF TABLES	. XVII

CHAPTER 1 PROJECT OVERVIEW

1.1	PROBLEM DEFINITION	1.1
1.2 1.2.1 1.2.2	PROPOSED CAKING MECHANISMS HUMIDITY CAKING AMORPHOUS LACTOSE CAKING	1.2 1.2 1.2
1.3		1.2
1.4	OVERALL PROJECT AIMS	1.3

CHAPTER 2 PHYSICAL PROPERTIES OF LACTOSE

2.1	LACTOSE CHEMISTRY	•	•	 		 	 	 		•		•	•		• •	•	•				•	•	 2	<u>)</u>	1

2.2 2.2.1 2.2.2 2.2.3	LACTOSE FORMS CRYSTALLINE LACTOSE AMORPHOUS LACTOSE PARTIALLY AMORPHOUS LACTOSE	2.1 2.1 2.3 2.3
2.3 2.3.1 2.3.2 2.3 2.3	CHARACTERISATION OF LACTOSE POWDERS PARTICLE SIZE DISTRIBUTION DENSITY 3.2.1 Particle Density 3.2.2 Bulk Density, Tapped Density and Porosity	2.5 2.5 2.5 2.5 2.6
2.4 2.4.1 2.4.2 2.4 2.4	THERMAL PROPERTIES 2 SPECIFIC HEAT CAPACITY 2 EFFECTIVE THERMAL CONDUCTIVITY 4.2.1 Experimental Measurement of Effective Thermal Conductivity 2.4.2.1.1 Measurement by the Infinite Cylinder Method 2 2.4.2.1.2 Measurement by the Guarded Hot Plate Method 2 4.2.2 Summary of Lactose Thermal Conductivity Measurements 2	2.7 2.7 2.7 2.7 2.9 2.12 2.13

2.5	PROPERTIES OF LACTOSE SOLUTIONS	2.13
2.5.1	SOLUBILITY	2.13
2.5.2	SURFACE TENSION	. 2.14
2.6	CLOSURE	2.15

CHAPTER 3

MOISTURE RELATIONS IN LACTOSE

3.1		3.1
3.2	THE DEGREE OF BINDING OF MOISTURE IN LACTOSE	3.1
3.2.1		3.1
3.2.3	BOUND AND FREE MOISTURE USED IN THIS WORK	3.3
3.3	MOISTURE SORPTION ISOTHERMS	3.3
3.3.1	PUBLISHED LACTOSE ISOTHERM DATA	3.4
3.3	3.1.1 Crystalline Lactose	3.4
3. 	3.1.2 Amorphous, Spray-dried and Freeze-dried Lactose	3.5
<u>১.১.८</u> २२२	CAPERIMENTAL MEASUREMENT OF LACTOSE ISOTHERM DATA	3.0
3.3.4	B-LACTOSE	3.14
3.3.5	Amorphous Lactose	3.16
3.3.6	MIXTURES OF CRYSTALLINE AND AMORPHOUS LACTOSE	3.18
3.4 3.4.1 3.4.2 3.4.3	Rates of Moisture Sorption on to Lactose 3 Sorption on to α-Lactose Monohydrate 3 Sorption on to Amorphous Lactose 3 Sorption on to Mixtures of Crystalline and Amorphous Lactose 3	8.20 3.21 3.22 3.28

3. 5		3.31
3.6	WATER ACTIVITY MEASUREMENT	3.33
3.7	PROPERTIES OF AMORPHOUS LACTOSE	3.34
3.7.1	GLASS TRANSITION TEMPERATURE	3.34
3.7.2	VISCOSITY OF AMORPHOUS LACTOSE	3.36
3.7.3	QUANTIFICATION OF AMORPHOUS LACTOSE IN LACTOSE POWDERS	3.37
3.7.4	CRYSTALLISATION OF AMORPHOUS LACTOSE	3.38
3.7	7.4.1 Product of Amorphous Lactose Crystallisation	3.38
3.7	7.4.2 Crystallisation Kinetics	3.41
	3.7.4.2.1 Polymer Crystallisation Kinetics	3.41
	3.7.4.2.2 Prediction of Crystallisation Rate in Amorphous Lactose	3.43
3.7.4	.2.2.1 Sorption-desorption phenomena	3.46
3.7.4	.2.2.2 β to α -lactose conversion	3.47
3.7.4	.2.2.3 Crystallisation rate	3.49
3.8	EFFECTIVE MOISTURE DIFFUSIVITY IN A PACKED BED OF LACTOSE	3.54
3.9	CLOSURE	3.55

