

# Improved Lactose Powder Properties by In-situ Coating with Additives during Spray Drying

Michael Brech and Justin J. Nijdam

Department of Chemical and Process Engineering, University of Canterbury, Christchurch, New Zealand

Email: michael.brech@pg.canterbury.ac.nz, justin.nijdam@canterbury.ac.nz

David Pearce and Payel Bagga

Fonterra Research Centre, Palmerston North, New Zealand

Email: {david.pearce, payel.bagga}@fonterra.com

**Abstract**—The ability of different additives to enrich on the droplet surface during spray drying of aqueous lactose solutions was investigated. The goal of this study was to find an additive coating that increases spray-dryer yield, improves powder flow and prevents caking due to lactose crystallization during storage. The additives tested were Nacaseinate (NaCas), gelatin, lecithin, anhydrous milk fat (AMF) and hydroxypropyl methylcellulose (HPMC). The aqueous lactose solutions had 10 weight% solids content with a lactose-to-additive dry weight ratio of 9:1. All additives enrich significantly on the droplet surface at the expense of lactose during spray drying. Every additive except AMF enhances the glass transition temperature at the particulate surface and therefore reduces particle stickiness, as demonstrated by improved powder flow and/or product yield compared with the pure lactose powder. Low product yield for HPMC-containing lactose powder can be explained by its low bulk density, which reduces the separation efficiency of the cyclone. Poor powder flow for lecithin-containing lactose powder can be explained by its agglomerated nature. AMF is not suitable as an additive due to a low product yield and poor powder flow, a result of stickiness due to the low melting point of fat. None of the additives improve the wetting time of lactose powder, since lactose is already highly hydrophilic, and most of the additives tend to make the particle surface more hydrophobic. NaCas and HPMC are the most promising additives with regards to improving powder flow. NaCas results in a higher product yield than HPMC, but HPMC is the only additive that provides a protective coating for lactose capable of preventing the formation of a hard brittle cake upon lactose crystallization during storage at ambient room conditions.

**Index Terms**—spray drying, lactose, stickiness, caking, crystallization, microencapsulation

## I. INTRODUCTION

Spray dried amorphous lactose powder is a common food component in the dairy industry prone to stickiness, which causes low spray drier yields and caking during subsequent storage [1], [2]. This occurs due to the highly hygroscopic nature of amorphous lactose, which absorbs moisture from the environment, reducing in viscosity to the extent that individual particles become sticky, inter-

particle bridges between adjacent particles form and crystallization occurs [1], [3]. More specifically, a phase transition occurs from the amorphous, solid-like state to a rubbery liquid-like state at a temperature known as the glass transition temperature [1]. Increasing the water content of the lactose reduces the glass transition temperature. In the rubbery state, the lactose molecules gain a certain degree of mobility, which allows them to re-orientate into crystalline structures, adding a brittle and hard quality to any cake that forms. Caking can be avoided in practice so that powder remains free-flowing and easy to handle by ensuring that the glass transition temperature stays above the storage temperature. This is normally achieved by storing the powder in a dry state.

Particle coating during spray drying, also called *in-situ coating*, exploits the phenomenon of solute segregation within the drying droplet. Several authors have shown that, during the spray drying of aqueous solutions of proteins and disaccharides, the surface active proteins preferentially accumulate at the droplet surface at the expense of the disaccharide, which results in higher protein concentrations on the surface than within the core of the spray dried particles [2], [4]-[6]. In addition to proteins, various surfactants, fats, large carbohydrates and polymers have also been found to enrich at the droplet surface at the expense of disaccharides during spray drying [6]-[9]. Spray dried lactose could benefit from in-situ coating of such “additives” during spray drying to increase the glass transition temperature of the particulate surface [10] and hence decrease particle stickiness, increase product yields and improve powder flows. The coating might also (or rather) act as a physical barrier against moisture entering into the particles to prevent lactose stickiness, or against lactose bridges forming between neighbouring particulates to avoid caking.

In this study, different coating materials are investigated with regards to their ability to 1) enrich on the droplet surface during drying, 2) provide a protective coating around the lactose in order to delay or prevent lactose crystallization and caking, and 3) increase product yield and improve functional powder properties, such as powder flow and wettability.

