

High shear treatment of concentrates and drying conditions influence the solubility of milk protein concentrate powders

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Received 10 January 2012; accepted for publication 28 May 2012; first published online 24 September 2012

The solubility of milk protein concentrate (MPC) powders was influenced by the method used for preparing the concentrate, drying conditions, and the type of dryer used. Increasing total solids of the ultrafiltered concentrates (23% total solids, TS) by diafiltration to 25% TS or evaporation to 31% TS decreased the solubility of MPC powders (80–83% protein, w/w dry basis), with ultrafiltration followed by evaporation to higher total solids having the greater detrimental effect on solubility. High shear treatment (homogenisation at 350/100 bar, microfluidisation at 800 bar or ultrasonication at 24 kHz, 600 watts) of ultrafiltered and diafiltered milk protein concentrates prior to spray drying increased the nitrogen solubility of MPC powders (82% protein, w/w dry basis). Of the treatments applied, microfluidisation was the most effective for increasing nitrogen solubility of MPC powders after manufacture and during storage. Manufacture of MPC powders (91% protein, w/w dry basis) prepared on two different pilot-scale dryers (single stage or two stage) from milk protein concentrates (20% TS) resulted in powders with different nitrogen solubility and an altered response to the effects of microfluidisation. Microfluidisation (400, 800 and 1200 bar) of the concentrate prior to drying resulted in increased long term solubility of MPC powders that were prepared on a single stage dryer but not those produced on a two stage spray dryer. This work demonstrates that microfluidisation can be used as a physical intervention for improving MPC powder solubility. Interactions between the method of preparation and treatment of concentrate prior to drying, the drying conditions and dryer type all influence MPC solubility characteristics.

Keywords: Milk protein concentrate, powder, shear, solubility, microfluidisation.

The process for manufacture of MPC powders involves the ultrafiltration and diafiltration, and optionally evaporation of the retentate prior to drying. MPC powders may be made as low or high heat products (Getler et al. 1997; Huffman & Harper, 1999). MPC powders have been used in cheese and a range of food applications including in meat, bakery and dairy products, high protein drinks and desserts (Zwijgers, 1992).

Commercial MPC powders (~ 80% protein) vary widely in their solubility and functionality (De Castro-Morel & Harper, 2002). The lack of consistency in the properties of MPC powders detract from their ability to be used as reliable raw ingredients in many applications. The loss of solubility during MPC powder production and upon storage is a limiting factor for its use. This is because good solubility is a prerequisite for many functional properties of protein powders. The loss of solubility of MPC powders during

storage has been attributed to the casein component (Anema et al. 2006) and the slow release of casein micelles from the powder matrix on rehydration (Mimouni et al. 2010a, b). The solubility of MPC powders after production and during storage may be improved by controlling processing conditions such as the temperature and extent of ultrafiltration, diafiltration and evaporation of the liquid milk and concentrate and the drying conditions (Novak, 1996; De Castro & Harper, 2001, 2003; Schuck, 2009). Processing interventions used to improve the cold water solubility of MPC powders have generally involved the addition of monovalent ions prior to drying (Carr, 2002), removal of calcium ions by using a cation exchanger (Bhaskar et al. 2003; Dybing et al. 2003), acidification to low pH followed by ultrafiltration/diafiltration or addition of a calcium chelating agent (Schuck et al. 2002; Bhaskar et al. 2003). Recently it was shown that physical treatment of the concentrate using static high pressure could also be used to improve the solubility of MPC powders (Udabage et al. 2012).

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Earlier studies have established that the properties of an evaporated milk concentrate, which are affected by pre-treatment of milk prior to concentration and extent of concentration, affect the properties of skim and whole milk powders (Baldwin et al. 1980; Bloore & Boag, 1981; Snoeren et al. 1982, 1983; Bienvenue et al. 2003). However, to date, systematic studies on the effects of physical processing steps during preparation of the milk concentrates on the solubility of MPC powders are lacking. High shear treatments which have the potential to alter the properties of casein micelles and the viscosity of milk concentrates may be expected to affect powder properties. It is well known that high shear causes thinning of milk concentrates (Snoeren et al. 1982; Trinh et al. 2007) and the viscosity of feed into the dryer affects powder properties (Schuck et al. 2005).

It was of interest to examine the use of high shear treatments applied prior to drying on MPC powder properties. The high shear treatments of interest to this study are homogenisation, microfiltration and ultrasonication. These shear treatments have generally been used to reduce the particle size of the fat globules. Homogenisation is a standard process used in dairy processing where the milk is pumped through a small orifice at high pressure. The combination of turbulence and cavitation experienced by the milk as a result of homogenisation causes reduced particle size of fat globules. Homogenisation may also alter proteins depending on the homogenisation pressure used. While only small changes in casein micelle are obtained with conventional homogenisation pressures used in the dairy industry (Walstra, 1980), use of higher homogenisation pressures (>100 MPa) causes micellar disintegration and reduces casein micelle size (Sandra & Dalgleish, 2005; Lodaite et al. 2009). In microfluidisation, fluids are guided into microchannels of specific configurations at high force to an impingement area. The particles in the flow stream are subject to shear, impact and cavitation forces which have the capacity to reduce the size of particles (Maa & Hsu, 1999). Microfluidisation has generally been used for the preparation of fine emulsions. However, the high shear and significant impact forces generated during microfluidisation also disintegrate and fragment casein micelles (Dalgleish et al. 1996). Ultrasound has applications across many industries and has been most used for the purpose of dispersing and homogenisation in the food industry (Leonelli & Mason, 2010). Ultrasound waves create cavitation bubbles which grow in size until they become unstable and collapse, generating chemical and mechanical effects which cause changes in materials. Changes in casein micelle size have been observed on ultrasonication of skim milk (Nguyen & Anema, 2010).

The objective of this work was to evaluate the influence of high shear treatments and drying conditions on MPC powder solubility. Initially, the solubility of MPC produced from milk protein retentates prepared by (1) ultrafiltration (UF) only, (2) ultrafiltration and diafiltration (UF/DF) or (3) ultrafiltration followed by evaporation (UF/Evaporation) was examined. The effects of the application of various shear treatments

(homogenisation, microfluidisation or ultrasonication) of UF/DF concentrates on MPC solubility were also assessed. To understand the interaction between concentrate treatment and drying conditions, the effects of drying variables and dryer type were evaluated for selected concentrates.