CHAPTER 4

MODELLING HEAT AND MOISTURE TRANSPORT IN BULK LACTOSE

4.1		. 4.1
4.2	FORMULATION OF TRANSPORT MODEL	4.1
4.2.1	CONCEPTUAL MODEL	. 4.1
4.2.2	Assumptions	. 4.2
4.2.3	VALIDITY OF ASSUMPTIONS	. 4.2
4.:	2.3.1 Local Thermal Equilibrium	. 4.2
4.2	2.3.2 Local Moisture Equilibrium	. 4.3
4.2	2.3.3 Negligible Convection	. 4.4
4.2	2.3.4 Negligible Heat and Moisture Transport Due to Changes in Air Density	. 4.4
4.2	2.3.5 Mode of Moisture Movement	. 4.5
4.2	2.3.6 Other Assumptions Made	. 4.5
4.2.4	MATHEMATICAL FORMULATION	. 4.6
4.3	NUMERICAL SOLUTION OF TRANSPORT MODEL	4.6
4.3.1	SELECTION OF NUMERICAL SOLUTION METHOD	. 4.6
4.3.2	NUMERICAL SOLUTION	. 4.7
4.4		4.8
4.4.1	CHECKS AGAINST PREVIOUSLY VALIDATED SOLUTIONS	. 4.8
4.4.2	NUMERICAL ERROR CHECKING	. 4.9
4.4.3	MATHS CHECKING SUMMARY	. 4.9
A E	MATURNATION MODEL EVALUATION	1 10
4.0		4. IU
4.5.1	MODEL PREDICTIONS	4.10
4.		4.10

4.5.1.2	Water Vapour Concentration	4.11
4.5.1.3	Absolute Humidity	4.11
4.5.1.4	Relative Humidity	4.12
4.5.1.5	Moisture Content	4.13
4.5.2 HEAT	LOSSES DUE TO THERMAL EXPANSION	4.14
4.5.3 EXPE	RIMENTAL DATA COLLECTION	4.14
4.5.3.1	Experimental Apparatus Design	4.14
4.5.3.2	Experimental Data Collection	4.16
4.5.4 MODE	L PREDICTION ACCURACY	4.19
4.5.4.1	Temperature Prediction Accuracy	4.19
4.5.4.2	Surface Relative Humidity Prediction Accuracy	4.20
4.6 CLOS	SURE	1.27

CHAPTER 5 CAKING MECHANISMS

5.1	INTRODUCTION
5.2 5.2.1 5.2.2 5.2.3 5.2.4	CAKING MECHANISM OVERVIEW5.1HUMIDITY CAKING5.2AMORPHOUS SUGAR RE-CRYSTALLISATION5.3ALTERNATIVE MECHANISMS5.4EFFECT OF PRESSURE AND CRYSTAL CONTACT AREA5.4
5.3	CAKING MECHANISMS UNDER CONSIDERATION IN THIS WORK
5.4 5.4.1 5.4.2 5.4 5.4 5.4 5.4 5.4.3 5.4.4	CAKING STRENGTH MEASUREMENT5.6CAKING STRENGTH REQUIREMENTS FOR THIS WORK5.7AVAILABLE STRENGTH MEASUREMENT METHODS5.74.2.1Tensile Strength5.74.2.2Shear Strength and Cohesion Tests5.84.2.3Compaction and Compressibility Measurements5.84.2.4Other Methods5.9METHOD DEVELOPMENT5.9METHOD PERFORMANCE5.10
5.5 5.5.1 5.5.2 5.5.3 5.5.4	CAKING BY LIQUID BRIDGING AND SUBSEQUENT DRYING5.12MECHANISM OVERVIEW5.12LIQUID BRIDGING5.12SOLID BRIDGING5.16SUMMARY OF THE LIQUID BRIDGING AND DRYING CAKING MECHANISM5.17
5.6 5.6.1 5.6.2 5.6.3 5.6.4	CAKING DUE TO AMORPHOUS LACTOSE FLOW AND CRYSTALLISATION5.18MECHANISM OVERVIEW5.18RUBBER BRIDGING AND STICKING5.19RELATIVE RATES OF AMORPHOUS FLOW AND CRYSTALLISATION5.203.1Sticking of Thin Layers of Amorphous Lactose5.21RELEVANCE OF AMORPHOUS LACTOSE TO CAKING IN BULK LACTOSE5.24
5.7	CLOSURE