## II. MATERIALS AND METHODS

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The coating materials (additives) tested were sodium caseinate (written NaCas for the sake of brevity), lecithin, anhydrous milk fat (AMF) hydroxypropyl methylcellulose (HPMC) and gelatin. NaCas ( $M_w \sim 24$  kDa), lecithin ( $M_w \sim 0.75$  kDa) and AMF were supplied by Fonterra Research Centre (Palmerston North, New Zealand). HPMC ( $M_w \sim 22$  kDa), gelatin ( $M_w \sim 20$ -22 kDa) and  $\alpha$ -Lactose monohydrate ( $M_w \sim 0.34$  kDa) were supplied by Sigma Aldrich (Germany). Solutions of lactose and an additive with dry weight ratio of 9:1 and total solids content of 10 wt% were prepared in distilled water. AMF emulsions of 20 wt% solid content (95 wt% AMF, 5 wt% NaCas) for use as an additive were previously prepared by a 2-step homogenization. All solutions were spray dried in a laboratory spray drier (NIRO Atomizer, Copenhagen, Denmark) with dimensions of 175 x 92.5 cm at an air flow of  $105 \pm 5 \text{ m}^3 \text{ h}^{-1}$ , inlet/outlet temperatures of  $160/75 \pm 1 \text{ }^\circ\text{C}$ , atomization pressure of 0.6 bar, and solution feed rate and temperature of  $1.6 \pm 0.2 \text{ kg h}^{-1}$  and  $40 \pm 1 \text{ }^\circ\text{C}$ , respectively. Three repeats were performed for each type of powder to obtain an estimate of uncertainty for the different powder analyses.

Surface tension measurements of all solutions were performed prior to spray drying using the pendant drop technique (CAM 2008 surface tension meter). Five to ten pictures were taken for each droplet and 3 to 5 droplets were analysed for each solution to obtain an estimate of uncertainty.

X-ray photoelectron spectroscopy (XPS) was used to measure the surface composition of spray dried particles (Kratos Axis Ultra DLD, Manchester, UK). Relative atomic concentrations at the particle surface (10 nm depth resolution) of carbon, oxygen and nitrogen were recorded and the appropriate surface concentrations of the different powders were calculated using the matrix calculation described by Fäldt *et al.* [11].

Powder flowability was indirectly determined by measuring the fraction of powder that passed through a vibrated sieve with a mesh size of approximately 0.65x0.65 mm. The powder was vacuum dried before measurement to remove all moisture. Three repeat runs were performed to obtain an estimate of uncertainty.

Wettability of powders was measured according to a modified form of the method described by Freudig *et al.* [12]. Essentially, the time for 0.1 g of powder placed on water at  $50 \text{ }^\circ\text{C}$  to sink below the surface was measured. Bulk density of vacuum-dried powder was determined by filling a 10 mL measuring cylinder with 1 mg of previously vacuum dried powder and tapping it constantly until no further volume reduction was observed. Three to five repeat measurements were performed.

The particle size distribution of the spray dried powder was measured by a Microtrac particle size analyser (Microtrac ASVR X100, Leeds & Northrup, U.K.) using isopropanol as the suspending agent. Any given suspension was ultra-sonicated for one minute before particle size was measured under constant stirring until no decrease in particle size was observed, which

suggested that the particle agglomerates had broken up. A Scanning Electron Microscopy (SEM) was used (Philips XL30S FEG) to capture images of the different powders, before and after the powder was stored at ambient conditions for one week to allow lactose to crystallize.

