Ultrasonics Sonochemistry 21 (2014) 1658–1665

Contents lists available at ScienceDirect

Ultrasonics Sonochemistry

journal homepage: www.elsevier.com/locate/ultson

Dissolution and reconstitution of casein micelle containing dairy powders by high shear using ultrasonic and physical methods

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ARTICLE INFO

Article history:
Received 18 March 2014
Received in revised form 14 April 2014
Accepted 14 April 2014
Available online 19 April 2014

Keywords:
Ultrasonication
Powder dissolution
Shear
Casein micelles
Milk protein concentrate
High pressure homogenization

ABSTRACT

The effect of shear on the solubilization of a range of dairy powders was investigated. The rate of solubilization of low solubility milk protein concentrate and micellar casein powders was examined during ultrasonication, high pressure homogenization and high-shear rotor–stator mixing and compared to low-shear overhead stirring. The high shear techniques were able to greatly accelerate the solubilization of these powders by physically breaking apart the powder agglomerates and accelerating the release of individual casein micelles into solution. This was achieved without affecting the structure of the solubilized proteins. The effect of high shear on the re-establishment of the mineral balance between the casein micelles and the serum was examined by monitoring the pH of the reconstituted skim milk powder after prior exposure to ultrasonication. Only minor differences in the re-equilibration of the pH were observed after sonication for up to 3 min, suggesting that the localized high shear forces exerted by sonication did not significantly affect the mass transfer of minerals from within the casein micelles.

1. Introduction

In the food industry, rapid dissolution (used here interchangeably with solubilization) of dairy powders is desirable to avoid prolonged processing times, increased production costs, and reduced product quality [1,2]. Important dairy powders include skim milk powder (SMP), milk protein concentrate (MPC), and micellar casein (MC) powder. It is important that the functionality of the protein within these powders is retained during drying, storage and reconstitution with water if downstream use is to be effective. Generally, the protein powder needs to be dispersed and dissolved to be fully functional and edible as an ingredient. MC and MPC have generally poor solubility, which is known to decrease during storage, particularly at high ambient temperature and humidity [4]. The insolubility of MPC is thought to be due primarily to the casein component of the powders [3], with the release of casein micelles from the powder matrix observed to occur slowly on rehydration at ambient temperatures [5,6]. According to Anema et al. (2006) the decrease in solubility of MPC85 upon storage could be accounted for entirely by the reduced solubility of the casein micelles. A possible mechanism for this decrease in solubility is the formation of a network of cross linked proteins through hydrophobic and/or hydrogen bonding at the surface of the powder acting as a barrier to water transport and subsequently inhibiting the hydration of the MPC85 particles (Anema et al., 2006). In another study microscopic images showed protein contributing to the formation of a monolayer of close-packed micelles at the surface of the particles [7]. These authors postulated the cross-linking of micelles by non-micellar proteins and by the close association of micelles at the surface. Mimouni et al. (2009) observed the rehydration of MPC powder to occur in two overlapping steps: the break-up of powder agglomerates into primary particles and the dissolution of material from the powder particles. The latter was considered to be the rate limiting step in the dissolution of MPC powder by low-shear mixing.

Some attempts have been made to produce high protein dairy powders with increased solubility through the application of static high pressure [8], high shear [9] or ultrasound [9,10] to concentrates, the addition of sodium caseinate or polydextrose [11], and the addition of mineral salts before drying [12]. In our labs, we have also observed that the use of high intensity ultrasound prior to spray drying can delay the decline in solubility of MPC powders with storage (unpublished data). Although these approaches have shown some promise, the limited solubility of most commercially available MPC and MC powders still remains an issue for their use. While MPC powder can be rapidly solubilized by increasing the

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http://dx.doi.org/10.1016/j.ultsonch.2014.04.006
1350-4177/

temperature during reconstitution (e.g. to 60 °C) [13], this is not always practical or cost-effective to implement. The use of shear to accelerate the solubilization of these powders is therefore of interest. A recent study by McCarthy et al. (2014) showed that ultrasound (20 kHz/70.2 W) increased the solubilization of MPC powders with ultrasound (20 kHz/70.2 W) [14]. However, their study involved a stirring pre-treatment step at 50 °C, which aids the dissolution of the powder particles to an extent. However, a thorough investigation of the effects of ultrasound on the dissolution of casein-containing powders at more industrially relevant temperatures is yet to be performed, and the mechanisms of solubilization using ultrasound have not been compared to other shear techniques.