CHAPTER 6 PREDICTION OF CAKING IN BULK LACTOSE

6.1	INTRODUCTION
6.2 6.2.1 6.2.2 6.2.3 6.2.4 6.2.5 6.2.6	CAKING IN PURELY CRYSTALLINE LACTOSE POWDERS6.1OVERVIEW OF CAKING IN CRYSTALLINE LACTOSE6.1INCLUSION OF STRENGTH PREDICTION INTO THE TRANSPORT MODEL6.1EXPERIMENTAL DATA COLLECTION6.3EVALUATION OF CAKING STRENGTH PREDICTIONS6.5INVESTIGATIONS OF CONDITIONS REQUIRED FOR LUMPING IN BULK LACTOSE6.6AVOIDANCE OF LUMPING IN PURELY CRYSTALLINE LACTOSE6.10
6.3	CAKING IN FRESHLY MILLED OR DRIED LACTOSE CONTAINING AMORPHOUS
	LACTOSE
6.3.1	OVERVIEW OF REQUIREMENTS FOR AMORPHOUS LACTOSE RELATED CAKING
6.3.2	MODIFICATIONS TO THE TRANSPORT MODEL TO INCLUDE THE EFFECTS OF AMORPHOUS
622	
0.3.3	MIGRATION 6.12
634	PREDICTIONS OF MOISTURE TRANSPORT WITH AMORPHOUS LACTOSE CRYSTAL USATION 6.13
6.3	4.1 Crystallisation to Anhydrous Lactose Product
6.3	$8.4.2$ Crystallisation to α -Lactose Mono-hydrate
6.3.5	EXPERIMENTAL VALIDATION
6.3.6	MODEL PREDICTION ACCURACY
6.3.7	AVOIDANCE OF AMORPHOUS LACTOSE RELATED CAKING IN BULK LACTOSE
6.4	CLOSURE

CHAPTER 7

CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

7.1		. 7.1
7.2	SUGGESTED FUTURE RESEARCH	. 7.2
Refe	ERENCES	. 8.1
Арре	ENDIX A-1 NOMENCLATURE	A1.1
Арре	ENDIX A-2 PSYCHROMETRIC PROPERTIES OF AIR	A2.1
Арре	ENDIX A-3 TRANSPORT MODEL FORMULATION	A3.1
Арре	ENDIX A-4 FINITE DIFFERENCE APPROXIMATIONS	A4.1
Арре		A5.1

LIST OF FIGURES

Figure 2.1	α-Lactose chemical structure.	2.1
Figure 2.2	Muta-rotation between lactose forms	. 2.1
Figure 2.3	Electron micrograph of α -lactose monohydrate at 2000x magnification	on 2.3
Figure 2.4	Electron micrograph of β -lactose at 2000x magnification	2.3
Figure 2.5	Electron micrograph of <i>supertab</i> lactose at 100x magnification	2.3
Figure 2.6	Particle size distributions of lactose powders	. 2.5
Figure 2.7	Typical heating curve for an infinite cylinder full of bulk lactose	2.10
Figure 2.8	Semi-log plot of lactose cylinder heating time temperature history	2.10
Figure 2.9	Solubility of lactose as a function of temperature	2.14
Figure 2.10	Surface tension of saturated solutions of lactose	2.15
Figure 3.1	Literature isotherm data for α -lactose monohydrate	. 3.5
Figure 3.2	Summary of amorphous lactose isotherm data from the literature	. 3.6
Figure 3.3	Moisture sorption isotherm of α -lactose monohydrate	. 3.8
Figure 3.4	Third stage sorption isotherm model predictions for α -lactose	2 10
Eiguro 3.5	Moisture content versus conjulary radius for special dense of leaters	3.10
Figure 5.5	monohydrate	2 1 2
Figuro 3.6	Capillary appendix isotherm for a lastose monohydrate	3.13 2.14
Figure 3.0	Capitally condensation isotherm for α -factose mononlyulate \dots	5.14
Figure 5.7	salt solutions for 1 month	3 16
Figure 3.8	Moisture sorption isotherm for amorphous lactose	3 18
Figure 3.9	Effect of small amounts of amorphous lactose on the sorption isother	m
i igui e eie	of α -lactose samples as predicted from the additive isotherm model	3.19
Figure 3.10	Sorption isotherm of <i>supertab</i> lactose	3.19
Figure 3.11	Sorption rates measurement apparatus	3.20
Figure 3.12	Rates of sorption of moisture on to crystalline lactose	3.21
Figure 3.13	Comparison of observed moisture content with that predicted	
0	from bulk air relative humidity	3.22
Figure 3.14	Rate of moisture sorption on to spray dried amorphous lactose	
0	subjected to a step change in relative humidity from 0 to 43.5% RH	3.23
Figure 3.15	Comparison of experimental sorption rates with analytical solution .	3.25
Figure 3.16	Comparison of moisture diffusivity of some foods as a function of	
-	moisture content	3.26
Figure 3.17	Predicted rates of moisture sorption into varying thicknesses of	
	amorphous lactose	3.30
Figure 3.18	Comparison of drying techniques for the measurement of	
	moisture content in α-lactose monohydrate	3.31
Figure 3.19	Glass transition temperature of amorphous lactose as a function of	2 25
		5.55