Materials and Methods

Production of MPC powders

Powders were manufactured on a pilot scale at the CSIRO Division of Food and Nutritional Sciences (Werribee, Victoria 3030, Australia) from (1) fresh skim milk, (2) milk reconstituted from a commercially prepared skim milk powder (Tatura Milk Industries, Tatura, Victoria, Australia), or (3) commercially produced concentrates (Murray Goulburn Co-Operative Co. Ltd, Leongatha, Victoria, Australia).

Effect of ultrafiltration, diafiltration and evaporation

Fresh skim milk was pasteurised at 72 °C for 15 s and processed through a Pasilac plate and frame ultrafiltration (UF) plant (Invensys APV, Clayton North, Victoria 3168, Australia) at 50 °C using a 10000 MW membrane for the production of 22.9% TS UF retentates from a ~ 5-fold concentration of the skim milk. A portion of this UF retentate was diafiltered to obtain a 24.9% TS concentrate. The remaining UF retentate (22.9% TS) was evaporated to 31.4% TS on a Bertuzzi APV scraped surface evaporator (Invensys APV, Clayton North, Victoria 3168, Australia). The concentrates were spray dried on a single stage Niro Production Minor (GEA Process Engineering Australia Pty. Ltd., Blackburn, Victoria 3130, Australia) fitted with a rotary atomiser using two drying conditions (inlet/outlet 175/75 °C or 190/90 °C) to produce MPC powders. Figure 1 gives an overview of the production processes used.

Effect of various shear treatments of concentrates

On a separate occasion, skim milk reconstituted from skim milk powder, was pasteurised at 72 °C for 15 s and then ultrafiltered (UF) and diafiltered (DF) through a Pasilac plate and frame ultrafiltration (UF) plant using a 10,000 MW membrane at 50 °C to obtain an ~ 22% TS concentrate for preparation of MPC powders (82–83% protein, w/w dry basis). Concentrates were sub-sampled. A portion was used directly for spray drying for the control MPC powder. The remaining concentrate was subjected to the desired shear treatment. The interventions used were homogenisation (350/100 bar) of the concentrate through a Rannie 3060 homogeniser (Invensys APV, Clayton North, Victoria 3168, Australia), microfluidisation (800 bar) using a M-210EH-B pilot microfluidiser (Microfluidics, Newton, MA 02464, USA), or ultrasonication (24 kHz, 160 ml/min @ 600 watts) using a Hielscher UP400S ultrasonic generator (Hielscher Ultrasonics GmbH, Teltow, Germany). The concentrates

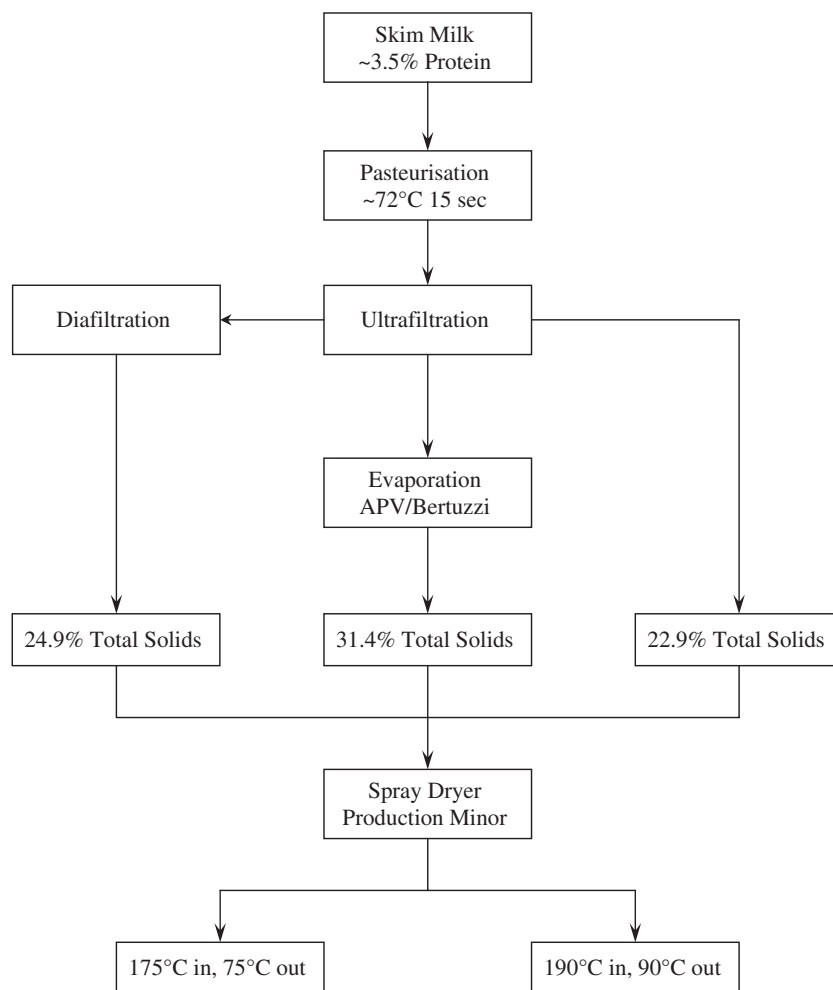


Fig. 1. MPC powder produced using different concentration processes and drying conditions. Fresh skim milk was used for production of MPC powder with 80–83% w/w protein, dry basis; in=inlet temperature and out=outlet temperature of spray dryer.

were then spray dried on a single stage dryer (Drytec Ltd, Kent, TN9 IRA, England) using an inlet and outlet temperature of 175 °C inlet and 75 °C respectively. The experimental scheme used is depicted in Fig. 2. The MPC powders were analysed after manufacture. Samples were packed in barrier bags and kept in a dry goods store (~ 22 °C) and removed at intervals during storage for analysis.

