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Physical and rehydration properties of milk protein concentrates: comparison of spray-dried and extrusionporosified powders

Jean-Marie Bouvier • Maxime Collado • Darren Gardiner • Maxwell Scott • Pierre Schuck

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Abstract Milk protein concentrates (MPCs) must be completely dispersed and dissolved in water to fully present their functional properties. However, MPC powders exhibit low solubility and dispersibility and require long rehydration times. In addition, the powders have limited shelf life as their solubility decreases during storage. It is proposed that improvements in the instant powder drying process will improve the rehydration behaviour of MPC powders. In this paper, the physical and rehydration properties of extrusion-porosified MPC powders were examined and compared with those of conventional spray-dried MPCs. This work clearly confirmed that spray-dried MPC powders showed a low dispersibility index (38%), and after 5 h of rehydration, no sub-micron casein micelles were observed in the dispersed phase. In contrast, extrusion-porosified MPC powders exhibited a very high dispersibility index (96%), and the dispersed phase contained only sub-micron particles after 2 h of rehydration. The results suggest that the improved dispersibility is attributed to the dissociation of casein micelles caused by the extrusion porosification treatment prior to drying. Changes in the microstructure of extrusion-porosified MPC powder particles are proposed to support the significant improvement of the rehydration behaviour.

J.-M. Bouvier (⊠) • M. Collado Clextral SAS, ZI de Chazeau, BP. 10, 42702 Firminy, France e-mail: jmbouvier@clextral.com

D. Gardiner Murray Goulburn Co-op, Ltd, 140 Dawson Street, Brunswick, VIC 3056, Australia

M. Scott Max Scott Consulting Pty, 11 Shamrock Close, Narre Warren, VIC 3805, Australia

P. Schuck INRA, UMR 1253 STLO, 35042 Rennes, France

P. Schuck Agrocampus Rennes, UMR 1253 STLO, 35042 Rennes, France



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1 Introduction

The worldwide food industry produces millions of tons of instant powders each year. The powders reduce transport and storage costs by eliminating water from products and are also utilised for industrial purposes. They are manufactured using accepted technologies such as spray drying, drum drying and freeze drying, in particular. There has been increasing interest in the development of highly functional powders such as techno- and bio-functional ingredients which require good cold water solubility in a relatively short time whilst maintaining the integrity of the powder components. Thus, improving powder functionality, particularly the rehydration characteristics, is becoming critically important. Moreover, the food industry is dedicated to more cost-effective processing technologies regarding capital intensity, energy consumption and process flexibility.

Sustainable production of consistent, highly functional powders is a critical consideration for the dairy industry. In fact, dairy powder production is a key area where real improvements are actively sought to upgrade the functionality of dairy powders [such as milk protein concentrates (MPCs)]. Such powders are characterised by a low solubility index and poor cold water solubility, as a substantial fraction of material remains undissolved when subjected to normal conditions of reconstitution (at room temperature and moderate agitation in a relatively short time, less than 1 h) (Schuck et al. 1994; Havea 2006). Improvement in solubility can be achieved by dissolving at high temperatures and/or at high shear rates and by increasing rehydration time (to several hours), which is not practical or economical for most industrial processes. Moreover, MPC powders show further loss of solubility upon storage, especially when maintained above room temperature and at moisture contents higher than 5% (Anema et al. 2006; Baldwin and Truong 2007). The loss of solubility of the MPCs is not due to the formation of insoluble material. Mimouni et al. (2010a) showed that the release of casein micelles from powder particles is the rate-limiting step of the MPC rehydration process, as the slow-dissolving material consisted almost entirely of caseins and colloidal salts, whereas the whey proteins, lactose and non-micellar minerals (sodium and potassium) dissolved almost instantaneously into the water. This has been confirmed by the recent study undertaken by Schokker et al. (2011) where no changes at a molecular or intra-micellar level were observed between powders of differing solubility, hence indicating that the loss of solubility is probably controlled by higher order structural changes. Chemical additions such as salt (Schuck et al. 2002) and chelating agents (Bhaskar et al. 2001), dairy ingredient additions such as sodium caseinate (Schokker et al. 2011) and physical treatment such as high-pressure processing (Udabage et al. 2012) are technological aids that can delay the loss of solubility; but their effects on the rate of MPC rehydration are still limited.