### III. RESULTS AND DISCUSSION

Fig. 1 shows that all tested additives dominated the surface of spray dried lactose/additive particles, despite the additive concentration only being 10% of the dry weight. For comparison, the surface concentrations of AMF, NaCas, gelatin, lecithin, and HPMC were 64%, 70%, 72%, 73%, and 90%, respectively. This demonstrates the ability of these materials to accumulate on the droplet surface at the expense of lactose during the short time-frame of spray drying, as also found in other studies [2], [4]-[9]. With the exception of AMF, a significant driving force for this accumulation is likely to be the surface activity of the tested additives [2], [4], [5], [8], [9]. Solute diffusion within the drying surface will also have an effect, with the diffusion rate dependant on molecular size of the solute and solution viscosity [6]. In addition, droplet drying time as influenced by air inlet temperature and droplet size would have an effect by limiting the time available for the solute to diffuse towards the surface [5]. Furthermore, surface accumulation may also be affected by thermodynamic aspects, such as equilibrium surface concentrations and maximum packing densities of adsorbed species, which depend on the charge, structure, flexibility and re-arrangement of the molecule at the surface [13], [14]. There is currently no conclusive evidence within the literature about which of these physical mechanisms dominates surface enrichment. Various mechanisms of fat enrichment on the droplet surface during drying have been proposed. Fäldt and Bergenstål [15] state that fat may leak out of the fat globules due to increasing emulsion instability when fat globules within a droplet approach each other as the drying droplet shrinks. Kim *et al.* [16] suggest that larger fat globules are preferentially present at the surface of emulsion droplets when leaving the atomization device and are disrupted during atomization and thus appear at high concentrations as free fat on the surface of emulsion droplets.

The following surface tensions of the aqueous solutions (1 wt% additive in solution) prior to spray drying were measured:  $63.2 \pm 0.9 \text{ mNm}^{-1}$  for lactose/gelatin,  $56.2 \pm 0.9 \text{ mNm}^{-1}$  for lactose/lecithin,  $53 \pm 3 \text{ mNm}^{-1}$  for lactose/AMF and  $51.3 \pm 0.7 \text{ mNm}^{-1}$  for lactose/NaCas and  $49.2 \pm 0.8 \text{ mNm}^{-1}$  for lactose/HPMC. The surface tension of pure water was  $72.5 \pm 0.4 \text{ mNm}^{-1}$ , which shows that the additives reduced surface tension and were therefore present at the air/water surface during drying [4], [17]. HPMC reduced surface tension more than the other additives due to its high surface activity. This correlates well with the relatively high surface concentration of HPMC compared with the surface concentrations of the other additives (Fig. 1). The

surface tension data obtained in this study for HPMC and NaCas agree with the data from other authors, who measured surface tensions between 46 and 48 mN m<sup>-1</sup> for 1 wt% HPMC solutions [8], [18] and 50 to 52 mN m<sup>-1</sup> for 1 wt%  $\beta$ -casein solutions [18].

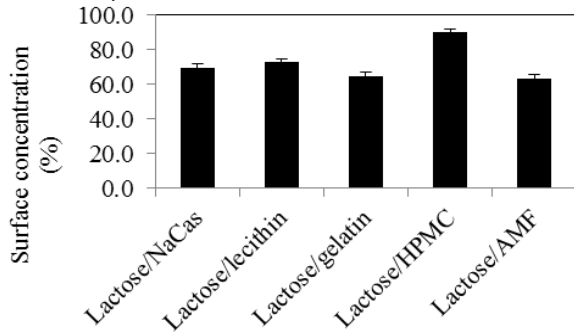


Figure 1. Surface composition of different spray dried lactose/additive powders (90/10 dry wt%)

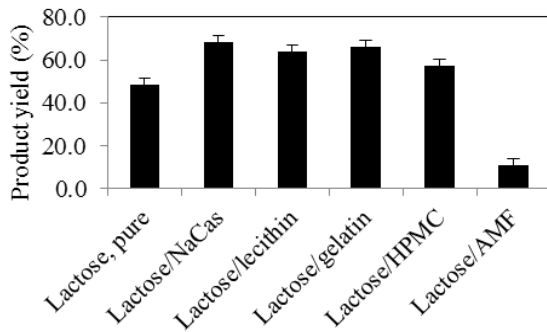


Figure 2. Effect of different coating additives (10 wt% of total solid content) on the spray drier yield