Effects of microfluidisation pressure and dryer type

On a separate occasion, commercially-produced concentrates (20% TS concentrate) were obtained on three consecutive days for preparation of MPC powders (91% protein, w/w dry basis). These concentrates were dried directly, or microfluidised (400, 800 or 1200 bar) then spray dried using the single stage dryer (Drytec), as described above. Portions of commercial concentrates (20% TS concentrate) were also used dried directly or microfluidised (800 bar) then spray dried using a two stage dryer (FSD 4 spray dryer, NIRO Australia Pty. Ltd., Blackburn, VIC 3130).

The temperature conditions of the FSD dryer were as follows: Inlet/outlet 170 °C/70 °C; static fluid bed 70 °C; powder 60 °C. The other parameters were: Feed pressure 50 bar; HP nozzle 77/21, cyclone 100 to 110 mm H₂O, drying chamber – 15 to – 10 mm H₂O, SFB Airflow 110 to 120 mm H₂O and pump speed 14–16%. Figure 3 shows the conditions used for production of MPC powders from commercial concentrates. The MPC powders were analysed after manufacture. Samples were packed in barrier bags and kept in a dry goods store (~ 22 °C) and removed at intervals during storage for analysis.

Analyses of powders

Gross composition analyses. The moisture content was calculated as weight loss after drying of concentrates or powders at 102 °C in a Contherm Digital Series Five laboratory oven. The total nitrogen content of powders was determined in a LECO FP-2000 analyser according to

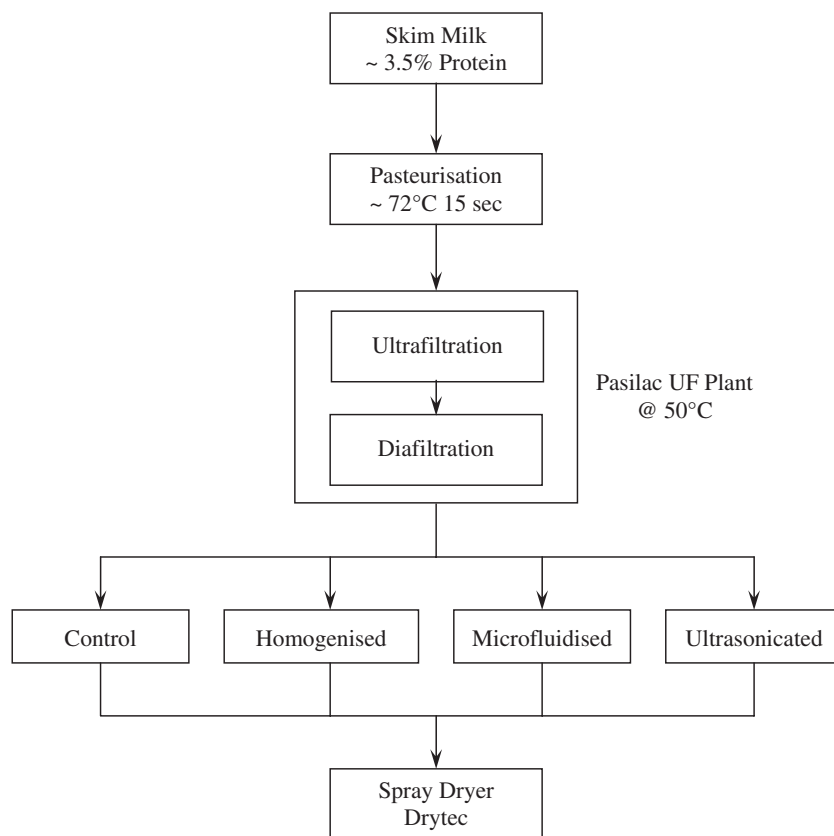


Fig. 2. MPC powder production. Skim milk reconstituted from skim milk powder was used. Ultrafiltration and diafiltration was carried out to obtain 22% TS concentrate for production of MPC powder (82% w/w protein, dry basis).

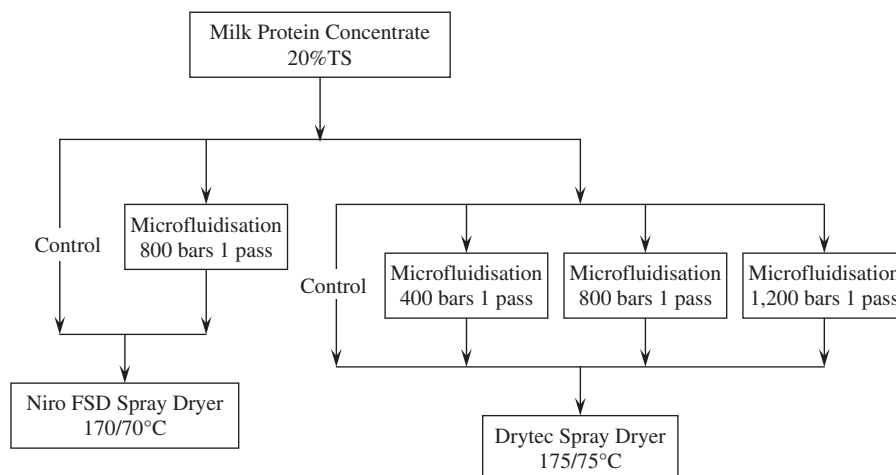


Fig. 3. MPC powder production from commercial concentrate. Concentrates at 20% TS intended for manufacture of MPC powder (91% w/w protein, dry basis) was obtained from a commercial producer to evaluate the effects of microfluidisation using two different spray dryers.

International Dairy Federation method 185 (IDF, 2000) using EDTA as a standard.

Nitrogen solubility and insolubility index. The nitrogen solubility was determined using IDF method 173 (IDF, 1995).

