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Sonication of milk protein solutions prior to spray drying and the subsequent effects on powders during storage

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ABSTRACT

Solutions of 10 wt% solids, reconstituted from three types of dairy powders (milk protein concentrate, calcium caseinate and whey protein concentrate) were sonicated prior to lab scale spray drying at inlet and outlet temperatures of 180 °C and 80 °C respectively. The aim was to investigate the effects of sonication on the surface composition and morphology of the resulting powders. The effects of storage on the functional behaviour of these powders were also observed over 60 days at 25 °C, under two different humidity conditions (23.1% and 75.3%). No significant changes to the surface composition (fat, protein and lactose) of the sonicated and non-sonicated powder samples occurred during storage. The microstructure of non-sonicated powder samples showed the appearance of particle agglomerates upon storage, whereas this was not observed with sonicated samples. Importantly, it is shown that sonication prior to drying can increase powder stability during storage and delay the loss of powder solubility. When powders are reconstituted, the increase in solution viscosity that is normally associated with long term storage is also slowed. This is beneficial to industry where powder stability is important for reconstitution and use in the manufacture of secondary dairy products.

1. Introduction

Milk is often spray dried for preservation, long-term storage and ease of shipping and distribution. The resulting powder has an average shelf life of 0.5–2 years (Fyfe et al., 2011). Various powders are prepared from milk, each with specific application. For example, Milk Protein Concentrate (MPC) is a high-protein milk powder commonly used in the food industry as a functional ingredient and valued for its nutritional quality. Whey protein concentrate (WPC) powders are also essential ingredients in health bars and nutritional formulae and are emerging as key ingredients in energy gels and pastes. They are one of the few ingredients that have been shown to modulate immune functions (Perez-Cano et al., 2008). Calcium caseinate (CaCas) is also a protein derived from milk casein and is used widely as a processed food ingredient as well as a dietary supplement for bodybuilders and athletes.

Evaporation of moisture in a drying droplet simultaneously leads to migration of milk components toward the surface to replace the aqueous phase. As the components migrate at different rates, the surface skin has a significantly different composition to the core, with higher levels of fat and lower levels of lactose (Kim et al., 2002; Kentish et al., 2005). This surface composition can affect functional properties such as the stickiness, wettability, particle size distribution, bulk density, and flowability of the powders (Kim et al., 2002; Nijdam and Langrish, 2005). Since fat is more hydrophobic than lactose, the high fat concentrations on the particle surface also slows the wetting process during re-constitution (Murrieta-Pazos et al., 2012). Conversely, the powder particles can also contain inter-particle pores and these facilitate re-wetting during reconstitution (Murrieta-Pazos et al., 2011). Generally, the ability of dairy powders to rehydrate declines during storage (Fyfe et al., 2011). In high-protein milk powders, this decline is related to the micellar casein content of the powder, as these micelles tend to aggregate near the surface of the powder particle and are released into solution more slowly than whey proteins (Mimouni et al., 2010a,b). It is claimed that the micellar casein forms a porous, gel-like structure that restrains the dispersion of individual micelles into the surrounding liquid phase without preventing water penetration and solubilisation of nonmicellar components. During storage of the powder, increased interactions occur between and within micelles, leading to compaction of micelles and the formation of an aggregated skin of casein micelles packed closely together.

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Our prior research has shown that sonication can lead to increased heat stability in milk and whey solutions, with the solution viscosity remaining low after further heat treatment. These effects are maintained after spray drying (Ashokkumar et al., 2009; Zisu et al., 2010). Augustin et al. (2012) have shown that shear of any type (homogenisation, microfluidisation) or ultrasonication of MPCs prior to spray drying can increase powder solubility upon storage to some degree. Udabage et al. (2012) observed a similar increase in solubility using high hydrostatic pressure treatment and attributed this improvement to an increase in the non-micellar content of the proteins. The main aim of this work was to introduce sonication prior to spray drying to observe if this can also influence the storage stability of a range of spray dried, high protein dairy powders. Specifically, we studied the surface composition, size and microstructure of the dried powder particles and their solubility and viscosity upon re-constitution.

2. Materials and methods

2.1. Materials

MPC, WPC and CaCas powder samples were obtained from MG Nutritionals (Cobram, Victoria, Australia). The compositions of the powders are shown in Table 1.

Powders were reconstituted in water of greater than 18 MΩ cm^{-1} (MilliQ, Merck Millipore, Victoria, Australia) to obtain 100 g/kg (w/w) solutions. The solutions were continuously stirred for one hour at room temperature (22 ± 3 °C) to ensure complete mixing and stored overnight at 4 °C. On the following day, the solutions were equilibrated at 25 °C for 1 h prior to further processing.

2.2. Methods

60 mL solutions were sonicated in a glass vessel equipped with a cooling jacket using a 20 kHz, 450 W Ultrasonic horn (19 mm diameter, Branson Sonifier 450, Danbury, CT) at an amplitude of 50% for 1 min. The actual power delivered to the solution was 31 W as determined by calorimetry, which equates to an applied energy density of 31 J/mL. During sonication, chilled water was circulated continuously through the cooling jacket to maintain the sample temperature at 6 ± 4 °C.