Figure 3.20	Dependence of viscosity on temperature above glass transition temperature predicted by the WI E equation	3 36
Figure 3.21	Isothermal DSC plot for amorphous lactose at 65°C and 0.22.3	3.45
Figure 3.22	Some manual DSC plot for amorphous factose at 05 C and 0.22 $u_w \dots$	5.15
rigure 0.22	crystallisation product	3 45
Figure 3.23	Comparison of moisture sorption results for amorphous lactose from	5.45
1 igure 0.20	Niediek (1982) and isotherm determined from this work at 20° C	3 46
Figure 3.24	Conversion of β to α -lactose monohydrate as indicated by moisture	5.10
rigare 0.2 r	uptake for ß and supertab lactose	3 47
Figure 3.25	Predicted conversion from β to α -lactose after one month	3 48
Figure 3.26	Moisture content change in amorphous lactose during crystallisation	3 50
Figure 3.27	Crystallinity as a function of time during amorphous lactose	5.50
rigure 0.27	crystallisation	3 50
Figure 3.28	Avrami plot for amorphous lactose crystallisation	3.50
Figure 3.29	Prediction of crystallisation progress using Avrami equation	3 52
Figure 3.30	Comparison of crystallisation times between this work and	5.52
rigure 0.00	the literature	3 52
Figure 3.31	Avrami model predictions of time to reach 90% crystallinity	3 53
	Avram model predictions of time to reach 2070 crystaminty	5.55
Figure 4.1	Conceptual model for heat and moisture transport in bulk lactose	. 4.1
Figure 4.2	Temperature time profile for the heating of a 100mm slab	4.10
Figure 4.3	Temperature profile through lactose slab	4.11
Figure 4.4	Water vapour concentration time profile within the slab	4.11
Figure 4.5	Humidity time profile for lactose slab	4.12
Figure 4.6	Relative humidity profile development through the lactose slab	4.13
Figure 4.7	Moisture content profile development through the lactose slab	4.13
Figure 4.8	Transport model validation experimental rig	4.16
Figure 4.9	Experimental temperature profiles for a 78mm lactose slab	4.17
Figure 4.10	Experimental surface relative humidity profile for a 78mm slab	4.18
Figure 4.11	Experimental temperature profiles for a 100mm lactose slab	4.18
Figure 4.12	Experimental surface relative humidity profile for 100mm slab	4.19
Figure 4.13	Predictions for 78mm slab temperature profiles	4.20
Figure 4.14	78mm slab surface relative humidity profile predictions	4.21
Figure 4.15	Effect of porosity on surface relative humidity predictions	4.21
Figure 4.16	Effect of diffusivity on surface relative humidity predictions	4.22
Figure 4.17	Effect of initial temperature on surface relative humidity predictions	4.23
Figure 4.18	Effect of initial water activity on surface relative humidity predictions	4.23
Figure 4.19	Effect of slab thickness on surface relative humidity predictions	4.24
Figure 4.20	Sorption isotherm model fitting limits	4.24
Figure 4.21	Effect of changing isotherm shape on surface relative humidity	
	predictions	4.25
Figure 4.22	Best attempt at surface relative humidity predictions	4.26
Figure 4.23	Model predictions for 100mm slab experimental temperature profiles	4.26
Figure 4.24	Model predictions for 100mm slab experimental surface relative	
	humidity profiles	4.27