Briefly, a mass of powder equivalent to 1 g of protein (total nitrogen $\times 6.38$) was dispersed in water at room temperature ($\sim 22^\circ\text{C}$) for two hours and the pH adjusted to 7.0. The solutions were made up to 100 ml. Fifty millilitres of each sample was transferred to tubes and centrifuged for 10 min at 3000 g (Beckman J-6 centrifuge, JA 4.2 rotor). The

Table 1. Composition of milk protein concentrates and milk protein concentrate powders made from fresh skim milk

Sample†	% Protein in Powder (g/100 g)	% Protein in solids	Insolubility index (ml/50 ml)	Nitrogen solubility (%)
Powders (80% protein, dry basis) from UF (only) concentrates				
SD175/75 (UF)	74.1 ± 0.23	80.43 ± 0.25	4.9 ± 0.1	91.3 ± 0.47
SD190/90 (UF)	75.1 ± 0.01	79.45 ± 0.02	5.7 ± 0.3	70.1 ± 1.00
Powders (83% protein, dry basis) from UF/DF concentrates				
SD175/75 (UF/DF)	76.8 ± 0.07	82.15 ± 0.07	11.0 ± 0.1	83.9 ± 2.23
SD190/90 (UF/DF)	78.2 ± 0.08	83.38 ± 0.09	12.0 ± 0.1	55.0 ± 1.28
Powders (80.5% protein, dry basis) from UF/Evaporated concentrates				
SD175/75 (UF/Evap)	76.3 ± 0.15	81.29 ± 0.16	13.3 ± 0.3	71.0 ± 0.21
SD190/90 (UF/Evap)	77.1 ± 0.11	80.91 ± 0.11	20.5 ± 0.5	49.3 ± 0.28

†SD 175/75 indicates that concentrates were spray dried at 175 °C (Inlet temperature) and 75 °C (Outlet temperature); SD 190/90 indicates that concentrates were spray dried at 190 °C (Inlet temperature) and 90 °C (Outlet temperature). The suffix represents the method of preparation of the concentrate. The moisture content of all powders was 5–6% except SD175/75 which had 8% moisture; Data are the mean of duplicate analysis ± SE

supernatant was decanted and filtered through a Whatman No 1 filter paper. The nitrogen content of the whole solution and filtered supernatant were determined in a LECO FP-2000 Nitrogen analyser. The insolubility index of powders was determined following IDF method 129A (IDF, 1988). Briefly, samples of powders (10 g) was dispersed in water (100ml) at room temperature (~ 22 °C), mixed for 90 s and then transferred to graduated conical tubes and centrifuged for 10 min at 160 g (Beckman J-6 centrifuge, JA 4.2 rotor). Supernatants were removed by suction, the sample diluted with water, and re-centrifuged and the volume of insoluble material measured.

Particle sizing of milk powders. The measurement of particle size was made using a Malvern Mastersizer 2000 laser diffraction system. A sample (~ 4.0 g) of each powder was dispersed in 20 ml of isobutanol. A sub-sample of this dispersion was added dropwise to circulating isobutanol in the Mastersizer 2000. The measurement was made using refractive indices of 1.533 for the particle and 1.394 for the dispersant (isobutanol). The general-purpose model for irregular particles was used to analyse the data.

Powder morphology using scanning electron microscopy (SEM). The samples were placed on an aluminium sample holder using double sided adhesive carbon tape. They were then coated with 4 nm of chromium using a Dynavac Xenosput magnetron coater. The samples were imaged using a Hitachi S4100 Cold Field Emission Scanning Electron Microscope with an accelerating voltage of 1 kV and a working distance of 5 mm.

Results

Effect of ultrafiltration, diafiltration and evaporation of skim milk at pilot-scale

Powders (80–83% protein, w/w dry basis) were made from (1) ultrafiltered concentrates (23% TS, 18.4% protein),

(2) UF/DF concentrates (25% TS, 20.9% protein) and (3) UF/evaporated concentrates (31% TS, 25.4% protein). The insolubility index of MPC powders increased while the nitrogen solubility decreased as the total solids and protein content of the ultrafiltered concentrates were increased by diafiltration or evaporation (Table 1). A comparison of the insolubility index and nitrogen solubility for MPC powders made from UF retentate that had been concentrated by evaporation (UF/Evap) and those made from diafiltered UF retentate (UF/DF) suggests that concentration by evaporation was more detrimental to the insolubility of the MPC powders, despite the higher protein content of the powder made from UF/DF concentrate (Table 1). Higher drying temperatures (190/90 °C compared with 175/75 °C) reduced the solubility of MPC powders from the same concentrate (Table 1). A separate spray drying run confirmed that MPC powders (76% protein, w/w dry basis) made from the same UF retentate had a higher insolubility index and lower nitrogen solubility when dried at 190/90 °C compared with 175/75 °C (inlet temperature / outlet temperatures) (data not shown).

Effects of different shear treatments of concentrates produced on a pilot-scale

The solubility of MPC powders (~ 82% protein w/w, dry basis) after manufacture was improved when concentrates were subjected to high shear treatments prior to drying (Table 2). Of the shear treatments applied, the greatest enhancement in nitrogen solubility was obtained with microfluidisation compared with either ultrasonication or homogenisation. The insolubility index of powders prepared from microfluidised concentrates was the lowest. Nitrogen solubility data over storage showed that MPC powders made from microfluidised concentrates were more soluble than control powders and powders made from homogenised or ultrasonicated concentrates (Table 2). Overall the data demonstrated that microfluidisation was the most effective of the shear treatments examined for improving MPC solubility on production as well as during storage.