This paper will present and compare the physical and rehydration properties of MPC powders that have been obtained by two different processes: the conventional spray drying process and the extrusion porosification process.

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2 Materials and methods

2.1 MPC powders

Two types of MPC powders were used in this study: one spray-dried MPC85 (socalled SD-MPC) which was provided by Murray Goulburn Co-op Ltd. (Brunswick, Victoria, Australia); and one extrusion-porosified MPC85 (so-called EP-MPC).

EP-MPC was produced by the extrusion porosification process which enabled high solids concentrates to be dried (Durand et al. 2008). The process consisted of three main processing units:

- High solids vacuum evaporation: the unit was fed with 21% total solids MPC85, provided by Murray Goulburn Co-op Ltd., Brunswick, Victoria, Australia. The concentrate was processed at a temperature of approximately 48 °C to produce a high solids MPC concentrate of 38% dry solids.
- Twin screw extrusion–aeration: the high solids MPC concentrate was fed into an intermeshing twin screw extruder (32-mm screw diameter) and mixed with a gas (carbon dioxide) to produce a high solids textured foam; while controlling the time–temperature load in the screw–barrel assembly. The processing conditions were as follows: temperature of 20 °C; residence time of less than 1 min; pressure of 6–8 bar; CO₂ concentration of approximately 0.85–0.95 standard litre per kilogramme of concentrate.
- Intensified powder drying: the twin screw extruder-aerator produced a high solids textured foam (38% dry solids) that was delivered to a drying chamber. In the drying chamber, atomized particles of the foam were dried by direct contact with hot air where inlet and outlet temperatures were typically in the ranges of 170-220 and 55-75 °C, respectively; while the residence time varied in the range of 15-20 s. The resulting porous structure of the powder particles led to faster moisture transfer during drying which allowed the outlet temperature of the particles to be reduced significantly. It varied in the range of 45-55 °C, which is 15-20 °C lower than the outlet temperature observed in conventional spray drying. EP-MPC powder particles were not subjected to secondary agglomeration.

SD- and EP-MPC powders had similar compositions that were: 86.7% *w/w* milk proteins (with approximately 20% whey protein and 80% casein), 4.1% *w/w* lactose, 1.4% *w/w* fat, 2% *w/w* ash, and 5.8% *w/w* water. The particle size characteristics were as follows: for SD-MPC, d(0.1), d(0.5) and d(0.9) were 37, 73.2 and 128.9 μ m, respectively; whereas for EP-MPC, d(0.1), d(0.5) and d(0.9) were 32.8, 66 and 125.7 μ m respectively. After manufacturing, the SD-MPC and EP-MPC powders were conditioned in sealed polyethylene-paper sacks and stored at less than 10 °C for 1 month until analysed.

2.2 Physical characterization of MPC powders

Bulk (BD) and tapped (TD) densities were determined by use of the Carr's method (Carr 1965) according to Haugaard Sorensen et al. (1978) and Schuck et al. (2012). Bulk density (BD) was the weight of a defined volume of powder (1 m^3) in its natural



state with no compaction; expressed in kilogramme per cubic metre. Tapped density (TD) was the density obtained after controlled compaction of the powder; expressed in kilogramme per cubic metre. True density (TrD) of the powder particles was determined by gas pycnometry with both helium and nitrogen on a Pycnomatic ATC instrument (Thermo Fisher Scientific, Courtaboeuf, France); expressed in kilogramme per cubic metre. Occluded air (OA) content was defined as the difference between the true density and the tapped density, by use of the following equation: $OA=(1/TD - 1/TrD).10^5$; expressed in cubic centimetre per 100 gramme. Each sample was analysed in triplicate.

