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Review

Strategies to enhance the rehydration performance of micellar casein-dominant dairy powders

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ABSTRACT

Due to their excellent nutritional (e.g., high calcium) and functional (e.g., heat stability and gelation) properties, the use of protein-enriched, micellar casein-dominant dairy powders, including milk protein concentrate/isolate and micellar casein concentrate, has increased considerably among food and beverage manufacturers. However, the poor and often inconsistent rehydration properties of these powders in water, specifically their low dispersibility and solubility (attributed to protein–protein interactions related to the high proportion of micellar casein), remains a significant challenge. This review provides a detailed analysis of the main physical (e.g., injection of gas, ultrasonication) and chemical (e.g., ion exchange, pH adjustment) processing strategies that have been applied, at both laboratory and pilot-scale, to enhance the rehydration performance of high-protein, micellar casein-dominant dairy powders. The information provided will support the advancement of dairy ingredient research and the technological development of nutritional powders that can be used across several industrial applications.

Contents

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1. Introduction

Milk protein concentrate (MPC), milk protein isolate (MPI), micellar casein concentrate (MCC) and sodium caseinate (NaCas) are some of the many casein-dominant powders currently available from the dairy industry. Two of the largest global producers of casein ingredients are the New Zealand and Irish dairy industries, producing 57,000 and 55,000 tonnes in 2019, respectively (Bord Bia, 2020). MPC and MPI powders are produced by ultrafiltration (UF) and diafiltration (DF) of skim milk, followed by evaporation and spray drying, while microfiltration (MF) is used in the production of MCC, by partially removing whey proteins. The final products normally contain at least 80% protein (w/w) and are extensively depleted in lactose and mineral salts. Applications of such micellar casein-dominant powders include medical nutritional beverages for individuals with disease-related malnutrition, performance nutrition bars for athletes, follow-on infant formulas, as well as cheese, yoghurt and ice cream (Agarwal, Beausire, Patel, & Patel, 2015).

For many applications, rehydration of a powder in water or an aqueous medium is required for complete expression of protein functionality (Fang, Selomulya, Ainsworth, Palmer, & Chen, 2011); therefore, achieving efficient dissolution of high-protein powders is normally essential for ingredient users (Freudig, Hogekamp, & Schubert, 1999). For example, Karam, Gaiani, Hosri, Hussain, and Scher (2016) reported that the rehydration state of micellar casein (MC) powder influenced the textural and rheological properties of acid milk gels, whereby graininess decreased and gel firmness increased as the MC ingredient became more soluble with rehydration time. Furthermore, for the consumer, complete rehydration of powdered ingredients is a key quality indicator.

Rehydration of micellar casein-dominant powder is a complex process influenced by several factors (e.g., powder composition, powder density and structure, solvent composition and temperature) but generally constitutes five stages: (i) wetting, (ii) sinking, (iii) swelling, (iv) dispersion and (v) solubility or dissolution, as described by Crowley, Kelly, Schuck, Jeantet, and O'Mahony (2016). The most commonly reported techniques in the literature to characterise these stages of rehydration include, but are not limited to, wetting behaviour using contact angle (Crowley et al., 2015, 2018), capillary rise and immersional wetting (Ji, Cronin, Fitzpatrick, Fenelon, & Miao, 2015; Selomulya & Fang, 2013); dispersion by particle size analysis (static light scattering) following stirring (Gaiani, Banon, Scher, Schuck, & Hardy, 2005; Jeantet, Schuck, Six, Andre, & Delaplace, 2010) and solubility by determining changes in total solids or protein content of a powder dispersion before and after centrifugation (Bansal, Truong, & Bhandari, 2017; Eshpari, Tong, & Corredig, 2014). However, it is evident that substantial variation exists with respect to the experimental parameters used for many of these techniques (e.g., for solubility determination, there are differences in the concentration of the dispersions, temperature of powder reconstitution and centrifugation conditions), which can make the comparison of results challenging. Furthermore, the authors are aware that in industrial settings, a glass slide is often used as an indicator of rehydration state by submerging it in a reconstituted product to observe the presence of insoluble material or flecks. Although this is a rapid method, it is highly subjective, and further demonstrates the uncertainty and discrepancy in how the rehydration properties of high-protein dairy powders are assessed. Furthermore, off-line techniques such as particle size analysis may not always be available to dairy processors with limited resources.

Previous reviews by Crowley et al. (2016) and Felix da Silva, Ahrné, Ipsen, and Hougaard (2018) have mainly focused on the manufacture, characteristics and stages involved in the rehydration of high-protein dairy powders, as well as advanced analytical techniques (e.g., nuclear magnetic resonance relaxometry) used for monitoring rehydration. However, the objective of this review is to specifically provide an overview of the main processing and formulation strategies that have been investigated to modify the rehydration properties of high-protein, micellar casein-dominant dairy powders.

2. Scientific basis for poor and inconsistent rehydration properties

Research investigating why high-protein, micellar casein-dominant dairy powders express poor rehydration performance, both after spray drying and during storage, has presented several mechanisms responsible for the development of insolubility (Fig. 1). Anema, Pinder, Hunter, and Hemar (2006) suggested that a network of casein micelles at the powder particle surface, formed by non-covalent bonding (e.g., hydrophobic interactions and/or hydrogen bonds), was responsible for the low solubility of MPC, with increasing storage time and temperature accelerating this deterioration in solubility. The low lactose content of MPC also facilitates protein–protein interactions as lactose would provide spatial separation of the casein micelles. This was supported by Havea (2006) who reported that the constituents of the insoluble material in MPC were linked together by non-covalent interactions.

Le, Bhandari, and Deeth (2011) reported a correlation between the development of Maillard reaction products during MPC powder storage and a decrease in solubility. A subsequent study by Le, Holland, Bhandari, Alewood, and Deeth (2013) identified α_{S1}-casein as the predominant component of the insoluble fraction in MPC following storage and reported that methylglyoxal, formed in the advanced stages of the Maillard reaction, was capable of inducing non-disulphide, covalent cross-linking of the proteins. However, Nasser et al. (2018) reported that lactose, expected to be a key reactant in the Maillard reaction, did not play a significant role in the loss of solubility of MPC powder during storage. Indeed, Nasser et al. (2017) established a relationship between loss of α-helix protein structure and a decrease in solubility of MC powder during storage. Mimouni, Deeth, Whittaker, Gidley, and Bhandari (2009) reported that structural collapse and fragmentation of MPC powder particles during rehydration was restricted by the presence of a network of micellar casein at the surface of powder particles. Mimouni, Deeth, Whittaker, Gidley, and Bhandari (2010a) suggested that the loss of solubility of MPC powder during storage was caused by altered rehydration kinetics (i.e., impaired dispersion), due to the persistence of a closely-packed skin of casein micelles at the powder particle surface, while a study by Mimouni, Deeth, Whittaker, Gidley, and Bhandari (2010b) demonstrated that rehydration of MPC was characterised by distinct populations of slow (casein and colloidal mineral) and fast (whey protein and lactose) dissolving components, and that incomplete dispersion was not directly due to the formation of insoluble material during storage or reduced water penetration. Research by Schuck et al. (1998, 2002), has suggested that the high micellar casein content of native phosphocaseinate (NPC) reduces the transfer of water and subsequent rehydration of powder particles. Finally, despite high-protein dairy powders containing a low quantity of fat, this component is often over-represented at the surface of spray dried powder particles and Gaiani et al. (2009) reported that lipids also migrated from the bulk to the surface of NPC powder particles during storage, thereby increasing wetting times.