Table 2. Properties of MPC powders made from concentrates (~ 22% TS) that were homogenised (350/100 bar), microfluidised (800 bar) and ultrasonicated (24 kHz, 160 ml/min @ 600 watts) prior to spray drying†

Treatment	% moisture	Protein % in powder	Protein % in solids	Insolubility index (ml/50 ml)	Nitrogen solubility % (Initial)	Nitrogen solubility % (After 8 months)
None	5.60±0.29	78.28±0.16	83.02±0.31	10.1±0.3	70.14±0.12	51.08±0.01
Homogenisation	6.32±0.59	77.75±0.31	82.95±0.62	9.5±0.5	74.46±0.10	58.69±0.01
Microfluidisation	5.45±0.15	77.27±0.57	81.77±0.61	6.5±0.6	89.52±0.24	68.71±0.03
Ultrasonication	5.10±0.19	78.14±0.10	82.33±0.19	9.6±1.9	74.69±0.14	55.11±0.04

†MPC powders made from reconstituted skim milk. Standard error calculated from duplicate analysis of two replicated trials conducted one month apart

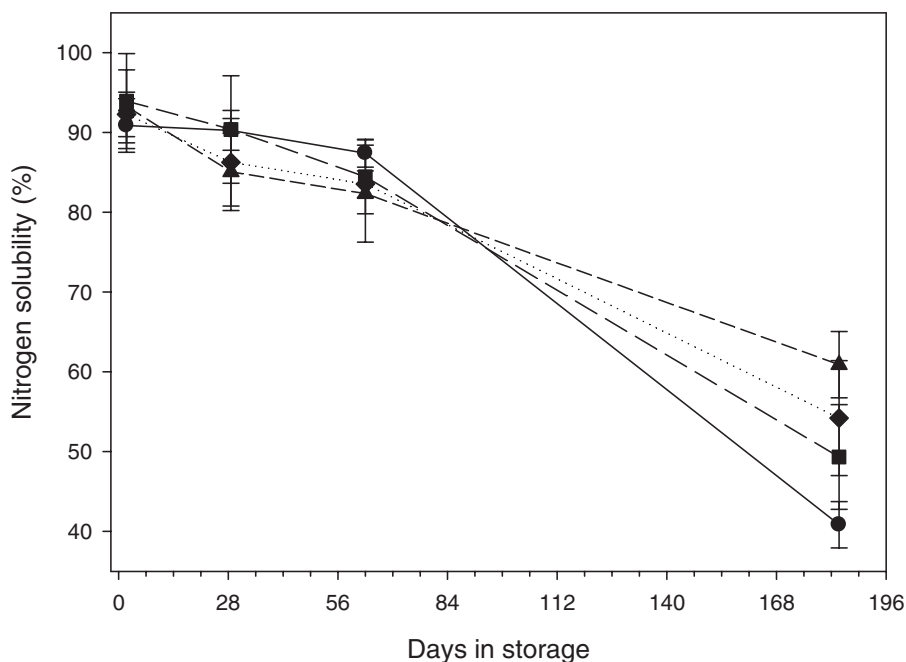


Fig. 4. Comparison of nitrogen solubility of MPC powders (91% w/w, dry basis) during storage. Commercial concentrates were either dried directly (control ●) or microfluidised (400 ■, 800 ◆ and 1200 bar ▲) prior to drying. Data indicate average±SD from three independent powder processing runs.

MPC powders made from commercial concentrates

Effect of microfluidisation pressure. Figure 4 shows the effects of different microfluidisation pressures (400–1200 bar) on nitrogen solubility over time. Immediately after manufacture and over 2 months storage, there was little difference in solubility between MPC powders made from microfluidised and control (non-microfluidised) concentrates. It should be noted that the transportation of the commercially produced concentrates from the commercial plant to the pilot-plant at CSIRO led to an unavoidable delay of approximately four hours between production of the concentrate and drying. This delay could have led to thickening of the concentrates that could not be completely reversed by high shear, and which could impact on the nitrogen solubility data.

At six months storage, all samples which had been microfluidised prior to drying showed higher solubility than their respective controls. Increasing the pressure used for microfluidisation of concentrates prior to drying resulted

in improved nitrogen solubility of MPC powders at six months storage.

Effect of dryer type. Figure 5 shows marked differences in solubility between the powders produced on the FSD and Drytec dryers made from commercial concentrates which had not been microfluidised. MPC powders made on the Drytec dryer had consistently higher solubility over storage. Although the solubility of MPC powders on long term storage (6 months) that were produced by the Drytec dryer was improved when the concentrate was microfluidised (800 bar) prior to drying (Table 3, Fig. 1), microfluidisation of the concentrate prior to drying on the FSD dryer did not influence MPC solubility (Table 3). This suggests an interaction between the shear treatment of concentrates and type of dryer used.

Particle size analysis showed that powders produced on the Drytec dryer had a volume weighted average particle size (D[4,3]) of 48 µm while those produced on the FSD dryer

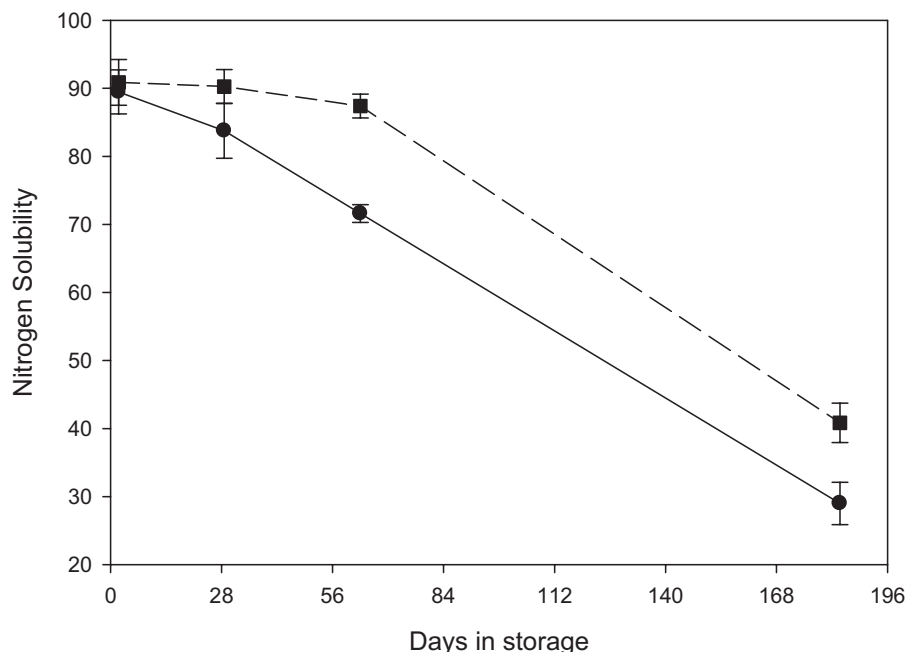


Fig. 5. Comparison of nitrogen solubility of MPC powders (91% protein, w/w dry basis), produced on two different dryers, during storage. Two pilot scale spray dryers [Single stage (Drytec) ■; Two stage (FSD) ●] were used to dry commercially produced concentrates. Data indicate average \pm SD from three independent powder processing runs.

had an average size of 277 μm . Microfluidisation of the concentrates before drying reduced the average size to 45 μm for powders produced on the Drytec and 171 μm for powders produced on the FSD dryer. These trends were generally confirmed by the electron micrographs of powders (Fig. 6).