2.3 Scanning electron microscopy

For scanning electron microscopy (SEM) measurement, powder samples were placed on a carbon double-sided adhesive tape and put on an aluminium sample holder, then coated with 1 nm of platinum–palladium alloy by use of Cressington 208R sputter coater (Cressington, England). Powder samples were examined with a field emission scanning electron microscope (S4300SE/N, Hitachi, Japan) at 1.2 kV (magnification range of ×70 to ×700). Un-chopped and chopped (chopping at ambient temperature by use of an injector blade) SD- and EP-MPC powder samples were observed. The images were produced at CSIRO Materials Science and Engineering (Belmont, Victoria, Australia).

2.4 Rehydration properties

The rehydration behaviour of MPC powders (SD- and EP-MPC powders) was characterised by the wettability index, the dispersibility index and the solubility index; which were determined according to Schuck et al. (2012). The wettability index was determined from the results obtained during the rehydration of the powders without agitation; it is expressed in second. MPC powder is considered wettable if the wettability index is lower than 120 s. The dispersibility index was determined from the results obtained during the rehydration and controlled filtration; it is expressed in percent. MPC powder is considered dispersible if the dispersibility index is equal to or higher than 95%. And the solubility index was determined from the results obtained during the rehydration of the powders after agitation and controlled centrifugation; it is expressed in percent. MPC powder is considered dispersible if the solubility index is equal to or higher than 95%. And the solubility index was determined from the results obtained during the rehydration of the powders after agitation and controlled centrifugation; it is expressed in percent. MPC powder is considered soluble if the solubility index is equal to or higher than 95%. Each sample was analysed in triplicate.

2.5 Rehydration kinetics

Particle size distributions were measured by static light scattering using a Malvern Mastersizer 2000 apparatus (Malvern Instruments Ltd, Malvern, UK) operating with two laser sources (466 and 632.8 nm). A sample of 200 ml of 8% (w/w) MPC powder suspension was prepared (mixing powder and reverse osmosis filtered water using an RW20 overhead stirrer at 500 rpm for 2 min), and added at 22 °C to the dispersion unit (Hydro 2000G cell unit with a recirculation pump speed of 1,250 rpm). The light scattering pattern was obtained every 5 min over 5 h. Malvern measurements allowed



the specific surface area (SSA) of the dispersed particles to be determined. SSA was defined as the total surface area of the particles by their total weight; expressed in square metre per gramme.

3 Results and discussion

3.1 Physical characterization of powders

As shown in Table 1, the bulk and tapped densities of SD-MPC powder are 356 and 519 kg.m⁻³, respectively; whereas for EP-MPC powder, the bulk and tapped densities are 215 and 430 kg.m⁻³, respectively. This shows that the bulk and tapped densities of EP-MPC powder are notably lower than those of SD-MPC powder. This result is confirmed by the volume of occluded air that is notably higher in EP-MPC particles (146 mL.100 g^{-1}) than that of SD-MPC particles (108 mL.100 g^{-1}). Images from SEM of MPC powders are presented in Fig. 1 (panels a and b for SD-MPC powder; panels c and d for EP-MPC powder) at two different magnifications (a and c: ×200; b and d: ×500). The surfaces of both powders were essentially smooth. Figure 1 shows significant differences between the microstructures of the two powders. The particles in the SD-MPC powders had irregular shapes, rather dense shells and large internal holes. In contrast, the EP-MPC particles had spherical shapes and highly porous matrices with numerous holes and communicating channels. The diameters of the holes were in the range of approximately 1 to 10 µm. This observation concurs with the previous results relative to lower bulk and tapped densities, and higher volume of occluded air of EP-MPC powder.