The spray drier yield increased considerably when using the additives NaCas, lecithin, or gelatin, while AMF resulted in poor yields compared with pure lactose. Due to lactose having a relatively small molecular size (molecular weight approximately 0.34 kDa), its glass transition temperature is lower than those of the additives NaCas, lecithin and gelatin, which have molecular weights ranging from 0.75 kDa to 24 kDa. This trend of increasing glass transition temperature with increasing molecular weight of a food polymer has been clearly demonstrated by Roos and Karel [19]. Hence, the enrichment of longer-chain molecules on the surface of a droplet, containing a relatively small molecule such as lactose, increases the overall glass transition temperature there and thus reduces particle stickiness. This results in higher spray drier yields compared with pure lactose, as also found by Wang and Langrish [2]. AMF was an exception due to its low fat melting temperatures (ranging from -40 to +40 °C [20]) and hence sticky nature, thus fat on the surface of spray dried lactose particles reduced the yield considerably. Lactose powder with HPMC had lower spray drier yields than lactose powders with NaCas, gelatin and lecithin. This is despite HPMC (~22 kDa), NaCas (~24 kDa) and gelatin (~20-22 kDa) all having similar molecular weights, which implies that these additives have similar glass transition temperatures and should therefore reduce surface stickiness equally as effectively. However, due to the

lower measured bulk density of lactose/HPMC powder compared with the other powders (Fig. 3), the particles may have been too light to be efficiently separated by the cyclone of the spray dryer, which resulted in lower product yields than occurred for the denser lactose/lecithin, lactose/NaCas and lactose/gelatin particles. This demonstrates that spray dryer yield in this work is affected not only by the stickiness of the particles, but also by the effect of particulate density on the efficiency of the spray dryer cyclone.

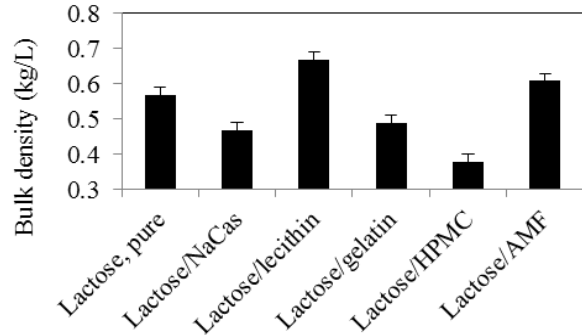


Figure 3. Effect of different coating additives (10 wt% of total solid content) on bulk density

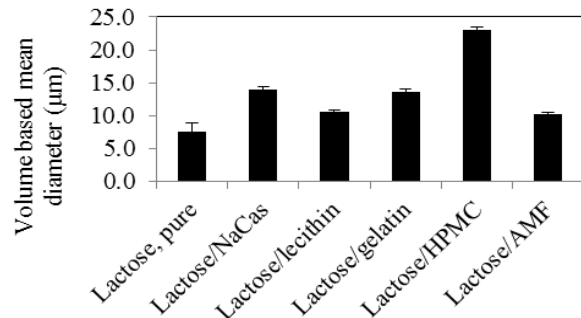


Figure 4. Effect of different coating additives (10 wt% of total solid content) on volume based mean diameter

The variation in bulk density with the addition of different additives can be explained by the flexibility of adsorbed molecules at the particle surface, which changes the visco-elasticity of the particle wall [21]. Dickinson [21]-[23] showed that the presence of flexible proteins such as caseins, and polymers such as HPMC, cause the formation of an elastic film on the air/water interface. HPMC in particular provides significant film elasticity due to its long highly flexible molecular structure [8], [16]. This allows more expansion of particulates when vapour vacuoles form inside the particles during drying [5]. The same conclusions can be drawn for NaCas and gelatin and explains the observed increase in particle size and reduction in bulk density of lactose powders that contain these additives compared with lactose powders that contain non-flexible molecules, such as lecithin or fatty acids (in AMF) on their particle surfaces (Fig. 3 and 4). Millqvist-Fureby and Smith [9] measured a reduction in particle size (compared with pure lactose) when lecithin was used as an additive, whereas our study showed an increase in particle size as shown in Fig 4, although the bulk density also increased, as shown in Fig 3. However, Fig. 5 shows that, in our work,

lecithin/lactose particles formed strong agglomerates which may not have broken up into individual particles during the particle size measurement.