Discussion

The removal of water from proteins during concentration and drying places a stress on the proteins. There is a change in solution properties as the water activity is reduced on concentration and drying and this can result in destabilisation of proteins, resulting in a change in the protein structure and functionality of the protein powder that is not fully reversible on the addition of water (Kitabatake et al. 1989; Gaiani et al. 2010). Both concentration and shear treatments affect the physical properties of concentrates. In addition high shear treatments, such as microfluidisation and ultrasonication, can alter the structure of the milk proteins (Dagleish et al. 1996; Nguyen & Anema, 2010). A change in solubility for spray dried powders made from differently prepared concentrates may be due to differences in the viscosity of the concentrates fed to the dryer and/or alterations to casein micelles caused by the different concentration, shear treatments, drying regimes and type of dryer used in MPC powder manufacture.

Effects of concentration methods

The increased concentration of mineral salts, higher %TS and hence probable higher viscosity of evaporated retentate

(UF/Evap, 31% TS) compared to that of the diafiltered retentate (UF/DF, 25% TS) possibly contributed to the higher insolubility of powders prepared from evaporated retentates (Table 1). When membrane processes (ultrafiltration, diafiltration) are applied in the production of the milk protein concentrate, there is an increase in protein content, altered mineral-protein equilibria composition and changes in concentration of lactose and low molecular weight components (Thompson & deMan 1975; Getler et al. 1997). The extents of these changes depend on the method of concentration and therefore the interactions between protein species in the concentrate will depend on the method used for preparation of the concentrate. Diafiltration concentrates proteins while reducing low molecular weight components (i.e. lactose, mineral salts etc.) in milk (Getler et al. 1997). This processing step increases the total solids and protein content of the concentrate without the application of heat. An increase in protein concentration as the protein:solids ratio is increased increases the interactions between the proteins, predisposing the protein to aggregate formation. Sugars have a protective effect on proteins during drying (Crowe et al. 1998), the increased interaction between proteins and the reduced lactose content will decrease the quality of MPC powders made from UF/DF concentrates obtained by diafiltration of UF retentates. A similar effect of increased protein interaction would be obtained when an ultrafiltered concentrate is evaporated. In this case there is only the removal of water. Increasing the total solids increases the viscosity of concentrate, leading to less efficient drying and reduced solubility of milk powders (Baldwin et al. 1980;

Table 3. Nitrogen solubility of MPC powders (91% protein in solids, ~ 5% moisture) made from commercial concentrates that were dried directly or microfluidised (800 bar) prior to spray drying on different dryers†

Storage time	Drytec dryer		FSD dryer	
	Nitrogen solubility of MPC powder (%)			
	Control	Microfluidised	Control	Microfluidised
Initial	90.77 ± 1.95	92.27 ± 1.6	89.47 ± 1.87	90.2 ± 1.5
1 month	90.27 ± 1.44	86.17 ± 3.27	83.77 ± 2.34	84.07 ± 1.3
2 month	87.43 ± 1.01	83.53 ± 0.95	71.60 ± 0.75	71.6 ± 1.66
6 month	40.83 ± 1.68	54.20 ± 4.16	29.00 ± 1.79	33.23 ± 2.22