Figure 5.1	Humidity caking mechanism 5.	5
Figure 5.2	Amorphous related caking mechanism 5.	6
Figure 5.3	Strength measurement apparatus 5.1	0
Figure 5.4	Typical force distance profiles for lactose samples using multi pin	
	penetrometry method 5.1	1
Figure 5.5	Powder strength after one day equilibration time	3
Figure 5.6	Powder strength after ten days equilibration time	3
Figure 5.7	Powder strength after fifty days equilibration 5.1	4
Figure 5.8	Powder strength after one day as a function of moisture content 5.1	5
Figure 5.9	Solid bridge strength as a function of equilibrium relative humidity	_
- : - : :	prior to drying	1
Figure 5.10	Comparison of solid bridge strength to originating liquid bridge	
	strength 5.1	7
Figure 5.11	Comparison of sticking and crystallisation times in amorphous	
	lactose 5.2	0
Figure 5.12	Additive isotherm method for quantification of amorphous lactose	
	content of freshly milled lactose sample	2
Figure 5.13	Freshly milled lactose strength compared with purely crystalline	
	powder strength stored over saturated salt solutions for one week 5.2	2
Figure 5.14	Strength of freshly milled lactose compared with strength of	
	crystalline lactose stored for fifty days over saturated salt solutions . 5.2	3
Figure 6.1	Typical strength development in a 100mm lactose slab subjected to	
U U	a 20°C temperature gradient	2
Figure 6.2	Typical strength profile through lactose slab at steady state when	_
	a 20° C gradient has been applied 6	3
Figure 6.3	Experimentally measured steady state strength profiles through	2
rigure 0.0	lactose slabs with applied temperature gradients for approximately one	
	day	А
Figure 6.4	Predicted and experimentally measured strength profiles for lactose	1
rigure 0.4	slab experiments	5
Eiguro 6 5	Strongth profile for slobe of increasing thickness as a function of	5
rigure 0.5	the fractional position into the surface	6
	Effect of initial product water estivity on cold surface strength and	0
Figure 6.6	Effect of initial product water activity on cold surface strength and	7
	$T = \frac{1}{2} \left[\frac{1}{$	7
Figure 6.7	lypical relative humidity profile for the cooling of a lactose slab	~
	from one side	8
Figure 6.8	Maximum strength as a function of initial water activity	8
Figure 6.9	Temperature profile through lactose slab cooled evenly from both	_
	sides	9
Figure 6.10	Strength profile through slab cooled evenly from both sides 6.	9
Figure 6.11	Comparison of moisture migration rates for increasing amorphous lactose	3
	fractions	2
Figure 6.12	Predictions of moisture transport with crystallisation for powder	
	containing 2.5% amorphous lactose at 20°C after subjection to 20°C	
	temperature gradient 6.1	4

Figure 6.13	Moisture migration in a lactose slab starting at a water activity	
	of 0.25 a_w showing no signs amorphous lactose crystallisation	6.14
Figure 6.14	Moisture migration with amorphous lactose crystallisation to	
	α-lactose monohydrate	6.15
Figure 6.15	Steady state amorphous lactose fraction profile through the	
	lactose slab	6.16
Figure 6.16	Hammer mill arrangement	6.16
Figure 6.17	Method used to raise water activity of lactose slab	6.17
Figure 6.18	Schematic of rig used to provide constant RH air	6.18
Figure 6.19	Experimental temperature profiles for 78 mm lactose slab	
	containing 4.5% amorphous lactose	6.19
Figure 6.20	Experimental surface relative humidity profiles for a 78 mm lactose	
	slab with 4.5% amorphous lactose initially present	6.19
Figure 6.21	Two-point isotherm results for lactose samples taken before and	
	after the slab experiment	6.20
Figure 6.22	Comparison of experimental and predicted temperature profiles	
	with amorphous lactose present	6.21
Figure 6.23	Comparison of experimental and predicted surface relative	
	humidity profiles with amorphous lactose present	6.22
Figure 6.24	Comparison of predictions for crystallisation to α -lactose	
	monohydrate with 20 and 50 nodes	6.24

LIST OF TABLES

Table 2.1	General properties of α -lacose monohydrate and β -lactose anhydride \ldots	2.2
Table 2.2	Summary of crystalline lactose used in this work	2.2
Table 2.3	Amount of amorphous lactose present on various industrial lactose	
	samples	2.4
Table 2.4	Particle density of the various crystalline lactose forms	26
Table 2.5	Bulk density, tapped density and porosity of lactose powders	2.6
Table 2.6	Solutions for the temperature change in an infinitely long cylinder	2.9
Table 2.7	Summary of thermal conductivity measurements by infinite cylinder	
	method	2.11
Table 2.8	Summary of thermal conductivity measurements by guarded hot plate	
	method	2.12
Table 3.1	tss-lsotherm parameters for α -lactose monohydrate	3.10
Table 3.2	GAB isotherm parameters for amorphous lactose	3.17
Table 3.3	Summary of amorphous lactose layer thickness estimates for	
	different lactose grades	3.28
Table 3.4	Constants <i>n</i> and <i>K</i> for the Avrami equation	3.42
Table 3.5	Effective moisture diffusivity models for flow in porous media	3.55
Table 4.1	Summary of isotherm model parameters for limiting cases	4.25
Table 5.1	Comparison of multi point penetrometer readings with observational	
	tests	5.11
Table 6.1	Summary of experimental conditions for strength profile measurement	
	of lactose slabs under the influence of a temperature gradient	64
		.

,