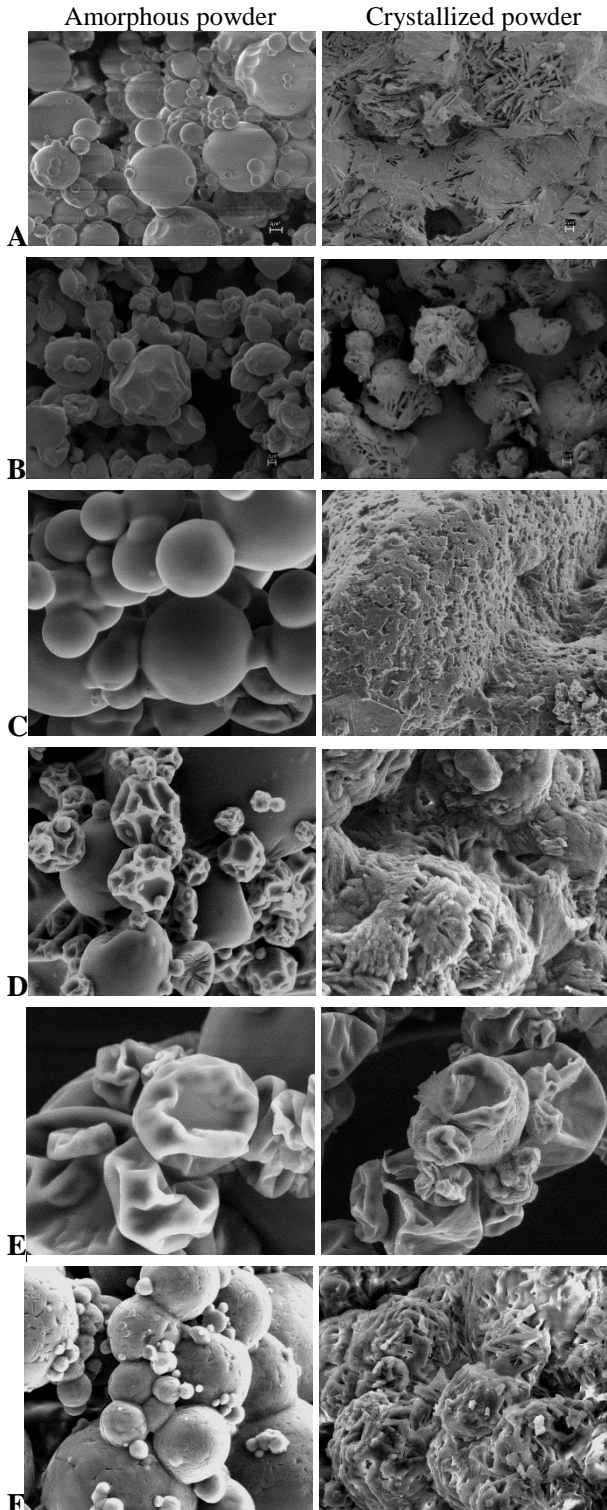


Figure 5. SEM photographs (5000 magnification). Effect of different coating additives (10 wt% of total solid content) on the particle morphology: left: Amorphous right: crystallized. A) Pure lactose, B) lactose/NaCas, C) lactose/lecithin, D) lactose/gelatin, E) lactose/HPMC, F) lactose/AMF

Fig. 5 shows the effect of the additive on the particle morphology. Pure spray dried lactose formed smooth spherical particles (Fig. 5A), while the addition of NaCas,