† Standard error calculated from analysis of three trials

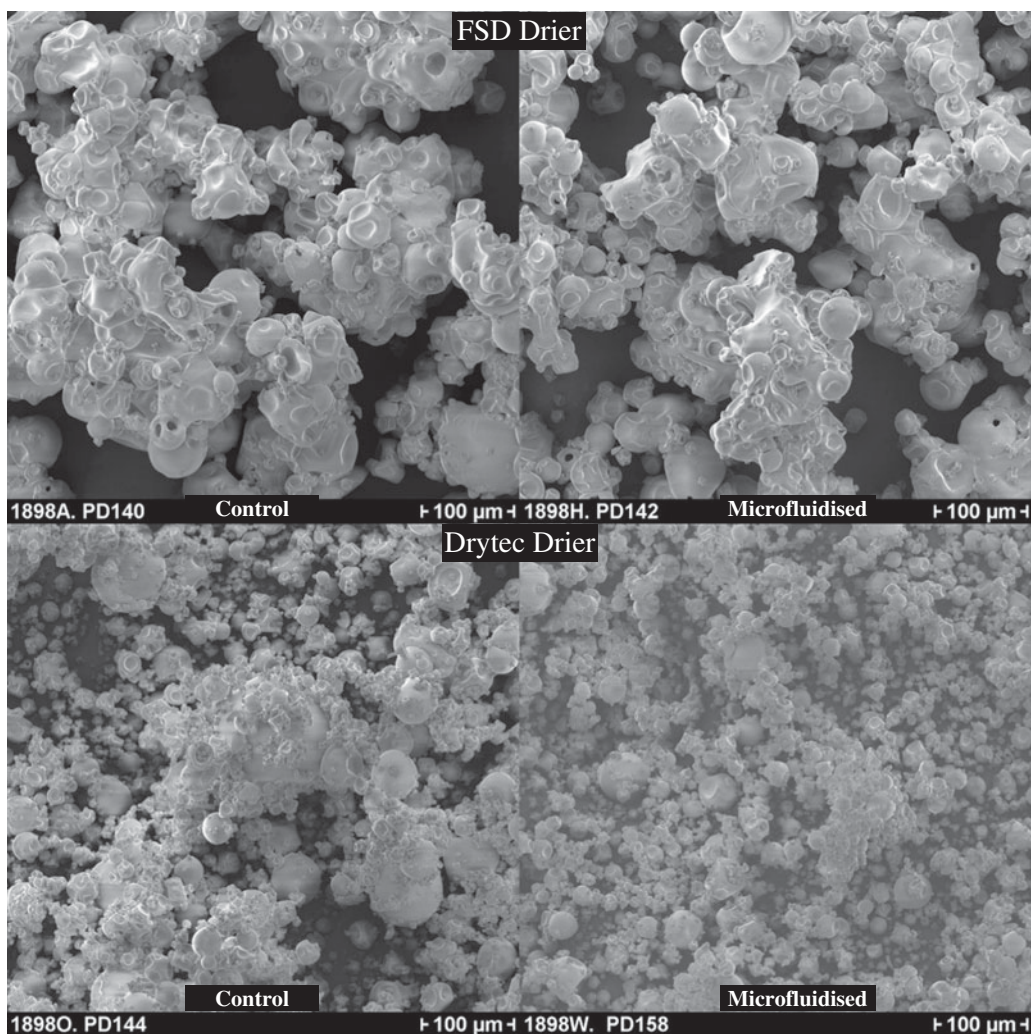


Fig. 6. Electron micrographs of MPC powders produced on different dryers [Single stage (Drytec); Two stage (FSD)] from non-microfluidised (Control) or microfluidised (800 bar) concentrates.

Bloore & Boag, 1982). The increase in the viscosity of the concentrate as protein:total solids ratio is increased in evaporated retentates leads to poor atomisation during drying, resulting in a powder with inferior solubility and hydration characteristics (Schuck, 2009). It is not possible to

discount the effects on MPC powder quality due to an alteration of the composition of the minerals and the distribution of the proteins between the micellar and serum phases of the milk concentrate prepared by UF/DF and UF followed by evaporation. Others have found that removal of

calcium ions improves the solubility of MPC powders (Bhaskar et al. 2003).

Effect of shear treatments

Absolute differences in nitrogen solubility between the concentrates produced on the pilot-scale and commercial scale may be related to an interplay of many factors including the initial composition and that of the concentrates and conditions of membrane processing. The improvement in MPC powder solubility over long term storage of concentrates subject to shear treatment suggests that physical interventions have the potential to be applied in place of previous strategies based on the use of additives (Bhaskar et al. 2003; Dybing et al. 2003; Schuck et al. 2002). High shear treatments such as ultrasound are known to decrease the viscosity of concentrates (Zisu et al. 2010) and the shear-thinning of concentrates given shear treatments prior to drying probably contributes to the improved nitrogen solubility of MPC powders. Shear treatments such as microfluidisation or ultrasonication cause disintegration of the protein components and alteration of protein structure (Dalglish et al. 1996; Nguyen & Anema, 2010) and the beneficial effects of shear on nitrogen solubility are likely to be a combination of physical changes as well as changes to protein species and structure. This will be particularly so where fragments of surface-active protein are formed due to disintegration of protein components. An altered distribution of surface active proteins in the concentrate will result in an altered powder surface composition, and therefore differences in solubility. Studies have shown that there is competitive absorption of proteins during spray drying and that the surface composition of the spray dried powder will be dependent on the protein species in the solution and their ability to attach and re-arrange at the air-water interface during drying (Landström et al. 2003).

Effects of drying conditions

Drying at higher inlet/outlet temperatures caused a loss in the quality characteristics of MPC powders (Table 1). Others found that when the outlet temperature of a spray dryer was increased from 75 to 90 °C at a constant inlet temperature of 200 °C, there were variable effects on solubility, moisture, hydration and bulk density of MPC powders with 70% protein (De Castro & Harper, 2001). Keeping the outlet temperature constant (90 °C) and increasing inlet temperatures from 200 to 300 °C increased powder particle size and decreased the ease of hydration (De Castro-Morel & Harper, 2003). Changes in powder quality may be related to altered composition of the powder surface. Recent studies have demonstrated that drying temperatures influence the distribution of proteins, lipids and lactose on the surface of high protein dairy powders (Gaiani et al. 2010). These authors also suggested that by drying at lower outlet temperatures, surface active components have the more time to migrate to the interface in contrast to when higher temperatures are

used, resulting in an altered powder surface composition (Gaiani et al. 2011), and hence altered rehydration properties. Similarly, differences in MPC solubility of powders resulting from the use of different dryers (single stage Drytec and two stage FSD) are related to the differences in the powder structure obtained as a result of differences in atomisation, temperature profiles in dryers during the drying and drying kinetics.

The physical properties of the concentrate, changes in protein composition and protein-mineral equilibria induced by processes used for concentrate manufacture, the drying conditions and dryer type, as well as the interaction between these factors have an influence on the solubility of MPC powders. Further research is required to understand how these macroscopic properties are related to events that take place on a molecular and microstructural level under different conditions of processing, especially how the partitioning of proteins, protein-mineral equilibria and surface-active components inherent in the milk influence the assembly of components at the air-water interface during drying.

This work was supported in part by the Dairy Ingredients Group of Australia (now part of Dairy Innovation Australia Ltd).

References

- Anema SG, Pinder DN, Hunter RJ & Hemar Y 2006 Effects of storage temperature on the solubility of milk protein concentrate (MPC85). *Food Hydrocolloids* **20** 386–393
- Baldwin AJ, Baucke AG & Sanderson WB 1980 The effect of concentrate viscosity on the properties of spray dried skim milk powder. *New Zealand Journal of Dairy Science and Technology* **15** 289–297
- Bhaskar GV, Singh H & Blazey ND 2003 Milk protein products and processes. US Patent US 2003/0096036
- Bienvenue A, Jiménez-Flores R & Singh H 2003 Rheological properties of concentrated skim milk: influence of heat treatment and genetic variants on the changes in viscosity during storage. *Journal of Agricultural and Food Chemistry* **51** 6488–6494
- Bloore CG & Boag IF 1981 Some factors affecting the viscosity of concentrated skim milk. *New Zealand Journal of Dairy Science and Technology* **16** 143–154
- Bloore CG & Boag IF 1982 The effect of processing variable on spray-dried milk powder. *New Zealand Journal of Dairy Science and Technology* **17** 103–120
- Carr A 2002 Monovalent salt enhances solubility of milk protein concentrate. PCT Patent WO 02/096208
- Crowe JH, Carpenter JF & Crowe LM 1998 The role of vitrification in anhydrobiosis. *Annual Review of Physiology* **60** 37–103
- Dalglish DG, Tosh SM & West S 1996 Beyond homogenization: the formation of very small emulsion droplets during the processing of milk by a microfluidizer. *Netherlands Milk and Dairy Journal* **50** 135–148
- De Castro M & Harper WJ 2001 Effect of drying on characteristics of 70% milk protein concentrate. *Milchwissenschaft* **56** 269–272
- De Castro-Morel M & Harper WJ 2002 Basic functionality of commercial milk protein concentrates. *Milchwissenschaft* **57** 367–370
- De Castro-Morel M & Harper WJ 2003 Effect of retentate heat treatment and spray dryer inlet temperature on the properties of milk protein concentrates (MPC's). *Milchwissenschaft* **58** 13–15
- Dybing ST, Bhaskar GV, Dunlop FP, Fayerman AM & Whitton MJ 2003 Modified milk protein concentrates and their use in making gels and dairy products. US Patent US2003/0054068

