

Table of contents

Scope of the thesis	I
Dedication	III
Acknowledgements	V
Table of contents.....	VII
Abbreviations and symbols	XI
1 Background.....	15
1.1 General aspects about whey and whey proteins.....	15
1.1.1 α-Lactalbumin.....	16
1.1.2 β-Lactoglobulin.....	18
1.1.3 Whey protein concentrates and isolates	20
1.2 Thermally induced denaturation of whey proteins.....	21
1.3 Kinetics of the heat-induced unfolding and aggregation of whey proteins.....	24
1.4 Mechanisms of heat induced gelation in systems containing whey proteins	27
1.5 Soluble aggregates, fluid gels and microparticles	32
2 Current state of knowledge.....	35
2.1 Separation methods and purification of whey proteins	35
2.2 Kinetics of the thermal induced denaturation and aggregation of whey proteins.....	36
2.3 Thermally induced gelation of whey proteins (quiescent gels)	45
2.4 Thermal gelation of whey proteins under shearing conditions (microparticulation).....	62
2.5 Spray drying of microparticulated whey proteins.....	71
2.6 Functional properties of microparticulated whey protein.....	72
2.7 Applications of microparticles in dairy products.....	76
3 Summary on the state of knowledge, motivation and objectives	81
4 Materials and methods	83
4.1 Whey protein fractionation.....	83
4.1.1 Raw materials and chemicals	83
4.1.2 Preparation of solutions and heating.....	83
4.1.3 Microfiltration	84
4.1.4 Ultrafiltration.....	84

Table of contents

4.1.5	Fractionation procedure.....	85
4.1.6	Study of the re-solubilization of aggregated α -La	86
4.2	Kinetics of the heat induced denaturation of whey protein fractions.....	87
4.2.1	Raw materials and chemicals	87
4.2.2	Preparation of the solutions.....	88
4.2.3	Heating of the solutions	88
4.3	Study of the gel properties without shearing	90
4.3.1	Heat induced gelation	90
4.3.2	Analysis of binding forces in heat set whey protein gels	90
4.3.3	Viscoelastic properties of the heat induced gels	91
4.3.4	Water holding capacity.....	92
4.4	Microparticulation.....	93
4.4.1	Microparticulation in a rheometer.....	93
4.4.2	Microparticulation in one stage closed loop.....	94
4.4.3	Microparticulation in pilot scale (three stage continuous process).....	96
4.4.4	Microparticulation in sequential form using a high pressure homogenizer	97
4.4.5	Particle size.....	99
4.4.6	Water holding capacity.....	99
4.4.7	Apparent viscosity	99
4.5	Spray-drying of microparticulated whey proteins	99
4.5.1	Spray-drying of microparticulated whey.....	99
4.5.2	Morphological analysis of the microparticles	101
4.5.3	Determination of solid content in suspensions of microparticulated protein.....	101
4.5.4	Determination of the powder moisture content (Karl Fischer titration)	101
4.6	Study of the functional properties of microparticulated whey proteins	101
4.6.1	Functional properties in model solutions.....	102
4.6.2	Application in ice cream	104
4.6.3	Application in yoghurt	106
4.7	Analytical methods common to all sections	108
4.7.1	Solids/Moisture content.....	108

Table of contents

4.7.2	Total protein content	108
4.7.3	Chromatographic analysis	109
4.7.4	DSC.....	111
4.7.5	Electrophoretic analysis (SDS-PAGE).....	111
4.7.6	Particle size analysis	115
4.7.7	Calcium concentration	116
4.7.8	Statistics.....	116
5	Results and discussion.....	119
5.1	Separation and purification of whey protein main fractions	119
5.1.1	Selective thermal precipitation of α -La.....	119
5.1.2	Separation of the precipitated fraction using diafiltration	125
5.1.3	Optimization of the diafiltration process	126
5.1.4	Study of the resolubilization of α -La after thermal precipitation	129
5.1.5	Effect of added citrate concentration	131
5.1.6	Effect of pH on resolubilization of α -La.....	132
5.1.7	Compositional evaluation of the separated fractions in batch.....	134
5.1.8	Assesment of the nativity of resolubilized α -La	136
5.2	Kinetics	138
5.2.1	Influence of pH on thermal denaturation of α -La and β -Lg	138
5.2.2	Influence of lactose on thermal denaturation of α -La and β -Lg	141
5.2.3	Influence of protein concentration and protein composition	146
5.3	Gelling properties under quiescent conditions (heat set gels)	149
5.3.1	Characterization of the binding forces in whey protein gels	150
5.3.2	Effect of pH and lactose concentration on the gel properties	152
5.4	Microparticulation of whey protein fractions (sheared gels)	157
5.4.1	Microparticulation in a rheometer	158
5.4.2	Microparticulation in one-stage Scraped Surface Heat Exchanger.....	161
5.4.3	Sequential processing with pre-heating followed by high pressure homogenization	173
5.4.4	Optimum conditions for particles with high denaturation degree and reduced size .	179
5.4.5	Validation of the results at pilot scale.....	179

Table of contents

5.5	Effects of spray drying on the size of microparticles	181
5.5.1	Characterisation of the starting product.....	181
5.5.2	Influence of drying temperature on particle sizes of MWP	182
5.5.3	Particle size of the aggregates after reconstitution in distilled water	185
5.5.4	Moisture content of the powder.....	186
5.5.5	Morphology	187
5.5.6	Validation of the results at pilot scale.....	189
5.6	Study of the functional properties of microparticulated whey proteins	191
5.6.1	Solubility	192
5.6.2	Stability of the particle size after with varying pH	192
5.6.3	Stability of particle size after repeated heating and homogenization.....	194
5.6.4	Surface hydrophobicity	196
5.6.5	Surface tension at the air/water interface.....	197
5.6.6	Emulsifying properties.....	198
5.7	Functional properties of microparticles in a dairy matrix.....	201
5.7.1	Ice cream	201
5.7.2	Yoghurt	204
6	General conclusions and outlook	207
6.1	Pilot-scale fractionation of α -La and β -Lg	207
6.2	Kinetics	208
6.3	Gelling behavior under quiescent conditions	209
6.4	Microparticulation.....	210
6.5	Spray-drying of microparticulated whey proteins	213
6.6	Functional properties of the microparticles.....	214
7	Summary	217
8	References.....	221