Several physical and chemical processing strategies have been investigated in an effort to resolve the aforementioned challenges. An overview of these approaches are given in Tables 1 and 2, while a schematic representation of the stages in the manufacturing process where some of these strategies may be implemented is

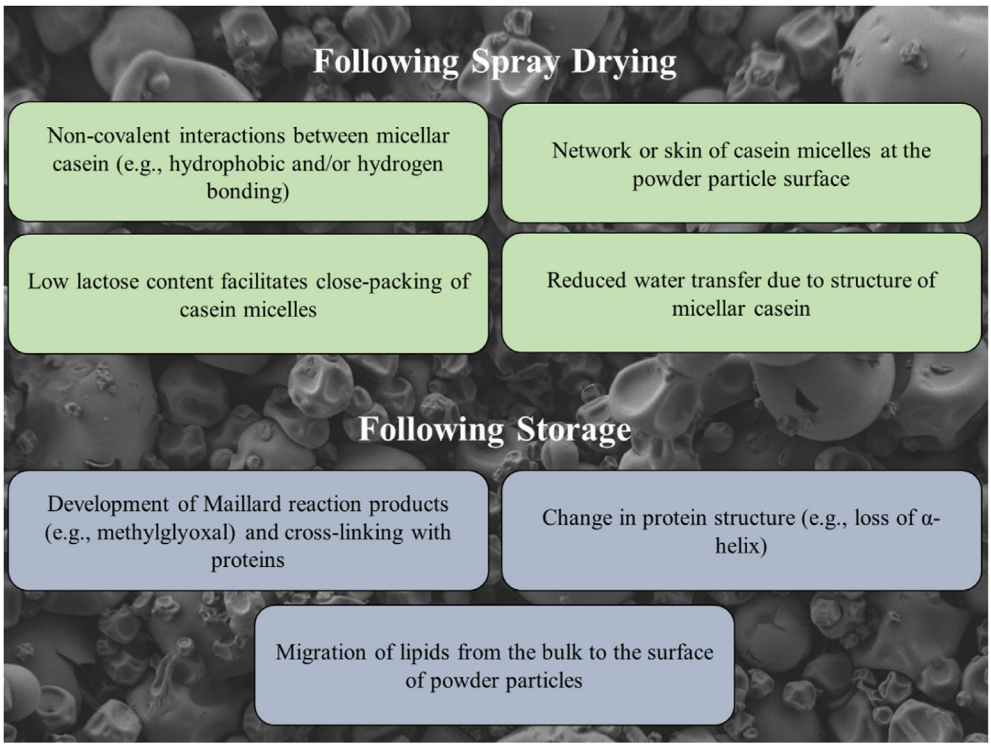

Fig. 1. Summary of research regarding the reasons for impaired rehydration of micellar casein-dominant powders following spray drying and on subsequent storage of spray dried powders.

given in Fig. 2. It is important to consider that many of the approaches discussed are applied for the purpose of creating a spray dried powder with enhanced rehydration properties, while other strategies are examined in the context of aiding powder solubilisation after spray drying.

3. Physical processing strategies to enhance powder rehydration

3.1. Addition of gas to the concentrate before spray drying

The addition of gases to dairy concentrates prior to spray drying has been investigated as an approach for modifying the physical and rehydration properties of powders. Marella, Salunke, Biswas, Kommineni, and Metzger (2015) injected carbon dioxide (CO_2) into skim milk before and throughout UF to modify the subsequent rehydration properties of MPC powder, with an improvement in powder solubility attributed to the solubilisation of calcium phosphate, caused by a reduction in pH due to the formation of carbonic acid (the effect of decreasing concentrate pH on subsequent powder rehydration is further discussed in Section 4.1.). Aside from altering the chemical composition of the powder (i.e., lower calcium content), gas injection has been used to improve rehydration performance by modifying the structure of powder particles. Bell, Hanrahan, and Webb (1963) produced skim milk powder with higher dispersibility by injecting compressed air into the product feed line of the spray dryer, between the high-pressure pump and atomisation nozzle. Recent studies by McSweeney, Maidannyk, O'Mahony, and McCarthy (2021a,b) demonstrated that nitrogen (N_2) gas injection prior to spray drying (i.e., between the high-pressure pump and atomisation nozzles) can improve the rehydration characteristics, particularly the dispersion and solubility, of MPC80 (i.e., 80%, w/w, protein). This improvement in water transfer was attributed to higher powder porosity and interstitial space, combined with lower powder density. Particle size distribution (PSD) analysis showed that the mean D_{90} value (i.e., the size of particles below which 90% of the sample lies), following reconstitution in ultrapure water (50 °C), was significantly lower for MPC powder produced using N_2 injection (0.4 μm) compared with the control (66 μm).

Bouvier, Collado, Gardiner, Scott, and Schuck (2013) used a novel technology called extrusion-porosification to produce MPC powders with a high dispersibility index (96%) compared with a conventionally spray dried MPC powder (38%). This process involved the incorporation of CO_2 into a high-total solids (38%, w/w) concentrate using a twin-screw extrusion-aeration system, followed by spray drying of a high-solids foam; after 2 h of rehydration, only sub-micron sized particles were present in the sample produced using extrusion-porosification, indicating complete dissolution. The formation of numerous pores within the powder particles and the partial dissociation of casein micelles were responsible for the improvements in water transfer and rehydration. It is evident that using gases such as N_2 and CO_2 during dairy processing can enhance the dispersion of dairy protein powders via changes in composition (e.g., reduced calcium content following the incorporation of CO_2 into the liquid concentrate), micellar casein structure and/or powder particle structure, depending on where in the process it is applied. However, an important consideration is the altered physical and bulk handling properties of such ingredients produced using gas injection (McSweeney et al., 2021a); for example, the injection of N_2 gas directly prior to spray drying can lower the particle and bulk density and produce cohesive powders that do not flow easily, thereby potentially presenting challenges in industrial powder handling processes.

Table 1
Overview of literature regarding physical processing strategies to enhance powder rehydration.[a]

Strategy	Powder	Measurement techniques	Results	Reference
Addition of gases				
CO_2 injection during membrane filtration	MPC80	Dispersion: Particle size distribution (PSD)	↑ dispersion	Marella et al. (2015)
		Solubility: Total solids (TS) before & after centrifugation (700×*g*, 10 min)	↑ solubility	
N_2 gas injection before drying	SMP	Solubility: TS before & after filtration (100 and 150 mesh funnel)	↑ dispersion	Bell et al. (1963)
	MPC80	Dispersion: PSD	↑ dispersion	McSweeney et al. (2021b)
		Solubility: TS before & after centrifugation (3000×*g*, 10 min)	↑ solubility	
Extrusion-porosification	MPC80	Dispersion: PSD and dispersibility index	↑ dispersion	Bouvier et al. (2013)
		Solubility: TS before & after centrifugation (160×*g*, 5 min)	↑ solubility	
High-shear treatment				
Microfluidisation before drying	MPC80 & 90	Solubility: Protein content before & after centrifugation (3000×*g*, 10 min)	↑ solubility MPC80	Augustin et al. (2012)
		Insolubility index: Sediment height after centrifugation (160×*g*, 10 min)	↔ solubility MPC90	
Homogenisation before drying	MPC80		↑ solubility	Augustin et al. (2012)
Hydrodynamic cavitation before drying	MPC80	Solubility: TS before & after centrifugation (700×*g*, 10 min)	↔ solubility	Li et al. (2018)
Homogenisation after drying	MPC80	Solubility: TS before & after centrifugation (700×*g*, 10 min)	↑ solubility	Sikand et al. (2012)
	MPC55 & 80	Dispersion: PSD	↑ dispersion	Warncke and Kulozik (2020)
	MPC80 & MC	Dispersion: PSD	↑ dispersion	Chandrapala et al. (2014a)
		Solubility: TS before & after centrifugation (4400 rpm for 5 min)	↑ solubility	
Hydrodynamic cavitation after drying	MPC80	Dispersion: PSD & analytical centrifugation (670×*g*, 3 h)	↑ dispersion	Pathania et al. (2018)
High-pressure processing				
Before drying	MPC85	Solubility: Protein content before & after centrifugation (3000×*g*, 10 min)	↑ solubility	Udabage et al. (2012)
Ultrasonication				
Before drying	MPC80		↑ solubility	Augustin et al. (2012)
	MPC80 & CaCas	Dispersion: PSD	↑ MPC solubility	Chandrapala et al. (2014b)
		Solubility: TS before & after centrifugation (2125×*g*, 5 min)	↔ CaCas solubility	
	MPC80	Dispersion: PSD	↑ dispersion	Yanjun et al. (2014)
		Solubility: TS before & after centrifugation (4400×*g*, 10 min)	↑ solubility	
After drying	MPC80 & MC		↑ dispersion ↑ solubility	Chandrapala et al. (2014a)
	MPC80	Dispersion: PSD	↑ dispersion	McCarthy et al. (2014)

Table 1 (*continued*)