Spray drying of the sonicated/non-sonicated solutions were performed with a laboratory spray drier B290 (Buchi Flawil, Switzerland). The inlet and outlet temperatures of the dryer were set according to the manufacturer's guidelines at 180 °C and 80 °C, respectively. The pump rate was set at 39% with the nozzle cleaner set at 5 (to reduce blockage of the nozzle). Powders were collected in a cyclone collector and immediately stored in a dry chamber at 4 °C. The moisture content of the spray dried powders varied between 5% and 7% w/w, with no differences found between sonicated and non-sonicated powder samples. The spray-dried powders were then dried in vacuum desiccators over P_2O_5 for a week to ensure the powders were uniformly fully dry prior to further testing.

The powders were then stored under two different relative humidity (RH) conditions (23.1% & 75.3%) in desiccators that were vacuum sealed and then placed in a temperature controlled room at 25 °C. The first condition (23.1%) was chosen to reflect typical environmental moisture levels. The second condition (75.3%) was chosen to be more extreme so that the rate of moisture related reactions would be accelerated. The equilibrium RH conditions were maintained using saturated salt solutions containing potassium acetate and sodium chloride respectively. The moisture content of each powder was measured by taking the weight of the powder sample every week for up to 60 days. Triplicate samples at each condition were evaluated.

Table 1
Composition of the powders according to manufacturer's specifications.

Composition	MPC (%)	CaCas (%)	WPC (%)
Protein	80.5	92.2	81.5
Fat	1.4	0.8	4.4
Lactose	5.6	0.25	10.4
Ash	7.3	3.8	
Moisture	5.2	5.4	

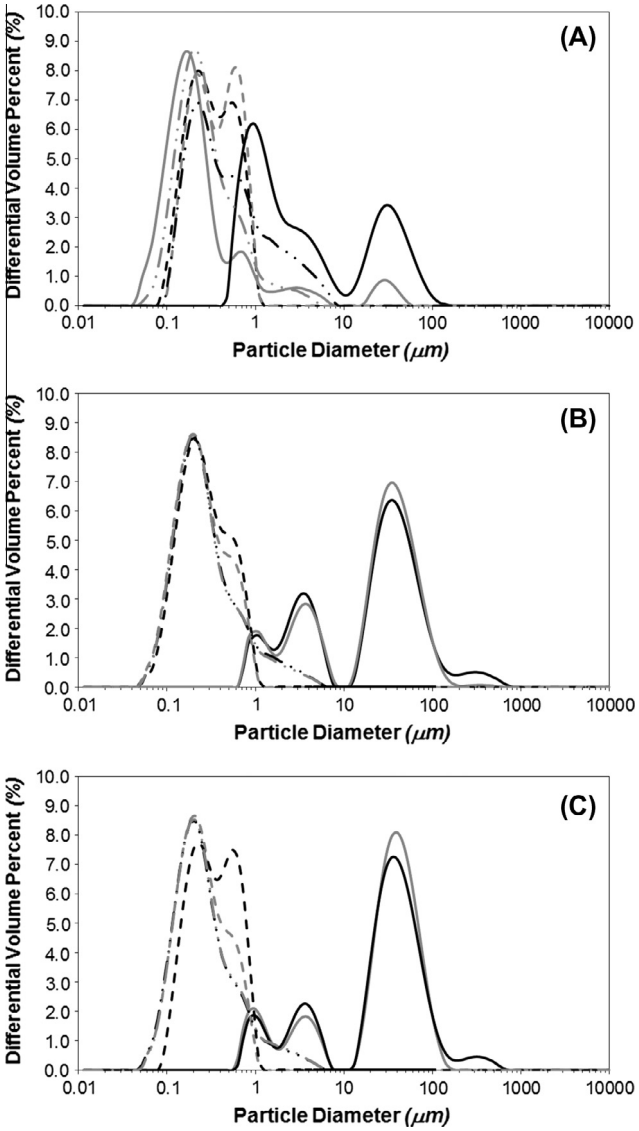

Fig. 1. Particle size distribution of reconstituted spray dried powders at (A) 0 days, (B) 60 days of storage at 23% relative humidity and (C) 60 days of storage at 76% relative humidity; (Black lines – Non Sonicated; Grey lines – Sonicated and (——— MPC; — — — WPC; ——— CaCas). The data is an average of two replicates.

2.3. Analysis

Differential Scanning Calorimetry (DSC) (Pyris 1 equipped with Intracooler II, Perkin Elmer 7, CT, USA) was used to determine the glass transition temperature of the dried powders. The purge gas used was dry nitrogen (20 ml/min). About 10–15 mg samples were scanned in hermetically sealed 50 ll DSC aluminium pans (Perkin Elmer). An empty aluminium pan was used as a reference.