Particle microstructure is determined by the characteristics of the powder drying process. As demonstrated in previous work (Tamime et al. 2007), the smooth surface appearance of powder particles was attributed to the compaction and shrinkage of the proteins (the casein micelles in particular) during air drying. Therefore, it would be assumed that both the SD- and EP-MPC particles would have smooth surface appearances. However, there are important differences in the matrix structures of the SD-MPC particles and the EP-MPC particles. In fact, conventional spray drying of MPC produces particles with a hollow interior surrounded by a highly compacted

Physical and rehydration properties	EP-MPC	SD-MPC	
Bulk density (BD), kg.m ⁻³	215±2	356±3	
Tapped density (TD), kg.m ⁻³	430±2	519±2	
True density (TrD), kg.m ^{-3}	$1,150\pm 2$	1,170±2	
Occluded air (OA), mL.100 g ⁻¹	$146{\pm}2$	107 ± 1	
Wettability index, s	$>120\pm0$	>120±0	
Dispersibility index, %	96±1	38±1	
Solubility index, %	98±0	99±0	

 Table 1
 Physical and rehydration properties of extrusion-porosified (EP) and spray-dried (SD) milk

 protein concentrate (MPC) powders





Fig. 1 Typical scanning electron microscopy pictures of spray-dried milk protein concentrate powder (a and b) and extrusion-porosified milk protein concentrate (c and d) powder

shell. Such structure is generated during the final stage of drying at the end of the falling drying rate regime, when no more wet areas remain on the particle surface. The drying rate is then determined by the rate of heat transfer and the rate of moisture transfer in the opposite direction through the shell. In this regime, the particle temperature increases, and the air that is trapped inside the particle expands; hence leading to particles with a hollow interior. In contrast, the extrusion porosification process produces particles with numerous holes of various sizes throughout the matrix, which result from the foam structure of MPC concentrate combined with the release of CO_2 in the early stage of air drying; hence increasing the porosity and specific surface area of EP-MPC powder particles. At the end of the falling drying rate regime of the extrusion porosification process, the high particle porosity increases the rate of moisture transfer within the particle and reduces the particle temperature increase, as confirmed by the relatively low outlet temperature of 45–55 °C.

3.2 Powder rehydration

Generally, MPC powders must be fully dispersed and dissolved to express their functional properties. Therefore, powder rehydration in water should be fast and complete. Table 1 gives the rehydration standards (such as wettability index, dispersibility index and solubility index) for EP- and SD-MPC powders. Wettability index exceeded 120 s for both powders; while solubility index was 98 and 99% for EP- and SD-MPC respectively. These are typical rehydration characteristics of MPC material. It is well-known that milk protein concentrates show relatively a high wettability

index which typically exceeds 120 s due essentially to the surface composition of MPC powder particles. Our results are in accordance with previous studies (Gaiani et al. 2007; Schuck et al. 2007). Though EP-MPC powder shows higher internal porosity than SD-MPC powder, its wettability index still exceeds 120 s. Further post-processing agglomeration may improve particle wettability.

When comparing dispersibility of the two powders, the EP-MPC powders showed a much higher dispersibility index than SD-MPC powders. It is well-known that casein-based powders (such as micellar casein powder, spray-dried MPC powder) show low dispersibility indexes. Hence, the dispersibility index of SD-MPC powders was typically 38% in this study. Unexpectedly, EP-MPC powders had a very high dispersibility index of 96%. With MPC material, it must be noted that dispersibility is the rate-controlling step in rehydration. Therefore, the high dispersibility index of the EP-MPC powders is significant as it should greatly decrease hydration time and dramatically improve the rehydration conditions from both technical and economic standpoints.