Strategy	Powder	Measurement techniques	Results	Reference
		Solubility: TS before & after centrifugation (700×*g*, 10 min)	↑ solubility	
Membrane filtration				
Cold (4 °C) microfiltration	MCC75	Wettability: Contact angle	↔ wettability	Crowley et al. (2018)
		Dispersion: PSD	↑ dispersion	
Microfiltration and acidification	MCC85	Insolubility index: Sediment height after centrifugation (700×*g*, 10 min)	↑ solubility	Schäfer et al. (2021)
Feed concentration using nanofiltration	MPC60	Insolubility index: Sediment height after centrifugation (900×*g*, 5 min)	↑ solubility	Cao et al. (2015, 2016)
Agglomeration and granulation				
Fluidised bed granulation with binders (lactose, sucrose or water)	MPI	Wettability: Washburn method	↑ wettability	Ji et al. (2015)
		Dispersion: PSD	↔ dispersion	
Addition of lecithin or tween 80 during fluidised bed granulation	MPI	Wettability: Wetting time & contact angle Dispersion: PSD Solubility: Analytical centrifugation	↑ wettability ↔ dispersion & solubility	Wu et al. (2020)
Agglomeration using fines return during co-drying	NPC & WPI	Turbidity sensor	↑ wettability ↓ rehydration time	Gaiani et al. (2007)
Agglomeration using fines return	MPC80	Wettability: Capillary rise	↑ wettability	McSweeney et al. (2021b)
		Dispersion: PSD	↓ dispersion	
		Solubility: TS content before & after centrifugation (3000×*g*, 10 min)	↓ solubility	
Rehydration conditions				
Influence of temperature, stirring speed & solid concentration	MCI	Dispersion: PSD	↓ rehydration time with ↑ in temperature	Jeantet et al. (2010)
Influence of temperature, agitator & stirring speed	NPC	Dispersion: PSD	↓ rehydration time with ↑ in stirring rate	Richard et al. (2013)

[a] Abbreviations are: MPC, milk protein concentrate; SMP, skim milk powder; MC, micellar casein; CaCas, calcium caseinate; MCC, micellar casein concentrate; MPI, milk protein isolate; NPC, native phosphocaseinate; MCI, micellar casein isolate. The number following the powder abbreviation denotes the approximate protein content (%, w/w).

3.2. High shear: homogenisation, microfluidisation and hydrodynamic cavitation

High-shear treatments, including homogenisation, microfluidisation and hydrodynamic cavitation (HC), have been investigated as processing technologies that could be used to improve powder rehydration, without altering the ingredients chemical composition. Microfluidisation is a form of homogenisation which operates on the principle that the liquid is divided into two or more microstreams which are directed towards each other using a high-pressure pump (McCrae, 1994), whereby a combination of turbulent flow, cavitation and shear reduce droplet size (Maa & Hsu, 1999). Augustin, Sanguansri, Williams, and Andrews (2012) reported the effect of homogenisation or microfluidisation of the liquid concentrate before spray drying on the solubility of high-protein MPC powders after production and subsequent storage at 22 °C for eight months. The solubility of the MPC powder produced following microfluidisation of the concentrate (800 bar) was 89.5% after manufacture and 68.7% after eight months of storage, while in comparison, concentrates homogenised at first- and second-stage pressures of 350 and 100 bar had solubility values of 74.5 and 58.7% after production and eight months of storage, respectively. The solubility of the control powder (i.e., no treatment) was 70.1 and 51.1% at these respective time points, but statistical significance was not provided. In a separate investigation within this study, microfluidisation was applied at three different pressures (400, 800 and 1200 bar) to liquid MPC before spray drying and it was reported that solubility of the MPC powders was not significantly different from the non-microfluidised powders after manufacture and 2 months of storage, suggesting its use before spray drying may not be worthwhile. Another study involving high-shear treatment of dairy concentrate, performed by Li, Woo, Patel, Metzger, and Selomulya (2018), investigated the use of HC prior to spray drying and reported that concentrate viscosity decreased but powder solubility was not noticeably changed by the HC process. This technology involves the generation and collapse of bubbles due to changes in pressure, with the accompanying release of energy, causing a powerful mixing effect, which reduces particle size (Gogate, 2011).

An alternative option of using high-shear to enhance powder solubilisation after a standard spray drying process has also been reported by Sikand, Tong, Vink, and Walker (2012), whereby powder reconstitution in 37 °C water, followed by homogenisation (138 bar), improved the solubility of MPC powder. The mean solubility index, which represented the quantity of sedimented material present following centrifugation, was significantly lower

Table 2
Overview of literature regarding chemical modification and formulation strategies to enhance powder rehydration.[a]

Strategy	Powder	Measurement techniques	Results	Reference
Adjustment of pH before, during or after membrane filtration				
Acidification (pH 6.7, 6.0, 5.7, 5.4)	MPC85	Dispersion: Particle size distribution (PSD)	↑ dispersion	Liu et al. (2019)
		Solubility: Total solids (TS) before & after centrifugation (700×*g*, 10 min)	↑ solubility	
Acidification (pH 6.7, 6.3, 5.9, 5.5)	MPC55	Solubility: Protein content before & after centrifugation (12,000×*g*, 25 min)	↑ solubility (pH restoration)	Luo et al. (2016)
Acidification (pH 6.6, 6)	MPC65 & 80	Dispersion: PSD	↔ dispersion	Eshpari et al. (2014)
		Solubility: TS before & after centrifugation (700×*g*, 10 min)	↑ solubility	
Alkalinisation (pH 6.9, 7.3, 7.6)	MCC75	Wettability: Contact angle	↓ wettability	Panthi et al. (2021)
		Dispersion: PSD	↑ dispersion	
Ion exchange				
Before drying	MPI	Solubility: TS before & after centrifugation (700×*g*, 10 min)	↑ solubility	Bhaskar et al. (2001)
Addition of calcium-binding agents				
Sodium phosphate (SP), trisodium citrate (TSC) or sodium pyrophosphate before membrane filtration	MPC80	Dispersion: PSD	↑ dispersion	Sun et al. (2017)
		Solubility: TS before & after centrifugation (4400×*g*, 10 min)	↑ solubility	
SP or TSC via co-drying, bi-drying & dry-mixing	NPC	Insolubility index	↑ solubility	Schuck et al. (2002)
		Nuclear magnetic resonance (NMR)	↓ rehydration time	
Citrate before drying	MC85	Dispersion: PSD	↑ solubility	Schokker et al. (2011)
		Solubility: TS before & after centrifugation (750×*g*, 15 min)		
SP, TSC or sodium hexametaphosphate after drying	MPC80	Dispersion: PSD	↑ dispersion	McCarthy et al. (2017)
		Solubility: TS before and after centrifugation (3000×*g*, 10 min)	↑ solubility ↓ turbidity	
Addition of monovalent or divalent salts				
KCl or NaCl during diafiltration	MPC80	Solubility: Protein content before & after centrifugation (20,000×*g*, 30 min)	↑ solubility	Sikand et al. (2013)
NaCl during diafiltration	MPC80	Solubility: TS before & after centrifugation (700×*g*, 10 min)	↑ solubility	Mao et al. (2012)
NaCl or $CaCl_2$ before drying	NPC		↑ solubility (NaCl)	Schuck et al. (2002)
NaCl or $CaCl_2$ before drying	MC85		↑ solubility (NaCl)	Schokker et al. (2011)
NaCl before dying	NPC	Insolubility index	↑ solubility	Davenel et al. (2002)
		Rehydration time: NMR		
NaCl or $CaCl_2$ after drying	NMC	Rehydration time: Turbidity sensor	↓ rehydration time	Hussain et al. (2011)
Enzymatic or chemical modifications of protein				
Crosslinking using transglutaminase before drying	MPC80	Wettability: Washburn method	↑ wettability	Power et al. (2020)
		Diffusion: Confocal laser scanning microscopy	↑ diffusion	
Chymotrypsin, trypsin and papain after drying	MPC80	Solubility: Protein content before & after centrifugation (10,000×*g*, 10 min)	↑ solubility (pH 4.6–7)	Banach et al. (2013)
Flavourzyme™, Neutrase™ and Protamex™ after drying	MPI	Solubility: Protein content before &	↑ solubility (pH 6.5)	Ryan et al. (2018)