gelatin and HPMC resulted in folded particle surfaces (Fig 5B,D,E). These results agree with the findings of other researchers on morphology of lactose particles that contain long flexible molecules, such as caseins or HPMC, which increase the elasticity of the particle wall [2], [5], [8]. Particle-wall elasticity allows more expansion of internal-vacuole-containing particulates in the hotter regions of the spray dryer and deflation of these particulates and folding of their surfaces in cooler regions of the drier [5]. The folding of the particle surface was clearest for HPMC and gelatin. Higher elasticities of HPMC- and gelatin-containing films may account for this finding, as both molecules are well-known for their ability to cross-link and form gel networks [23]-[27]. However, the bulk densities of lactose/gelatin and lactose/NaCas powders were the same within uncertainties (Fig. 3), while lactose/HPMC powder had a significantly lower bulk density (Fig. 3) and larger particles (Fig. 4), which suggests that HPMC-containing films caused the highest wall elasticity of the additives tested, most likely due to the more flexible structure of this polymer. AMF caused significant agglomeration of the spray dried particles due to the high stickiness of fat (Fig. 5F). Using lecithin as additive resulted in spherical particles similar in appearance to the pure lactose particles (compare Fig. 5A and C), although for reasons that are not clear, the lactose/lecithin powder appeared to agglomerate more. Nevertheless, the lack of folding on the particulate surfaces of both the lactose/lecithin and pure lactose powders indicates that lecithin did not increase the elasticity of the particle wall in the way that HPMC, gelatin and NaCas did, most likely due to its smaller molecular size and non-flexibility. This agrees well with the findings of Elverson and Millqvist-Fureby [9].

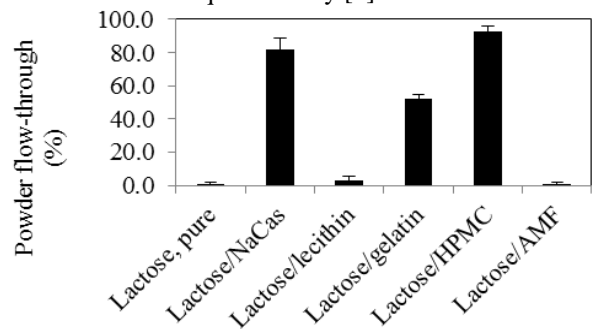


Figure 6. Effect of different coating additives (10 wt% of total solid content) on powder flow through a vibrating sieve lactose, B) lact/NaCas, C) lact/lecithin, D) lact/gelatin, E) lact/HPMC, F) lact/AMF

Fig. 6 shows the powder flows of the different spray dried powders. Using NaCas, HPMC and gelatin as additives increased the powder flow significantly, compared with pure lactose. The spray dried powders were all vacuum-dried to standardise them before measuring their flowability. Thus, during the flowability tests, the glass transition temperature of the particulate surfaces was well above ambient temperature so that particle stickiness could not directly affect powder flow. However, the influence of particle stickiness on the flowability test was indirect through weak agglomeration

of particles when the powder was in the collection jar beneath the cyclone of the spray drier for up to 30 minutes during spray drying. Temperatures of around 75 °C (corresponding to the air outlet temperature of the spray dryer) and relatively high moisture contents (between 3 to 5 %) in the collection jar meant that the glass transition temperature (at least of lactose) was exceeded by more than 10 °C, and hence particle stickiness could have occurred. The powder flow through the vibrating sieve was affected by observed powder agglomeration, as indicated by a coarser texture and more cohesive appearance of the powder, with improved powder flows being measured for less agglomerated powders such as those containing HPMC, NaCas and gelatin. The larger size of individual particles in these powders compared with pure lactose powder, as seen in Fig. 4, may have further aided in reducing powder agglomeration in the collection jar with a consequent improvement in powder flow. The best powder flows were measured for lactose/HPMC and lactose/NaCas powders. Why their flowability was higher than that of lactose/gelatin powder is not completely clear, given the similar molecular sizes and thus glass transition temperatures of these additives (NaCas  $M_w \sim 24$  kDa; HPMC  $M_w \sim 22$  kDa; gelatin  $M_w \sim 20$ -22 kDa). This could be explained by the larger measured size of lactose/HPMC particulates compared with lactose/gelatin particulates (Fig. 6). In addition, the lactose/gelatin particulates were much more folded than the lactose/NaCas particulates (Fig. 5). The increased surface folding may have increased friction between the particles and reduced the powder flowability for lactose/gelatin powder compared with lactose/NaCas powders, which both have comparable particle sizes. Lecithin did not improve powder flow considerably compared with gelatin, NaCas and HPMC, perhaps due to its relatively low glass transition temperature, which might explain the more agglomerated state observed for the lactose/lecithin powder (Fig. 5C). The smaller particulate size of lactose/lecithin powder compared with lactose/NaCas, lactose/HPMC and lactose/gelatin powders would also contribute to its lower powder flow. An AMF coating did not cause any improvement in the powder flowability, due to the stickiness of fat and hence highly agglomerated nature of the powder. This finding agrees well with the low product yield measured for AMF-coated powder (Fig. 2).