To study the difference between EP- and SD-MPC powders during rehydration, the rate of powder reconstitution was examined by measuring particle size reduction in the dispersed phase versus reconstitution time. Figure 2 presents the evolution of the size of powder particles in the dispersed phase. Hence, d(0.1), d(0.5) and d(0.9) are plotted versus time for both EP- and SD-MPC powders. For SD-MPC powders, the graph typically shows a slight increase of particle size (case of d(0.5) and d(0.9), or large particles, in particular) at low reconstitution time (after 5-min rehydration) due to particle swelling, and then a slow decrease of particle size when reconstitution time increases due to gradual erosion of primary powder particles. After 5-h rehydration, SD-MPC powder particles still remained large as their size ranged roughly between 20 and 80 μ m; and no sub-micron casein micelles were observed. This result confirms that reconstitution of conventional spray-dried MPC powders requires an extended time for full dissolution. This is in accordance with previous



Fig. 2 Particle size versus time during powder rehydration at 8% total solids and 22 °C; *open symbols*: spray-dried milk protein concentrate; *closed symbols*: extrusion-porosified milk protein concentrate; *white diamond, black diamond*: d(0.1); *white square, black square*: d(0.5); *white triangle, black triangle*: d(0.9)



studies of Mimouni et al. (2009, 2010a). The loss of reconstitutability of MPC material is dramatically affected by storage times. In this study, the MPC powders were stored for 1 month before analysis, thus decreasing the reconstitutability of SD-MPC material. According to previous studies (Mimouni et al. 2010a; Schokker et al. 2011; Udabage et al. 2012), reconstitution time of SD-MPC at 22 °C, after 1-month storage, might be in the approximate range of 480 to 720 min.

In contrast, the hydration of MPC powder is considerably modified when it is extrusion-porosified. In fact, as shown in Fig. 2, EP-MPC powders exhibited very different rehydration behaviour as sub-micron particles appeared after 17 min of rehydration. After approximately 2 h of rehydration, the dispersed phase contained only sub-micron particles, which means that full dissolution of MPC material was complete. Sub-micron particles in the colloidal suspension were in the approximate range of 80 to 200 nm, which according to Mimouni et al. (2010b), strongly suggests that these particles were casein micelles. This result shows that extrusion porosification processing considerably improves the reconstitutability of MPC material, as the reconstitution time of EP-MPC at 22 °C, after 1-month storage, was approximately 120 min. With EP-MPC powders, it must be noted that particle dispersibility is no longer the rate-limiting step in the hydration process. The evolution of particle size versus time (Fig. 2) showed a very sharp decrease in the particle size after some time of hydration, which depended upon the initial size of the particles: the lower the initial particle size, the shorter the time. This observation can be regarded as a "structural collapse" of the powder particles, which occurred after complete diffusion of water throughout the powder particles. Collapse time was directly correlated to the size of the powder particle. For instance, in the present study, the collapse times were approximately 17, 38 and 127 min for d(0.1), d(0.5) and d(0.9), respectively. Thus, the rehydration rate of EP-MPC powders is essentially controlled by water diffusion into porous powder particles, rather than by particle dispersibility in water.

Another way to compare the rehydration behaviour of EP- and SD-MPC powders consists of examining the specific surface area (or SSA) of MPC particles as a function of time during reconstitution at 22 °C (Fig. 3). After 15-min rehydration, SD-MPC powders showed a slight increase of SSA from 0.1 to 0.2 m^2 .g⁻¹ due to particle swelling. After 5-h rehydration, no significant increase of SSA was observed. In contrast, after 15-min rehydration, EP-MPC powders showed considerable increase of SSA from 0.1 m².g⁻¹ to about 12 m².g⁻¹, due to structural collapse of MPC particles. Then a gradual increase of SSA was observed from 12 m².g⁻¹ to about $30 \text{ m}^2.\text{g}^{-1}$ as the reconstitution time increased from 15 min to about 115 min. After approximately 115-min rehydration, SSA increased sharply during 15 min from about 30 to 45 m^2 .g⁻¹; and then a gradual increase of SSA was observed from 45 m^2 .g⁻¹ to about 60 m².g⁻¹ as the reconstitution time increased from 130 to 300 min. The SSA of EP-MPC material in suspension increased by approximately 2 orders of magnitude during the first 15 min of rehydration, confirming that powder dispersibility was not the rate-limiting step in the hydration process. When compared with SD-MPC, SSA of EP-MPC material in suspension increased by almost three orders of magnitude during the first 2 h of rehydration. This confirmed that extrusion porosification processing had a very positive effect on the microstructure of MPC powder particles that considerably enhanced the porosity of powder particles and hence remarkably improved powder reconstitutability.