Table 2 (*continued*)

Strategy	Powder	Measurement techniques	Results	Reference
		after centrifugation (3000×*g*, 10 min)		
Succinylation after drying	MPC85	Dispersion: PSD Solubility: Protein content before & after centrifugation (1200×*g*, 20 min)	↑ dispersion ↑ solubility	Shilpashree et al. (2015)
Addition of dairy proteins				
NaCas before diafiltration, before drying or dry-blending with MC	MC85		↑ solubility	Schokker et al. (2011)
NaCas via wet- or dry-blending	MPI	Dispersion: PSD	↑ dispersion	Bot et al. (2020)
Whey protein before drying	NPC	Rehydration time: NMR	↓ rehydration time	Davenel et al. (2002)
Whey protein-rich peptide hydrolysate before drying	MPC80	Protein solubility assay	↑ solubility	Torres-Hernandez et al. (2018)
Addition of molecular spacers				
Addition of lecithin nanovesicles before drying using microfluidisation	MPC80	Solubility: TS before & after centrifugation (1000×*g*, 10 min)	↑ solubility	Bansal et al. (2017)

[a] Abbreviations are: MPC, milk protein concentrate; MPI, milk protein isolate; NPC, native phosphocaseinate; NMC, native micellar casein; MC, micellar casein; MCC, micellar casein concentrate; NaCas, sodium caseinate. The number following the powder abbreviation denotes the approximate protein content (%, w/w).

(1.02 mL) when homogenisation was applied compared with non-homogenised MPC (1.79 mL). Similarly, Warncke and Kulozik (2020) investigated the effect of high-pressure homogenisation (HPH; 100–500 bar) on the solubility of reconstituted (45 min at 50 °C) MPC55, MPC80 and MC powders. MPC55 already had a monomodal PSD in the casein micelle size range (i.e., 150–200 nm) after stirring and further treatment using HPH did not alter solubility. However, for MPC85, a monomodal PSD in this range was obtained after HPH at 200 bar, while a pressure of 500 bar was required to dissolve the MC powder. Furthermore, HC has also been investigated as a physical processing strategy for accelerating rehydration of spray dried powders. Pathania, Ho, Hogan, McCarthy, and Tobin (2018) demonstrated that HC was more effective in rapidly rehydrating MPC powders in comparison with conventional high-shear treatment. The volume-weighted mean particle diameter ($D_{[4,3]}$) value was significantly lower for the HC dispersion (0.19 μm) compared with the sample prepared using conventional high-shear mixing (5.62 μm).

It has been suggested by Augustin et al. (2012) that when high-shear treatments are applied to the concentrate prior to spray drying, these technologies may decrease viscosity and/or alter protein structure, thereby improving solubility of the subsequent powder. However, the exact mechanism by which this occurs has not been elucidated and some studies have found no beneficial effect on powder solubility using this specific approach (Li et al., 2018). Alternatively, when these physical processing strategies are used to reconstitute spray dried powders, enhancement of solubility is generally attributed to energy input, which accelerates the breakdown of large powder particles and disrupts protein–protein interactions; however, their use may incur high capital and operating costs. Overall, these technologies do not address the challenge encountered by ingredient manufacturers in creating high quality,

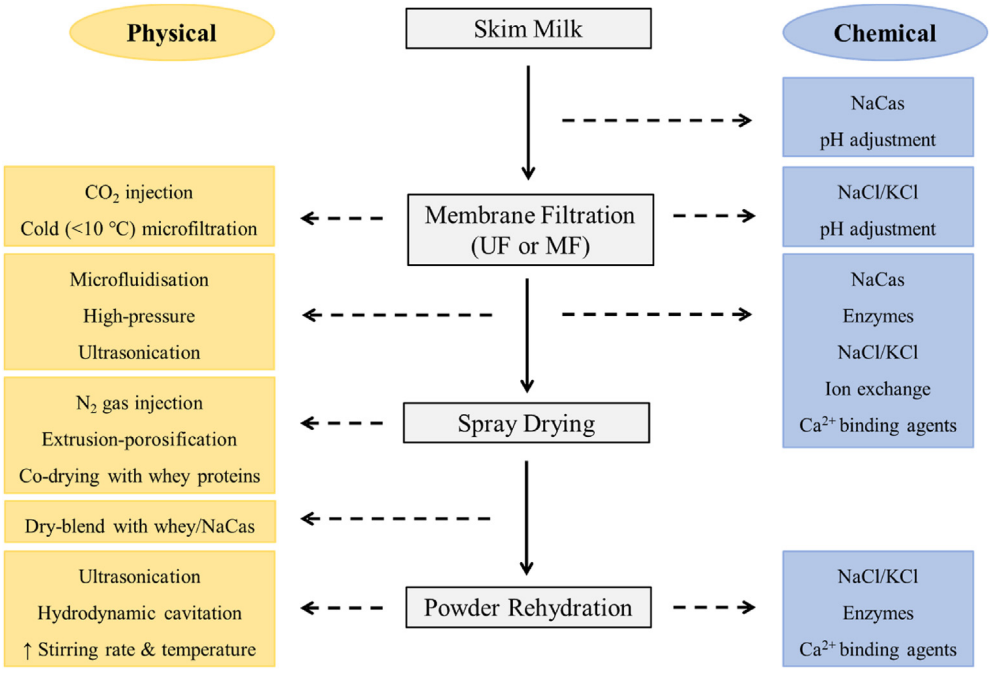

Fig. 2. Schematic representation of the stages during processing where physical and chemical modifications may be implemented to alter powder rehydration properties.

soluble powders for customers but would be useful for end-users who need to quickly reconstitute spray dried dairy powders for use in various applications.

3.3. High-pressure processing

The use of high-pressure (HP) treatment in dairy processing has been reviewed by Huppertz, Fox, de Kruif, and Kelly (2006) and Huppertz, Kelly, and Fox (2002), with some of the reported effects including whey protein denaturation and a change in casein micelle size, and the magnitude of these effects dependent on factors such as pressure and temperature. The potential use of HP treatment to enhance the rehydration characteristics of high-protein, micellar casein-dominant powders has been investigated by Udabage, Puvanenthiran, Yoo, Versteeg, and Augustin (2012). A range of pressures (100–400 MPa) and temperatures (10–60 °C) were applied to liquid MPC and the subsequent solubility of the MPC powder investigated after spray drying. The most significant improvement in solubility of the MPC was obtained when a pressure and temperature of 200 MPa and 40 °C, respectively, were applied to the concentrate, with the powder solubility value after this treatment being 85% compared with 66% for the MPC which received no HP treatment at 40 °C, and this was attributed to the partial dissociation of casein micelles to their non-micellar form. The authors also found that a high-protein powder produced by dry blending NaCas and whey protein isolate (WPI) had higher solubility than MPC, showing that micellar casein hinders the reconstitution process of these powders. Furthermore, it is important to note that MPC powders could not be produced when the concentrates were subjected to 200 MPa at 10 and 25 °C, or 400 MPa at 25, 40 and 60 °C, due to gelation caused by whey protein denaturation and dissociation of casein micelles. Cadesky, Walkling-Ribeiro, Kriner, Karwe, and Moraru (2017) also reported that HP processing (150–450 MPa) altered the physicochemical properties of liquid MPC and MCC, prepared at 2.5 and 10% protein (w/v). Dissociation of the casein micelles took place after the concentrates were subjected to a pressure of 150 MPa, while a gel formed after treatment at 450 MPa due to destabilisation and aggregation of casein micelles, with the denaturation of serum proteins also likely contributing. Therefore, gelation of concentrates would be an important factor to consider if HP were to be applied industrially for improving solubility of casein-dominant dairy powders. HP processing may be a useful strategy for partially dissociating casein micelles without altering the composition of the product or requiring the addition of other chemicals or ingredients; however, similar to high-shear treatments such as microfluidisation and HC, it may not be an economically feasible approach in terms of capital and operating costs.