- Gaiani C, Morand M, Sanchez C, Arab Tehrani E, Jacquot M, Schuck P, Jeantet R & Scher J** 2010 How surface composition of high milk protein powders is influenced by spray-drying temperature. *Colloids and Surfaces B: Biointerfaces* **75** 377–384
- Gaiani C, Mullet M, Arab-Tehrany E, Jacquot M, Perroud C, Renard A & Scher J** 2011 Milk proteins differentiation and competitive adsorption during spray-drying. *Food Hydrocolloids* **25** 983–990
- Getler J, Nielsen A & Sprogø J** 1997 Functional process for MPC. *Dairy Industries International* **62**(3) 25, 27
- Huffman LM & Harper WJ** 1999 Maximizing the value of milk through separation technologies. *Journal of Dairy Science* **82** 2238–2244
- IDF** 1988 Dried milk protein products: determination of insolubility index. *IDF Standard* 129A. Brussels: International Dairy Federation
- IDF** 1995 Dried milk protein products: determination of nitrogen solubility index. *IDF Standard (provisional)* 173. Brussels: International Dairy Federation
- IDF** 2000 Milk and milk products: determination of nitrogen content: routine method by combustion according to Dumas principle. *IDF Standard (provisional)* 185. Brussels: International Dairy Federation
- Kitabatake N, Indo K & Doi E** 1989 Changes in interfacial properties of hen egg ovalbumin caused by freeze-drying and spray-drying. *Journal of Agricultural and Food Chemistry* **37** 905–910
- Landström K, Arnebrant T & Bergenståhl B** 2003 Competitive protein adsorption between beta-casein and beta-lactoglobulin during spray-drying: effect of calcium induced association. *Food Hydrocolloids* **17** 103–116
- Leonelli C & Mason TJ** 2010 Microwave and ultrasonic processing: now a realistic option for industry. *Chemical Engineering and Processing* **2010** 885–900
- Lodaite K, Chevalier F, Armaforte E & Kelly AL** 2009 Effect of high-pressure homogenisation on rheological properties of rennet-induced skim milk and standardised milk gels. *Journal of Dairy Research* **76** 294–300
- Maa Y-F & Hsu CC** 1999 Performance of sonication and microfluidisation for liquid–liquid emulsification. *Pharmaceutical Development and Technology* **4** 233–240
- Mimouni A, Deeth HC, Whittaker AK, Gidley MJ & Bhandari BR** 2010a Rehydration of high-protein-containing dairy powder: slow- and fast-dissolving components and storage effects. *Dairy Science and Technology* **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. *Journal of Dairy Science* **93** 463–472
- Nguyen NHA & Anema SG** 2010 Effect of ultrasonication on the properties of skim milk used in the formation of acid gels. *Innovative Food Science and Emerging Technologies* **11** 616–622
- Novak Ā** 1996 Application of membrane filtration in the production of milk protein concentrates. *Bulletin of the International Dairy Federation* **311** 26–27
- Sandra S & Dalgleish DG** 2005 Effects of ultra-high-pressure homogenization and heating on structural properties of casein micelles in reconstituted skim milk powder. *International Dairy Journal* **15** 1095–1104
- Schuck P** 2009 Understanding the factors effectively the factors affecting spray-dried dairy powder properties and behaviour. In *Dairy-derived Ingredients, Food and Nutraceutical Uses*, pp. 24–50. (Ed. M Corredig), Cambridge, UK: Woodhead Publishing Limited and CRC Press LCC
- Schuck P, Davanel 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. *International Dairy Journal* **12** 51–57
- Schuck P, Méjean S, Dolivet A, Beaucher E & Famelart MH** 2005 Pump amperage: a new method for monitoring viscosity of dairy concentrates before spray drying. *Lait* **85** 361–367
- Snoeren THM, Damman AJ & Klok HJ** 1982 The viscosity of skim-milk concentrates. *Netherlands Milk Dairy Journal* **36** 305–316
- Snoeren THM, Damman AJ & Klok HJ** 1983 The viscosity of whole milk concentrate and its effect on the properties of dried whole milk. *Voedingsmiddelentechnologie* **16** 68–71
- Trinh B, Haisman D & Trinh KT** 2007 Rheological characterisation of age thickening with special reference to milk concentrates. *Journal of Dairy Research* **74** 106–115
- Thompson SJ & deMan JM** 1975 Concentration and fractionation of milk by ultrafiltration. *Canadian Institute of Food Science and Technology Journal* **8** 113–116
- 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. *Journal of Dairy Research* **79** 76–83
- Walstra P** 1980 The effects of homogenization on milk plasma. *Netherlands Milk and Dairy Journal* **9** 189–192
- Zisu B, Bhaskaracharya R, Kentish S & Ashokkumar M** 2010 Ultrasonic processing of dairy systems in large scale reactors. *Ultrasonics Sonochemistry* **17** 1075–1081
- Zwijgers A** 1992 Outline of milk protein concentrate. *International Food Ingredients* **3** 18–23.