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Fig. 3 Specific surface area versus time during powder rehydration at 8% total solids and 22 °C (*white circle*: spray-dried milk protein concentrate; *black circle*: extrusion-porosified milk protein concentrate)

It is worth noting that the extent of the SSA increase of EP-MPC powder particles during rehydration (an increase of two or three orders of magnitude) coherently corresponds to complete dissociation of sub-micron casein micelles. In fact, when a 70- μ m powder particle is converted to 0.1- μ m finer particles, the SSA of the particle system is multiplied by a factor of 700.

3.3 Changes in the microstructure of extrusion-porosified MPC particle

In a recent study, Mimouni et al. (2010b) showed that the surface microstructure of rehydrated spray-dried MPC85 powder was porous and characterised by a coarse network of casein micelles that appeared to be linked by short bridges and direct micelle-micelle interactions. When comparing fresh and aged spray-dried MPC powders, the authors observed that when aged powder (after 2-months storage) was rehydrated, it presented increased density and compaction of the casein micelles on the surface; hence leading to lower porosity and higher apparent extent of interactions between micelles. In another study that was published concomitantly, Mimouni et al. (2010a) demonstrated that water penetration into the powder particles was rather fast as non-micellar components (such as Na, K, lactose and whey proteins) were released into solution rapidly and quantitatively during rehydration of both fresh and aged spray-dried MPC85 powders. Thus, the limitation in the release of casein micelles into the solvent was unlikely to be a limitation of the water transfer to the interior of the powder particles; but more likely to be restricted by micelle-micelle interactions, which would become even stronger after extended storage of the powder in the dry state. In the present study, it was clearly demonstrated that SD-MPC powder showed a typical, high wettability index (>120 s which means low wettability due essentially to surface composition of MPC powder particles) and a low dispersibility index (38% which means very slow release of casein micelles into the solution). Hence, after long-term rehydration (5-h testing), no sub-micron population of individual casein micelles were observed in the solution. The



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results of this study are consistent with the work of Mimouni et al. (2010a, 2010b). The microstructure of a primary particle of conventional spray-dried MPC powder is schematically illustrated in Fig. 4 (a). The particle would have a large internal hole surrounded by a shell composed of casein micelles and non-micellar casein material. The shell might possibly show a few smaller holes (micrometre scale) throughout its matrix. Internal holes do not generally communicate with each other. In addition, according to Mimouni et al. (2010a) the shell would show pores (nanometer scale) between tightly packed clusters of linked casein micelles. Although these pores permit water transfer throughout the particle matrix, spray-dried MPC powder would exhibit a low dispersibility index because of numerous micelle–micelle interactions that restrain the release of casein micelles into the solution.

In contrast, extrusion-porosified MPC85 powders show remarkable rehydration properties. The results of this study have clearly demonstrated that EP-MPC powders showed a typical, high wettability index (>120 s) and a high dispersibility index (96%); after 2 h of rehydration, MPC material appeared to be completely dispersed into the solvent as the solution was composed of only sub-micron casein micelles. Hence, the question that interests product designers and process developers is "what changes in the microstructure of EP-MPC powder particles supports such improvement of the rehydration behaviour?" The microstructure of a primary particle of extrusion-porosified MPC powder is schematically illustrated in Fig. 4 (b). The proposed microstructure is based on two levels of particle porosity. The first level relates to numerous holes of different sizes (hole size in the approximate range of 1 to $10 \mu m$) that are randomly distributed throughout the particle matrix; this has been clearly observed in Fig. 1d. It is suggested that the internal holes resulted from the foam structure of MPC concentrate due to micromixing CO₂ gas into the viscous concentrate. The particle porosity was further reinforced by CO₂ flashing off from MPC foam droplets in the early stages of air drying in the drying chamber. The