3.4. Ultrasonication

There are two forms of ultrasonication (US) generally used in food processing: (i) low frequency (16–100 kHz), high intensity (10–1000 W cm^{-2}) and (ii) high frequency (100 kHz–1 MHz), low intensity (<1 W cm^{-2}) ultrasound (O'Sullivan, Park, Beevers, Greenwood, & Norton, 2017). Ultrasonic waves of high intensity induce changes to food systems through cavitation, capable of generating large increases in temperature and shear (O'Brien, 2007; O'Donnell, Tiwari, Bourke, Cullen, 2010). Chandrapala, Zisu, Palmer, Kentish, & Ashokkumar (2014b) performed US (frequency of 20 kHz, power of 31 W and amplitude of 50%) on reconstituted (i.e., stirred for 1 h at 22 °C followed by overnight storage at 4 °C) MPC and calcium caseinate (CaCas) dispersions prior to spray drying, and measured solubility initially and after storage (30 and 60 d at 25 °C) at a relative humidity (RH) of 23 and 76%. Powders had similar solubility values after manufacture; however, following 30 d of storage at 23% RH, US-MPC samples displayed higher solubility (~97%) than the MPC control (83%). After 60 d of storage, this trend persisted, with solubility values of ~88 and 63% for US and control MPC powders, respectively; in contrast, US did not alter the solubility of CaCas, remaining at ~90% throughout the study. The higher solubility of MPC powders after storage was attributed to the breakdown of whey protein-casein micelle aggregates during US. It is possible that the dispersions prepared for spray drying were not completely solubilised beforehand given the short reconstitution time, which may have contributed to the presence of large particles in the powder. Similarly, Augustin et al. (2012) performed US (24 kHz, 160 mL min^{-1} at 600 W) on UF retentate prior to spray drying and reported that the solubility of the MPC powder was only slightly improved, with the measured solubility for US and control MPC powders after manufacture being 74.7 and 70.1%, respectively, while after eight months of storage, solubility remained marginally higher (55.1%) for US-MPC compared with the control (51.1%).

It appears that the application of US prior to spray drying does not significantly alter powder solubility initially, but provides some protection against storage-induced loss of solubility. However, Yanjun et al. (2014) also investigated the relationship between the application of US (20 kHz, 12.5 W and 50% amplitude) to UF concentrates before spray drying and the solubility of the MPC powder. Solubility was significantly higher for the MPC which received 5 min of US pre-treatment (88.3%) compared with the control (35.8%). The authors attributed the increase in solubility to a change in protein structure and an increase in the presence of charged groups (e.g., COO^{-}), although this was not specifically measured. Similar to the results involving high-shear treatment of concentrates in Section 3.2., it is apparent that the exact mechanism by which US prior to spray drying could confer enhanced solubility to powders remains unclear.

US has also been investigated for its potential in accelerating powder solubilisation after the spray drying process. Chandrapala, Martin, Kentish, and Ashokkumar (2014a) compared the solubilisation of spray dried MPC and MC powders using US (20 kHz, 31 W, amplitude of 50%), HPH (single stage at 80 or 200 bar) or high-shear rotor-stator mixing (HSRSM; 17,500 rpm). The $D_{[4,3]}$ values for MPC and MC were considerably lower after US for 5 min (1.1 and 0.8 μm, respectively) compared with 5 min of HSRSM (25 and 52 μm, respectively). HPH performed similarly to US in reducing particle size as the $D_{[4,3]}$ was 1.2 and 0.3 μm for MPC and MC, respectively. Each of these three approaches provided an improvement in solubilisation of micellar casein-dominant powders as they accelerated the structural collapse of powder particles and the release of their constituents (e.g., caseins, minerals). McCarthy, Kelly, Maher, and Fenelon (2014) investigated the effect of US (20 kHz, 70.2 W and amplitude of 100%) and overhead stirring (450 rpm) on rehydration of MPC powder. PSD analysis showed that after 10 min of overhead stirring, the D_{90} of the MPC dispersion was 76.6 μm, compared with 0.41 μm after US for 1 min. Furthermore, the solubility of MPC dispersions after 10 min of overhead stirring in water at 25 and 50 °C was 45.8 and 89.7%, respectively, while solubility was 99.6% following US for 1 min. Similar to high-shear treatments described previously, US appears to be a useful technology in facilitating the rehydration of spray dried, high-protein dairy powders, but it could also present several challenges with implementation at an industrial scale. For example, the installation of an US system would involve significant capital costs, be difficult to incorporate into a continuous industrial process, it generally provides a localised effect and the probe could erode over time and contaminate the product with metal fragments.

3.5. Membrane filtration: micro-, ultra- and nanofiltration

As membrane filtration is the technological enabler in the production of high-protein products, it seems logical that interventions offering potential to improve subsequent powder solubility would be considered at this stage in the process, with several recent studies reporting the impact of membrane filtration unit operations and processing conditions on the physicochemical properties of the derived streams and subsequent spray dried powders. Crowley et al. (2018) produced MCC powders using MF and DF of skim milk at both cold (<10 °C) and warm (50 °C) temperatures, followed by spray drying. No differences were recorded between powders in their wetting behaviour or contact angle, as measured using optical tensiometry. PSD analysis demonstrated that MCC powders produced using cold MF had higher dispersibility than powders produced using warm MF; for example, after rehydration in water (50 °C) for 90 min, 48% of the particles had diameters <1 μm for MCC powders produced using cold MF, compared with 7.5% for powders produced using warm MF. This suggests that a higher proportion of casein micelles were present in solution (i.e., released from dissolved powder particles) following reconstitution of the cold MF powders. The superior dispersibility of MCC powder produced using cold MF was likely a result of several factors, including lower calcium, lower β-casein and higher whey protein content in such powders. Schäfer, Hinrichs, Kohlus, Huppertz, and Atamer (2021) used membrane filtration and pH adjustment to produce calcium-reduced MCC powders. This was achieved by concentrating the skim milk at pH 6.2 using MF, followed by acidification of the MF retentate to pH 5.6 and performing both MF and DF prior to spray drying. Powders depleted in calcium by approximately 50% had significantly higher solubility compared with the control powder, as they formed 3.1 and 4.7 mL of insoluble material, respectively.

France, Kelly, Crowley, and O'Mahony (2021) recently investigated the impact of temperature (4, 8, 12, 16 and 20 °C) and transmembrane pressure (0.05 or 0.30 bar) on membrane filtration performance and the physicochemical properties of the streams produced from the MF of skim milk. Concentrate viscosity was higher and membrane flux was lower when MF was performed at 4 °C, while protein retention by the membrane increased as the temperature and transmembrane pressure were increased. The effect of temperature (5, 20 and 50 °C) during UF of skim milk, the initial step in MPC manufacture, has been reported by Puri, Singh, and O'Mahony (2020). Similar to the previous study, permeate flux was lower at lower temperature, most likely due to increased viscosity, resulting in membrane fouling and the blockage of pores. The retentates produced at 5 and 20 °C had a significantly lower content of total calcium and phosphorus compared with that produced at 50 °C, suggesting that some colloidal calcium phosphate (CCP) was solubilised at the lower processing temperature. The effect of cold UF on the rehydration properties of MPC powders has not been established in the literature but would likely generate improvements in powder dispersion due to lower total calcium content. The industrial application of cold membrane filtration to manufacture high-protein, micellar casein-dominant powders would possibly be limited by the operating costs to maintain a low processing temperature, higher pressures to pass components of the viscous feed through the membrane and longer operating times to achieve the desired protein content in the retentate.

Cao et al. (2015) compared the use of nanofiltration (NF) or evaporation (EP) for concentration of UF retentate before spray drying on the physicochemical properties of MPC powders. The insolubility index (ISI) was significantly lower for NF-MPC (0.32 mL) compared with EP-MPC (0.90 mL), while the free sulfydryl group content of NF-MPC powder was significantly higher than that of EP-MPC. It is possible that the heat treatment received by the concentrate during EP may have caused the formation of protein aggregates which subsequently sedimented during centrifugation. A follow-up study by Cao et al. (2016) investigated the influence of storage on these powders over 24 weeks at 25, 35 and 45 °C. NF-MPC had better solubility compared with EP-MPC after storage; for example, after 24 weeks at 25 °C, the ISI was approximately 2.4 and 4.8 mL for NF- and EP-MPC, respectively. It is apparent that membrane filtration conditions and concentration processes applied prior to spray drying play a crucial role in manipulating the rehydration properties of micellar casein-dominant powders.