Shear forces can be generated using a number of different approaches, including application of ultrasound, the use of high-shear mixers, or passage through a high pressure homogenizer. The application of ultrasound in the food industry is of interest as a gentle but targeted method for improving the quality and safety of processed foods. Acoustic cavitation generated by an ultrasonic field induces physical processes in liquids including the creation of micro jets, shear forces, shock waves and turbulence [15]. It has been shown that ultrasound can be used to effectively break apart whey aggregates present after the dissolution of whey protein powders [16,17]. It was also shown that casein-whey aggregates present in milk can be disrupted by exposure to ultrasound, but that the physico-chemical properties of the casein micelles themselves remain unaffected [18]. High pressure homogenization is an alternative approach and is an established method in the dairy industry for disrupting the fat globules in milk. In homogenization, a fluid under pressure is forced through a small valve, resulting in a large pressure gradient between the inlet and the outlet of the valve [19]. Intense shear forces are produced that have been suggested to be the primary force for decreasing the globule size [20–22]. Finally, industrial mixers and blenders are used on a wide range of liquid/liquid or liquid/solid materials in the dairy industry. This includes the use of rotor–stator and sanitary in-line mixers for reconstituting dairy powders and making condensed milk. Rotor–stator mixers are capable of generating shear forces orders of magnitude higher than overhead stirring [23] by drawing liquid axially into the dispersion head at high rotation speeds of the rotor and forcing it out radially through slots in the stator. While all of these methods can generate shear that should accelerate dairy powder dissolution processes, their effectiveness and mechanisms of solubilization have not been directly compared.

Although SMP is rapidly dissolved, it has been shown that re-equilibration of the minerals and casein micelles is only partially and slowly reversible upon subsequent reconstitution [24]. During the manufacturer of SMP soluble calcium is taken up by the casein micelles, and is only released very slowly back into the serum on solubilization of the powders in water [24]. Incomplete reversal of these alterations to the mineral balance has been shown to adversely affect the functionality of reconstituted SMP during rennet gelation [25]. Under quiescent conditions the re-equilibration processes occur over many hours, practically limiting the use of reconstituted SMP. The effect of shear on the re-equilibration of the mineral balance has not been investigated and it is possible that the increased mass transfer could accelerate this process.

This work investigates the effect of shear on powder solubilization by examining in detail the behavior of low solubility MPC and MC powders during ultrasonication, high pressure homogenization, rotor–stator mixing and low-shear overhead stirring. In addition, the effect of shear on the rate of mineral re-equilibration between the casein micelles and serum is investigated by studying the reconstitution process of dissolved SMP.

2. Materials and methods

2.1. Materials

Low heat SMP, MPC80 and MC powders were obtained from a commercial supplier (MG Nutritionals, Cobram, Victoria, Australia). The composition of the powders was determined by the manufacturer using standard methods (Table 1).

2.2. Dissolution of powders

Samples (10 g) of SMP, MPC and MC powder were added to 100 ml bottles (Schott AG, Mainz, Germany) containing 90 g of MilliQ water at 20 °C and shaken vigorously for 20 s to achieve initial dissolution of the powders. Powder solutions were then subjected to one of four different processes: low-shear overhead stirring, rotor–stator mixing, ultrasonication, or high pressure homogenization.

Low-shear overhead stirring was performed using an overhead stirrer (Ika-Werke GmbH and Co., Staufen, Germany) rotating at 1200 rpm immersed in 60 ml of powder solution. Ultrasonication was performed using a 20 kHz, 400 W Ultrasonic horn (19 mm diameter, Branson Sonifier 450, Danbury, CT) at an amplitude of 50%. The power drawn and calorimetric power were 101 W and 31 W respectively, indicating an overall energy efficiency of 31% [26]. During sonication, chilled water was continuously circulated through the cooling jacket to maintain the sample temperature at <6 °C. High pressure homogenization was performed on 500 ml samples using a PandaPLUS 1000 homogeniser (GEA Niro Savi S.p.A., Parma, Italy) in a single stage at 80 or 200 bar and a flowrate of 3.73 ml/s. The power drawn at 80 Bar was 570 W [26]. Rotor–stator mixing was provided to 60 ml of powder solution using a T25 Basic Ultra-Turrax mixer fitted with a S25N-18G-ST dispersing tool (Ika-Werke GmbH and Co., Staufen, Germany) rotated at 17,500 rpm. The power drawn was 70 W and the calorimetric power was 9.8 W, indicating an overall energy efficiency of 14% [26]. Chilled water was circulated through the cooling jacket to maintain the solution temperature at <10 °C. The energy density was calculated using Eq. (1).

$$\text{Energy density (J/ml)} = \frac{\text{power drawn (W)} \times \text{time (s)}}{\text{volume (ml)}} \quad (1)$$

According to these calculations, similar energy densities of ~153 J/ml were obtained after ~1.5 min of ultrasonication, ~2.5 min of high shear mixing and 80 bar homogenization at 3.73 ml/s respectively.

2.3. Sample analysis

The extent of powder solubilization, expressed as% (w/w) of dry powder, was assessed using the method described by [27]. In this method, the mass fraction of dissolved material was determined by comparing the amount of solids in the bulk suspension (oven dried at 102 °C for 24 h) to that remaining in filtrates (1.6 μm pore

Table 1
Composition of the powders.

Composition (% w/w)	SMP	MPC	MC
Protein	36.5	80.5	85.5
Fat	0.53	1.4	1.6
Lactose	51.2	5.6	0.6
Ash	8.7	7.3	7.3
Moisture	3.5	5.2	5.3