Fig. 4 Hypothesis for the microstructure of primary powder particles (a: spray-dried milk protein concentrate; b: extrusion-porosified milk protein concentrate)

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second level of porosity was linked to a network of communicating capillaries (nanometer scale) separating casein micelles, that were generated around the micelles during CO_2 flash off in the early stages of air drying.

In conventional milk spray drying, injecting carbon dioxide into the feed before atomization has been proposed for controlling powder bulk density (Pisecky, 1997). In this case, CO₂ is injected into the pipeline prior to the high-pressure pump, at a slightly higher pressure by means of centrifugal pump. So far, CO_2 injection in conventional spray drying has not significantly improved hydration properties, probably because the CO_2 is not fully micromixed with the milk concentrate. The texture of the resulting foam remains incomplete and possibly unstable; hence leading to powder particles still composed of tightly packed clusters of linked casein micelles which restrain their release into water. In the extrusion–aeration unit of the extrusion porosification process, CO_2 gas is uniformly mixed with MPC material in the twin screw extruder-aerator; while part of the CO₂ is dissolved into water depending upon pressure and temperature conditions in the processing unit. Twin screw extrusion technology is a wellrecognised mixing technology that can achieve a high degree of intensive mixing for viscous media. In extrusion porosification of MPC material, twin screw extrusion technology should allow the protein material and dissolved CO₂ to be mixed thoroughly, and potentially the casein micelles to be dissociated when CO2 is flashed off in the early stages of air drying; thereby preventing the development of micelle-micelle interactions. The resulting powder particle would show numerous holes distributed throughout the protein matrix (casein micelles and non-micellar casein material) in combination with a network of communicating capillaries due to the dissociation of casein micelles. This would allow easy water penetration and a quick release of casein micelles during rehydration. A few clusters of bonded casein micelles could subsist after extrusion porosification depending upon the processing conditions.

The use of physical processes which dissociate the casein micelles, such as highpressure treatment before spray drying have already been applied by Udabage et al. (2012), to enhance rehydration properties of MPC powders. However, with the extrusion porosification treatment, the extent of dissociation of casein micelles is much higher, and the rehydration behaviour of MPC powders is considerably improved due to the intense micromixing in the twin screw extruder–aerator. Extrusionporosified MPC powder particles show a high dispersibility index because of their specific porous texture (micrometre-scale holes combined with nanometer-scale capillaries throughout the particle matrix) and the extent of dissociation of casein micelles. Of course, the hypothesised changes in the microstructure of EP-MPC powder particles as compared to that of SD-MPC powder particles must be confirmed and further work applying microscopy techniques in combination with other experimental approaches will be necessary to support or refine this hypothesis. This should be taken into account in the near future in order to monitor extrusion porosification conditions, and hence to decrease the rehydration time to under 1 h for MPC material.

4 Conclusion

This study has shown that MPCs can be processed at high solids content and still exhibit high dispersibility and solubility. This has been performed using an



innovative instant powder drying process, called the extrusion porosification process. The key segment of this process is the extrusion–aeration unit which allows thorough mixing of a gas (CO_2 in this study) into a viscous, high solids concentrate while controlling pressure and temperature conditions, resulting in an organised, porous texture after powder drying. This study proved that the use of extrusion porosification to texturize MPC85 material significantly upgraded the hydration behaviour of the MPC powder particles. Complete dispersion/dissolution of the extrusion-porosified powder was obtained within 2 h of rehydration, while conventional spray-dried MPC powder particles generally require over 8 h for rehydration. When compared to conventional spray-dried MPC powders, the results suggest that the dissociation of casein micelles is responsible for the reduction of rehydration time.