3.6. Agglomeration during spray drying and fluidised bed granulation

Agglomeration is generally used to improve the physical (e.g., flowability) and rehydration (e.g., wettability) characteristics of low-protein dairy powders such as whole milk and fat-filled powders (Písecký, 2012), but has recently been investigated as a strategy to modify the functionality of high-protein powders. Gaiani, Schuck, Scher, Desobry, and Banon (2007) spray dried WPI, NPC and NPC plus WPI concentrates, and produced agglomerated and non-agglomerated variants of the powders to investigate the influence of protein type and agglomeration on powder rehydration, with agglomeration performed by returning fine particles to the top of the drying chamber and bringing them into contact with the atomised feed. The wetting behaviour of agglomerated, casein-dominant powders was improved compared with the non-agglomerated powders, but dissolution was impaired. McSweeney et al. (2021b) produced agglomerated MPC powders using fines return during spray drying and reported greater capillary rise wetting and water diffusion, but impaired dispersion and solubility, for the agglomerated powders compared with non-agglomerated MPC.

When agglomeration is performed in a fluidised bed towards the end of the spray drying process, the term granulation is often used to describe this process of joining powder particles together using binding agents. Ji et al. (2015) granulated MPI powders in a fluidised bed system using water or binders (i.e., sucrose or lactose solutions). Wettability was higher for MPI agglomerated using lactose, while it was lowest for the non-agglomerated MPI. The quantity of water absorbed increased with increasing powder particle size for all samples. However, PSD analysis demonstrated that granulation and the use of hydrophilic binders did not result in any improvement in the dispersion and solubilisation of the MPI powders. Wu, Fitzpatrick, Cronin, Maidannyk, and Miao (2020) sprayed surfactants (Tween 80 and lecithin) onto MPI powder during granulation in a fluidised bed and reported that wetting times were lower for Tween 80 and lecithin coated powders in comparison with the MPI powder with no added surfactant (e.g., 15−50 s for MPI coated with Tween 80 compared with 36 min for the MPI control), most likely due to reduced surface tension on inclusion of surfactant. However, dispersion and solubility were not significantly improved by the use of these surfactants. Therefore, agglomeration during spray drying and the use of surfactants or binders in fluidised bed granulation can improve the instant properties of micellar casein-dominant powders but are generally ineffective in improving the key subsequent stages of rehydration (i.e., dispersion and dissolution).

3.7. Rehydration conditions

The selection of appropriate rehydration conditions (e.g., solvent temperature, total solids content, stirring rate, impeller

design) can play an important role in optimising the dissolution of casein-dominant powders and thereby increase process efficiency for manufacturers. Jeantet et al. (2010) investigated the effect of temperature (26–30 °C), total solids concentration (4.8–12%, w/w) and stirring rate (400–1000 rpm) on the rehydration characteristics of MC powder. Temperature played a significant role in the process as it was shown that a 4 °C increase in temperature had the same effect on rehydration kinetics as doubling the stirring rate from 400 to 800 rpm. Increasing the concentration of solids significantly increased the stirring rate required but did not affect rehydration time to the same extent as temperature. Therefore, it was suggested that temperature is a crucial parameter to consider when rehydrating casein-dominant dairy powders. Richard et al. (2013) monitored how temperature (25 and 30 °C), stirring speed (500–900 rpm) and agitator design (six-pitched-blade impeller or two impellers with right angled arrangement) influenced the rehydration behaviour of granulated and non-granulated NPC, WPI, NPC plus WPI and NPC plus lactose powders. Increasing stirring speed from 700 to 900 rpm reduced rehydration time by 25% on average; however, similar to previous work by Jeantet et al. (2010), rehydration was more sensitive to changes in temperature than stirring rate. Granulated powders required longer rehydration times, particularly for NPC powders, e.g., 380 min for granulated NPC compared with 220 min for non-granulated NPC at 900 rpm. The choice of impeller design impacted the rehydration of NPC powder in particular; the 6-pitched blade design resulted in greater particle breakdown due to greater energy dissipation, while the dual propeller design instead created more particle circulation. It is evident that higher temperatures and stirring rates are advantageous in accelerating the rehydration of micellar casein-dominant powders but would result in greater energy consumption.

4. Chemical modification and formulation strategies to enhance powder rehydration

4.1. Adjustment of pH before, during or after membrane filtration

Several studies have investigated the effect of reducing the pH of skim milk during membrane filtration and the subsequent solubility of the MPC powders produced. Liu et al. (2019) acidified skim milk (pH 6.7, 6.0, 5.7 and 5.4) using glucono-delta-lactone (GDL) before membrane filtration, followed by pH restoration of the retentate directly prior to spray drying. The amount of total calcium present in the reconstituted MPC powder was lowest for the sample pre-acidified to pH 5.4, which can be attributed to the passage of serum calcium through the membrane into the permeate following solubilisation of CCP. PSD analysis showed a decrease in particle size of MPC dispersions with decreasing pH from 6.7 to 5.4. Solubility values for the MPC dispersions increased with decreasing pH of pre-acidification and were slightly higher when the retentate pH was re-adjusted prior to spray drying compared with samples which were acidified only. The pH 6.7 sample had an initial solubility of 89% but this was just 19% after 84 d of storage at 40 °C; however, the pH 5.7 sample prepared from pH restored retentate had a solubility of 97 and 91% at these time points, respectively. Importantly, this demonstrates that storage-induced solubility loss can also be reduced when skim milk is acidified prior to membrane filtration and spray drying. Luo, Vasiljevic, and Ramchandran (2016) acidified skim milk (pH 6.7, 6.3, 5.9 or 5.5) prior to UF and freeze drying. Lowering the pH of the skim milk feed from 6.7 to 5.5 before membrane filtration resulted in a significant decrease in solubility of the reconstituted MPC powders from 77 to 32%. However, upon restoration of the MPC dispersion to pH 6.7, this trend was reversed, e.g., ~90 and 73% solubility for pH 5.5 and 6.7 samples, respectively. In addition to the effects on powder solubility, lowering the pH of the feed to 5.5 significantly reduced membrane flux as pores became blocked, and the factors contributing to this included changes in casein micelle size, solubilisation of salts from the micelle and increased viscosity. Eshpari et al. (2014) acidified skim milk to pH 6 using GDL prior to UF alone or UF combined with DF, and reported that acidification caused a significant decrease in the calcium content of MPC from 1.84 to 1.59 g 100 g^{-1} powder. Solubility was higher for the MPC which was acidified using GDL (~82%) before UF and DF compared with the control which received no GDL treatment (~72%). However, the PSD profiles following reconstitution of control and acidified MPC powders were similar, with monomodal peaks in the size range 10–300 μm. Thus, some disparities are apparent in the rehydration data available from experiments involving pH adjustment before membrane filtration and further work is required to ascertain the effects on both powder dispersibility and solubility. Alternatively, Panthi et al. (2021) increased the pH of MF retentates (pH 6.9 to pH 7.3 and 7.6) prior to freeze drying and reported that MCC powders had lower wettability but higher dispersibility with increasing retentate pH. The powder derived from the retentate that was re-adjusted from pH 7.6 to pH 6.9 had the highest dispersibility and this was attributed to changes in the ionic environment of the serum phase (e.g., higher calcium concentration resulting from partial solubilisation of CCP). This supports the positive effect of pH re-adjustment on powder rehydration performance that was reported in previous studies by Liu et al. (2019) and Luo et al. (2016).

The pH adjustment of dairy concentrate enables the mineral profile of the powder to be altered via a reduction in the CCP content, and this appears to enhance solubility of resultant powders. However, casein-dominant powders with reduced levels of micellar casein and calcium phosphate may not be suitable for applications such as cheese manufacture. Lucey and Fox (1993) discussed the significant role played by calcium and phosphate in the production of several cheeses, including their impact on rennet coagulation and gel strength, while Lin, Kelly, O'Mahony, and Guinee (2017) reported that an increased presence of non-micellar casein, generated by the addition of NaCas to skim milk, can adversely affect rennet gelation as it impairs the formation of a gel network. Another consequence of concentrate acidification to consider is that the permeate generated from such a process will contain higher levels of calcium and phosphorus, which may present challenges in down-stream processing (e.g., higher levels of demineralisation may be required).