In Fig. 7 the wetting times of the different powders are plotted. Since amorphous lactose is a very hygroscopic, polar molecule, it wetted instantly upon exposure to the water surface. A similar observation was made when using lecithin as an additive, which is also used as coating additive for milk powders to improve their wettability [9]. All other additives caused an increase in wetting time. Possible reasons for this could be the relatively high buoyancy of the larger particles of lower density, in particular for the lactose/HPMC powder, which showed a significant longer wetting time than the other powders. On the other hand, the adsorbed species might change the hydrophilic surface of the amorphous

lactose to a more hydrophobic surface, considering that proteins, surfactants and surface active polymers such as NaCas and HPMC tend to orientate their hydrophobic parts towards the air-phase upon adsorption, depending on their flexibility and the distribution of their hydrophilic and hydrophobic residues along their chain length [17]-[18].

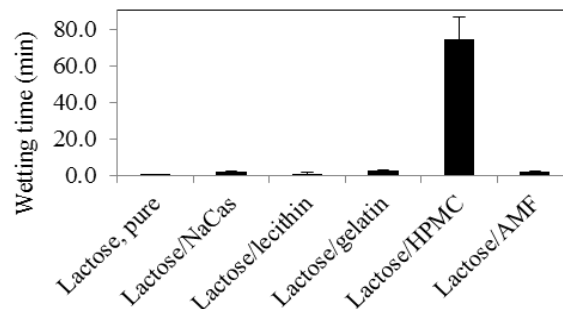


Figure 7. Effect of different coating additives (10 wt% of total solid content) on the wetting time of the powder lactose, B) lact/NaCas, C) lact/lecithin, D) lact/gelatin, E) lact/HPMC, F) lact/AMF

Powders were stored at ambient room conditions for one week to absorb moisture from the environment, and thus to allow lactose crystallization and powder caking. No additive tested in this paper prevented lactose crystallization and caking of the powder. Therefore, although the additives enriched at the particle surface, as can be seen in Fig. 1, they could not form an adequate physical barrier that prevented moisture diffusion into the particles and hence crystallization of lactose. Haque and Roos [10] showed, however, that additives such as NaCas and gelatin can at least delay crystallization and reduce crystallization rates.

The crystalline cake structure can be seen in Fig. 5. Only HPMC and, to a lesser extent, NaCas provided sufficient structural support at the surface of the particle to prevent a complete collapse of the particulate structures, as was observed for pure lactose and the other lactose/additive powders. In addition, lactose/HPMC powder was the only powder that did not transform into a hard brittle powder cake, but rather formed a softer pliable cake. Lactose/NaCas powder transformed into a hard, brittle cake upon crystallization similar to the other powders, although distinct particulates were preserved (Fig. 5). HPMC is a flexible polymer that may form a dense network (film) on the particle surface [27], capable of adding structural support to the particle during lactose plasticisation and subsequent crystallization. NaCas is a flexible polymer-like protein [15], [22], which may also have similar, although clearly not as effective, networking and film forming properties as HPMC. HPMC appeared to form a physical barrier on the particle surface that reduced caking by preventing lactose crystals in neighbouring particles from growing into each other. Gelatin was also expected to provide a certain degree of structural support due its film forming capability at the surface [24]-[26], but our work showed that HPMC and NaCas were significantly more effective in this respect.

#### IV. CONCLUSION

Adding small amounts of surface active additives to a lactose solution prior to spray drying can increase powder yield and flowability of lactose powders. High-molecular-weight surface-active molecules, such as proteins and polymers, accumulate on the droplet surface during drying at the expense of the smaller-molecular-weight lactose, and reduce particle stickiness by providing a coating material with relatively high glass transition temperature. NaCas is the most promising additive for improving both spray dryer yield and powder flow. HPMC results in the best powder flow, however it causes low powder bulk densities, relatively low product yields and long wetting times. AMF offers no improvement in product yield or powder flow due to its sticky nature, caused by the low melting-point of fat. Lecithin and gelatin both increase product yield, although, of these two additives, only gelatin results in a significant improvement in powder flow. None of the additives tested improve wettability above that of the pure lactose powder. Only HPMC prevents lactose from forming a hard brittle cake upon crystallization during storage at ambient room conditions.