In addition to powder texturization, the extrusion porosification process shows promise in instant powder processing from the operational standpoint, as it is well designed for sustainable processing (energy savings, reduction of capital intensity), and operational flexibility (ability to process various mixes and formulations while controlling the powder texture). This should be developed and discussed in future publications.

References

- Anema SG, Pinder DN, Hunter RJ, Hemar Y (2006) Effects of storage temperature on the solubility of milk protein concentrate (MPC85). Food Hydrocoll 20:386–393
- Baldwin AJ, Truong GNT (2007) Development of insolubility in dehydration of dairy milk powders. Food Bioprod Process 85:202–208
- Bhaskar GV, Singh H, & Blazey ND (2001) Milk protein products and processes, Patent WO01/ 41578.
- Carr RL Jr (1965) Evaluating flow properties of solids. Chem Eng 72:163-168
- Durand D, Bouvier J-M, Maller G, Scott M, Stevenson SR, Roberts SJ, Brisset A (2008) Procédé et installation de production en continu d'un produit poreux en poudre, Patent FR 2 907 029.
- Gaiani C, Schuck P, Scher J, Desobry S, Banon S (2007) Dairy powder rehydration: influence of protein state, incorporation mode, and agglomeration. J Dairy Sci 90:570–581
- Haugaard Sorensen I, Krag J, Pisecky J & Westergaard V (1978) Analytical methods for dry milk products, A/S Niro Atomizer (Editor), Copenhagen, Denmark, pp. 18–21
- Havea P (2006) Protein interactions in milk protein concentrate powders. Int Dairy J 16:415-422
- Mimouni A, Deeth HC, Whittaker AK, Gidley MJ, Bhandari BR (2009) Rehydration process of milk protein concentrate powder monitored by static light scattering. Food Hydrocoll 23:1958– 1965
- Mimouni A, Deeth HC, Whittaker AK, Gidley MJ, Bhandari BR (2010a) Rehydration of high-proteincontaining dairy powder: slow- and fast-dissolving components and storage effects. Dairy Sci Technol 90:335–344
- Mimouni A, Deeth HC, Whittaker AK, Gidley MJ, Bhandari BR (2010b) Investigation of the microstructure of milk protein concentrate powders during rehydration: alterations during storage. J Dairy Sci 93:463–472
- Pisecky J (1997) Handbook of milk powder manufacture. Niro A/S, Copenhagen, Denmark, p 159
- Schokker EP, Church JS, Mata JP, Gilbert EP, Puvanenthiran A, Udabage P (2011) Reconstitution properties of micellar casein powder: effects of composition and storage. Int Dairy J 21:877–886
- Schuck P, Piot M, Méjean S, Fauquant J, Brulé G, Maubois JL (1994) Dehydration of an ultra-clean milk and micellar casein enriched milks. Lait 74:47–63
- Schuck P, Davenel A, Mariette F, Briard V, Méjean S, Piot M (2002) Rehydration of casein powders: effects of added mineral salts and salt addition methods on water transfer. Int Dairy J 12:51–57
- Schuck P, Méjean S, Dolivet A, Gaiani C, Banon S, Scher J, Jeantet R (2007) Water transfer during rehydration of micellar casein powders. Lait 87:425–432



- Schuck P, Dolivet A, Jeantet R (2012) Analytical methods for food and dairy powders. Wiley-Blackwell, Oxford, pp 145-154
- Tamime AY, Robinson RK, Michel M (2007) Microstructure of concentrated and dried milk products. In: Tamime AY (ed) Structure of dairy products. Blackwell Publishing Ltd., Oxford, UK, pp 104-133
- Udabage P, Puvanenthiran A, Yoo JA, Versteeg C, Augustin MA (2012) Modified water solubility of milk protein concentrate powders through the application of static high pressure treatment. J Dairy Res 79:76-83