4.2. Use of ion exchange and calcium-binding agents

Reducing the calcium content of micellar casein-dominant dairy concentrates before spray drying has proven to be an effective approach for increasing solubility of resultant powders. Bhaskar, Singh, and Blazey (2001) described a process for producing a calcium-depleted MPI with improved solubility in water (20 °C); briefly, the retentate from UF of skim milk was acidified from pH 6.8 to 5.9 using citric acid and removal of calcium was performed using a strong cation exchange resin in the sodium form. After 1, 6, 15, 22 and 36 d of storage at 20 °C, the calcium-depleted powders (33, 50 and 83% calcium depletion) all showed 100% solubility. In comparison, control MPI powders had 70–80% solubility after storage for 1–6 d, and this was reduced to 50% after 15, 22 and 36 d.

In addition to ion exchange resins, calcium-binding agents have been used to reduce calcium contents and modify the functional properties of casein-dominant powders. Sun et al. (2017) added trisodium citrate (TSC), sodium pyrophosphate (SPP) and sodium phosphate (SP) to skim milk (0.3% of total solids) before membrane filtration. Calcium content was reduced significantly by the

addition of each calcium-binding agent. After stirring for 30 min, the median particle size was 40 μm for the control MPC, compared with 25, 20 and 25 μm for powders spray dried containing TSC, SP and SPP, respectively, while the solubility was 40, 67, 59 and 51% for control, TSC, SP and SPP powders, respectively. The sample with the highest solubility (83%) at that time point was one which contained a mixture of TSC and SPP (50:50). Schuck et al. (2002) produced NPC powders with added TSC or SP using three different manufacturing approaches: (i) co-drying (CD): calcium-binding agents added to NPC before spray drying, (ii) bi-drying (BD): mineral salt solution and NPC suspension spray dried together, and (iii) dry-mixing (DM): powders physically blended together after spray drying. NPC manufactured without additional calcium-binding agents had an ISI of 14.4 mL compared with <0.2 mL when SP (12 g 100 g^{-1} solids) and TSC (30 g 100 g^{-1} solids) were added before spray drying. Insolubility values were similar when SP and TSC were added using BD (1.8 and < 0.2 mL, respectively) but higher when SP and TSC were added via DM (13.9 and 7.5 mL, respectively). This suggests that the addition of calcium-binding agents should be performed prior to spray drying. TSC was more effective than SP at increasing solubilisation, as measured using a nuclear magnetic resonance (NMR) relaxometry technique; however, it is important to note this powder had lower protein content as greater amounts of this mineral salt were added. Similarly, Schokker et al. (2011) added citrate to the concentrate before drying and produced an MC powder with solubility of 79.5%.

Calcium-binding agents have also been used to promote powder dissolution after spray drying. McCarthy et al. (2017) added sodium hexametaphosphate (SHMP), SP or TSC (0–150 mEq L^{-1}) to MPC solutions prepared from reconstituted powder. PSD analysis showed that TSC and SHMP significantly improved the dispersion of MPC powders, particularly with increasing concentration of SHMP, while SP did not have a significant effect. Powder solubility was lower for the MPC control (89.7%) compared with 96.1 and 99.5% following the addition of 15 mEq/L of TSC and SHMP, respectively, with the changes in solubility attributed to the dissociation of casein micelles. Similarly, Nogueira et al. (2020) investigated the behaviour of demineralised and native casein micelle powders during rehydration, with calcium contents of 2.7 and 2.1 g 100 g^{-1} powder for control and demineralised samples, respectively. Following stirring at 50 °C for 1 h, large particles (>10 μm) were present in both samples and further analysis using electrophoresis demonstrated that non-covalent interactions played an important role in the formation of these aggregates. However, it is not possible to fully elucidate the reason for this as the type of calcium-binding agent used to manufacture the demineralised powder was not given.

Despite the reports of ion exchange and calcium-binding agents generally improving powder rehydration, it would be important to consider the limitations of their use. With the removal of calcium using ion exchange, the composition, technological (e.g., gelation) and nutritional properties of the powder would be altered and this should be carefully considered before their use in specific applications that require this micronutrient (e.g., clinical nutrition beverages). Moreover, the use of calcium-binding agents may alter ingredient listings, which may be undesirable in the food industry considering the increased consumer demand for more "clean label" products (Asioli et al., 2017).

4.3. Addition of monovalent or divalent salts

The incorporation of monovalent or divalent salts such as potassium chloride (KCl), sodium chloride (NaCl) and calcium chloride ($CaCl_2$) into dairy concentrates is a strategy that has been reported to modify powder dissolution. In a study by Sikand, Tong, and Walker (2013), the addition of NaCl or KCl (150 mM) to UF retentate during DF improved the solubility of MPC powder, whereby NaCl and KCl treated MPC powders had 100% solubility compared with 53% when no salt was added. The higher solubility of these MPC powders was likely related to the significantly lower calcium content of the powders with salt added during DF, suggesting that some solubilisation of CCP may have occurred during membrane filtration. Mao, Tong, Gualco, and Vink (2012) added increasing concentrations of NaCl (0–150 mM) to the retentate at the DF step during the manufacture of MPC, with solubility increasing with increasing concentration of NaCl added, e.g., after reconstitution for 30 min, solubility was approximately 95% with the addition of 150 mM NaCl, compared with only 33% for 0 mM NaCl. The number of exposed hydrophobic regions on the MPC proteins increased significantly, while average particle size and disulphide bond formation decreased significantly, with the addition of 50, 100 and 150 mM NaCl. The change in surface hydrophobicity suggests that NaCl caused a change in protein structure, while the decrease in the number of disulphide bonds could possibly account for the measured improvements in powder rehydration. In the study by Schuck et al. (2002), NPC powders with added NaCl and $CaCl_2$ were also produced. The ISI was 0.9 mL when NaCl was added (12 g 100 g^{-1} solids) by CD compared with 14.6 mL with $CaCl_2$ addition (11 g 100 g^{-1} solids). The positive impact of NaCl addition on NPC rehydration was related to the hygroscopic strength of salt rather than its effect on casein micelle hydration and structure. Schokker et al. (2011) reported that an MC powder which was manufactured by adding NaCl before DF had a solubility of 82.8%. Davenel, Schuck, Mariette, and Brulé (2002) also produced NPC powders containing additional NaCl. The reconstitution time, measured using NMR, and ISI values were 22 min and 14.4 mL for the NPC control, compared with 9.5 min and 9 mL when NaCl was added (12 g 100 g^{-1} solids) prior to spray drying, respectively. Carr, Bhaskar, and Ram (2004) also reported a process whereby NaCl added to UF retentate prior to spray drying was shown to improve powder solubility.

Hussain, Gaiani, Aberkane, and Scher (2011) used NaCl and $CaCl_2$ solutions, ranging in concentration from 0 to 12% (w/v), to reconstitute native micellar casein (NMC) powder and turbidity measurements were used to provide rehydration times for each solution. NMC alone had a rehydration time of 467 min, as indicated by turbidity stabilisation, but this was reduced to 238 and 192 min when the concentration of NaCl and $CaCl_2$ was 6% (1034 mM), respectively. The shorter rehydration time for the sample containing $CaCl_2$ appears to contradict a previous report of this salt not enhancing solubility when added before spray drying (Schuck et al., 2002), possibly due to differences in the stage of addition, concentration and measurement techniques. When salt concentrations of 6% were used, no swelling stage was observed, possibly due to changes in micellar structure, and it has been reported by Famelart, Le Graet, and Raulot (1999) that NaCl induced solubilisation of calcium and phosphorus when added to casein micelle suspensions but the addition of $CaCl_2$ did not cause any applicable modification. Similar to the removal of calcium as mentioned in Section 4.2., the addition of NaCl would negatively affect the nutritional content of the powder, particularly given its influence on cardiovascular health (Aaron and Sanders, 2013). However, KCl appears to be equally as effective for altering powder rehydration when added before spray drying, and may represent a more consumer-friendly and health-conscious alternative for powder end-users.