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**Michael Brech**, born in Frankfurt (Main) in Germany on the 5th of October 1982, is a postgraduate student at the Department of Chemical and Process Engineering of the University of Canterbury, Christchurch, New Zealand. He completed his undergraduate studies at the University of Karlsruhe (KIT) in Germany (2004-2010) in the subject of *Bioengineering*, where he specialized in "Biopharmaceutical purification" and "Product Engineering & Formulation".

Michael Brech completed several research projects during his undergraduate studies, one of them at the University of Almeria, Spain in 2008, where he investigated the growth and biodiesel production of different microalgae under varying growth and stress conditions. During his studies, he worked for 2 years at the Institute of Mechanical Process Engineering (MVM) of the KIT as Research Assistant (RA), where he investigated the agglomeration and mixing behaviour of different titanium dioxide powders within varying mixer parameter. At



the end of his undergraduate studies 2010, Michael Brech did a research project at the Australian Institute of Bioengineering and Nanotechnology (AIBN), Centre of Biomolecular Engineering, of the University of Queensland in Brisbane, Australia, where he developed an innovative, economic purification process for self-assembling peptides.

After his graduation 2010 as “Diplom Ingenieur” at the KIT, Michael Brech worked as Research Assistant (RA) at the AIBN to continue working on the purification of several designed, self-assembling peptides, where he submitted two scientific journal papers on *Bioseparation* with Prof. Anton Middelberg and also patented the developed purification process. In April 2011 he joined the Department of Chemical and Process Engineering at the University of Canterbury, Christchurch, New Zealand for studying towards a Ph.D., in cooperation with Fonterra Ltd., New Zealand, where he is currently investigating different coating technologies for spray dried lactose. Two scientific journal papers on this subject have been written and will be submitted in 2013.



**David L. Pearce** is the Process Engineering Manager at Fonterra Research and Development Centre, Palmerston North, New Zealand. He was awarded a B.E. (Hons) in Chemical and Process Engineering, with First Class Honours from the University of Canterbury, Christchurch, New Zealand in 1986. He completed his Ph.D. from the same university in 1990. The topic of his Ph.D. thesis was “Solubility of triglycerides in supercritical CO<sub>2</sub>”.

His supervisors were Dr. Pat Jordan and Prof. Arthur Williamson.

After completing his Ph.D. he spent 12 months as a postdoctoral worker in the high pressure thermodynamics lab of Prof. Jakob de Swaan Arons

at the Technische Universiteit in Delft, The Netherlands, followed by a period as a process engineer for John Brown Engineers (now Aker Process BV) in Zoetermeer, The Netherlands. Since joining the NZ dairy industry in 1994, he has worked mainly in milk powder production. During this time he has co-supervised 8 Ph.D. and Masters students, published more than 20 articles in reviewed journals, 28 conference papers and co-authored one book chapter and many internal Fonterra reports.

Dr. Pearce currently manages a team of 12 engineers and technologists providing engineering expertise to Fonterra’s product and process development projects. He is on the organising committees of the International Symposia of Spray Dried Dairy Products, is a Chartered Chemical engineer and a Corporate Member of the Institute of Chemical Engineers. He was awarded the Freehills Award for Excellence in Chemical Engineering at the 2012 CHEMECA conference.



**Justin J. Nijdam** was educated at the Chemical and Process Engineering Department at Canterbury University in New Zealand (PhD in 1998). He spent a number of years in New Zealand, Australia and Germany undertaking research in the areas of drying and particle technologies often using computational fluid dynamics (CFD) as a research tool. He returned to Canterbury University in 2007, where he is currently a senior lecturer teaching classes in

CFD, fluid mechanics, heat and mass transfer, design and analysis of experiments and technical communication. His research interests include wood processing (drying, sterilisation by Joule heating) and food processing (spray dryers, fluidised beds, filters, mixers).