4.4. Enzymatic or chemical modifications of protein

Enzymes are used to perform several functions in the dairy industry, most notably the role of chymosin in cheese curd formation and proteinases to decrease allergenicity and improve the digestibility of infant formula (Nongonierma and FitzGerald, 2011). Modifying dairy protein structure and functionality using enzymes has also been explored as a strategy to enhance the rehydration of high-protein powders. Power, Fenelon, O'Mahony, and McCarthy (2020) produced MPC powders which were enzymatically cross-linked using transglutaminase (TGase) prior to spray drying to maintain micellar structure and control viscosity, as well as depleted in calcium using SHMP (0–25 mM) to improve rehydration performance of resultant powders. Capillary rise wetting and water sorption values were higher for TGase treated than control powders, which suggests this enzymatic treatment had a positive effect on water absorption. Diffusion was higher for TGase treated powders compared with control powders, which increased with increasing concentration of SHMP. Alternatively, Banach, Lin, and Lamsal (2013) performed enzymatic hydrolysis of reconstituted MPC using three digestive enzymes (chymotrypsin, trypsin and papain) and one cysteine protease (papain). All enzyme treated samples displayed increased protein solubility in the pH range 4.6–7.0 compared with the control powder. Similarly, Ryan, Nongonierma, O'Regan, and FitzGerald (2018) investigated the influence of enzymatic modification on the functional properties of reconstituted MPI powders. The enzymes used were Flavourzyme™, Neutrase™ and Protamex™ and the solubility index of the MPI hydrolysates was measured over the pH range 2–8. At pH 6.5, the MPI control had ~35% solubility; however, after incubation for 180 min, the solubility was 90, 97 and 88% for the MPI samples enzymatically treated with Flavourzyme™, Neutrase™ and Protamex™, respectively. The authors attributed the increase in solubility to the formation of low molecular weight, hydrophilic peptides, while a limitation of protein hydrolysis in this case would be that it changes the product to an extent to which it may no longer retain its original ingredient identification.

Aside from the use of enzymes to alter the chemistry of dairy proteins, Shilpashree, Arora, Chawla, and Tomar (2015) chemically modified the dairy proteins in MPC powder using succinylation, whereby succinyl groups were transferred to the ε-amino group of lysine residues, resulting in a change in amino acid charge from positive to negative. MPC proteins subjected to succinylation (90%) using succinic anhydride had a solubility of ~78% at pH 6 compared with 30% for the control. In addition, the average particle diameter was 200 and 720 nm for modified (i.e., 90% succinylation) and control MPC proteins, respectively. The improvements in solubility were attributed to changes in protein charge and a decrease in protein–protein interactions. Further research on the use of enzymatic or chemical modifications of dairy protein concentrates or powder dispersions, their feasibility and behaviour during pilot or industrial-scale processing (e.g., evaporation and spray drying) and their impact on other techno-functional and sensory properties of powders are required.

4.5. Addition of dairy proteins

The addition of whey or non-micellar casein proteins to high-protein, casein-dominant powders may appear counterintuitive but is based on the concept that lowering the concentration of micellar casein or partially dissociating casein micelles can promote solubilisation without reducing the total protein content of the powder. Schokker et al. (2011) added NaCas to the concentrate at different stages of the process and investigated the subsequent powder rehydration properties initially and after storage. The MC powder produced when NaCas was added before DF (1.5%) had a solubility of 79.0% compared with 69.7% for the control. The solubility was higher when NaCas was added directly before drying compared to when NaCas was dry-blended with the spray dried powder. The improvement in MC reconstitution was attributed to increased levels of non-micellar casein and the two mechanisms proposed to explain this observation were: (1) non-micellar casein could preferentially adsorb at the air–water interface instead of casein micelles during spray drying which would prevent the formation of a network of casein micelles at the surface of the powder, and (2) non-micellar casein may act as a physical spacer molecule and prevent the association of casein micelles with each other. Bot, Crowley, and O'Mahony (2020) compared the addition of NaCas to MPI powder, by wet- or dry-blending, on dispersion and solubility. The MPI control (i.e., no NaCas added) had a solubility of 89.6% but this was 92.3 and 97.5% when NaCas was added (15% of total protein) via the wet- and dry-blending approaches, respectively. The PSD profile for the MPI control and MPI plus NaCas wet-blended samples were similar, with both having a monomodal peak in the size range 6–100 μm. However, the MPI plus NaCas dry-blended powders all had bimodal distributions, with a peak <1 μm and a second peak between 6 and 100 μm. This suggests that dispersibility increased as the proportions of NaCas dry-blended with MPI powder increased.

Davenel et al. (2002) added whey proteins to NPC before freeze drying and measured its rehydration performance using NMR. Freeze dried NPC had a reconstitution time of 32 min but this was 13 min for the sample enriched with whey proteins (i.e., 12% of total solids). Torres-Hernandez, Howell, and Bennett (2018) reported that adding a whey proline-rich peptide hydrolysate (DISSEP), produced from enzymatic hydrolysis of WPI, to reconstituted MPC could improve protein solubility following storage at 4 °C. The addition of dairy proteins provides dairy manufacturers with a practical and convenient approach for improving powder rehydration and may add further value to the incorporated ingredients. Nevertheless, this approach alters the original composition, physical state and often the protein profile of the powder (i.e., lower proportion of micellar casein) and does not resolve the fundamental issue of solubilising a micellar casein-dominant powder.

4.6. Addition of molecular spacers

The introduction of other food ingredients (e.g., soy lecithin) into high-protein concentrates to spatially separate micellar casein and reduce protein–protein interactions has recently been investigated by Bansal et al. (2017). Microfluidisation was applied to soy lecithin dispersions (5%, w/w) to create nanovesicles with an average hydrodynamic diameter of 82 nm. These dispersions were then added (1, 5 and 10% of milk solids, w/w) to the concentrate (11% total solids, w/w) prior to spray drying. The MPC powders containing 5% lecithin had significantly higher solubility at the beginning of the study and after 30 d of storage at 25 °C than the MPC powders containing 0 and 1% lecithin. Furthermore, after 90 d of storage at 25 °C, all powders containing lecithin nanovesicles had significantly higher solubility than the control MPC. However, after 180 d, no significant difference in solubility was observed between samples, while MPC powders containing 5 and 10% lecithin did not differ significantly during the study. Although this presents an interesting approach for modifying powder solubility, it alters the powders chemical composition which may limit its use in certain applications. The concept of adding molecular spacers or fillers such as Sephadex beads (Barden, Osborne, McMahon, & Foegeding, 2015) or glass beads (Thionnet, Havea, Gillies, Lad, & Golding, 2017) to cheese has also been reported, whereby they were used to replace milk fat and investigate the subsequent rheological

properties of low-fat cheese. Futher research is required to evaluate if other molecular spacers (e.g., whey protein nanoparticles) could be used to design innovative dairy product structures and enhance the rehydration properties of micellar casein-dominant powders.

5. Conclusion and perspectives for the future

Improving the rehydration performance of high-protein, micellar casein-dominant dairy powders remains a significant challenge and the selection of suitable processing strategies by manufacturers thereof is influenced by numerous, inter-related factors (e.g., capital and operating costs, bulk powder properties and end-user applications). Furthermore, any chemical or formulation changes made to the existing micellar casein-dominant powders available industrially need to be considered with respect to regulatory compliance and maintenance of established standards of identity, in addition to any potential changes to taste perception and consumer acceptance.

Although not the rate-limiting stage of rehydration, the wettability of these powders can be improved using food-grade surfactants (e.g., lecithin) or agglomeration. Altering dairy concentrate composition and physical state (e.g., dissociation of micellar casein and reduction of calcium content using ion exchange) or injecting gas directly prior to spray drying to influence powder particle structure, appear to be the most effective strategies at enhancing the dispersibility and solubility of micellar casein-dominant dairy powders. However, a strategy that successfully accelerates powder rehydration, without altering the chemical composition or physical properties of these types of powders, has not yet been developed. When the end-user needs to solubilise and rehydrate powders prior to their inclusion in food and beverage products, the use of high-shear or turbulence-inducing equipment (e.g., hydrodynamic cavitation) is essential. Further research is required to advance our knowledge of high-protein, micellar casein-dominant dairy powders, such as exploring additional or alternative drying technologies (e.g., electrostatic spray drying and spray freeze drying), developing universal analytical techniques for characterising the stages of powder rehydration, creating an international system for categorising or grading powder dispersibility and solubility, and establishing a fundamental and comprehensive understanding of insolubility development during dehydration and storage (e.g., the mechanisms and nature of casein micelle interactions).

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this review paper.

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