Journal of Food Engineering 240 (2019) 114–119

Contents lists available at ScienceDirect

Journal of Food Engineering

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

The effect of pH on the wetting and dissolution of milk protein isolate powder

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ARTICLE INFO

Keywords:
Milk protein isolate
Wettability
Dissolution
pH

ABSTRACT

Milk protein isolate (MPI) powder is an important food ingredient powder but it has poor wettability and dissolution ability which limit its functional usage. The effect of pH (5.4–8.4) on the wettability and dissolution of MPI powder is evaluated in this study. Contact angle and Washburn methods were used to measure the wetting behaviour of MPI powder. Its dissolution ability was measured by static light scattering in real-time after a premixing step. The results showed that the contact angle of droplets on compacted powder tablets exhibited no significant difference between pH conditions ($p>0.05$) and this was supported by the results of the Washburn method. It was found that MPI dissolution was strongly correlated with the appearance of submicron size casein micelles in the solution. During dissolution, higher pH caused an earlier appearance of this casein micelle submicron peak and a higher swelling peak associated with the swelling of casein micelle aggregates. This swelling of casein micelle aggregates indicates greater water penetration and loosening of the aggregate structure at higher pH, which facilitated faster dissolution of MPI powder, especially at a pH of 8.4. This study indicates changing alkaline environment could be potential to apply in MPI rehydration.

1. Introduction

High-protein milk powders have many applications, for example, in infant milk powder, beverages and emulsifiers (Crowley et al., 2016; Li et al., 2016). Milk protein isolate (MPI) powder is a category of high-protein milk powder, which is an important milk protein source (Chandan and Kilara, 2011). MPI is produced by membrane filtration of skim milk, where the ratio of casein to whey protein is kept nearly the same as that of raw milk. An obvious disadvantage of MPI powder is its poor rehydration ability due to poor wettability and slow dissolution rate because of the slow disassociation of proteins. The functionality of MPI is subsequently limited by its degree of rehydration. Consequently, the rehydration of MPI powder is of continued research interest (Cao et al., 2015; Hogekamp and Schubert, 2003).

Process of milk-protein powder rehydration and the solution conditions for powder dissolution are two main factors that influence powder rehydrating behaviours (Gaiani et al., 2005; Gaiani et al., 2006; Felix da Silva et al., 2018). The ionic environment of a solution influences the rehydrating behaviour of protein. KCl and NaCl, for instance, facilitated the dissolution of milk protein concentrate 80 powder (80% protein), which is due to the replacement of calcium in powder (Sikand et al., 2013, 2016). The pH also has an effect on the dissolution of milk protein (Felix da Silva et al., 2018). The solubility of α_{S}- and β-casein protein were compared in micellar casein, sodium caseinate and calcium caseinate from pH 1 to pH 12 (Post et al., 2012). The pH influenced the release of ions (including Ca^{2+}) from the casein proteins and consequently changed the rehydration behaviour (Crowley et al., 2014). Thus, the dissolution of high-protein powder, especially the casein-based powders, is sensitive to pH (Eshpari et al., 2014). Considering the above, research into the effect of pH on the rehydration process of MPI is of significance, but there is little published literature about the effect of solution pH on rehydration behaviour of MPI powders (Crowley et al., 2015).

In general, the rehydration of powders is divided into four steps: wetting, sinking, dispersion and dissolution (Forny et al., 2011; Richard et al., 2012). It is believed that the wetting and dissolution are prominent steps in the rehydration process (Gaiani et al., 2007). MPI displays poor wettability and dissolution compared with other milk protein powders, for example micellar casein (Ji et al., 2016b). The Washburn and contact angle methods have been used to characterize the wetting of milk protein powders (Felix da Silva et al., 2018). These two methods together can provide a comprehensive dynamic observation of powder wetting. The Washburn method shows the capillary rise of solutions into powders with varied physical properties (Hammes

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https://doi.org/10.1016/j.jfoodeng.2018.07.022
Received 9 May 2018; Received in revised form 17 July 2018; Accepted 21 July 2018
Available online 23 July 2018

et al., 2016; Hogekamp and Schubert, 2003). Tan used contact angle method to show how the chelator ethylene diamine tetraacetic acid (EDTA) and sodium citrate dihydrate (SCD) influenced the wettability of powders (Tan, 2016). Crowley et al. used contact angle method to show how the protein content in milk protein concentrate powders influences wettability (Crowley et al., 2015). However, the published works in the literature that investigate the effect of solution conditions on the wetting of milk powder by contact angle and capillary rise has not been reported.

In the dissolution of MPI powder particles, static light scattering allows characterizing the particle size of milk protein powders over time (Mimouni et al., 2009; Moughal et al., 2000). By analysing the decrease of particle size in real-time, the dissolution behaviour can subsequently be observed (Ji et al., 2016b). In previous literature on powder rehydration, the powder particle size was usually measured at a specific time interval using, for example, a Malvern Mastersizer. Real-time measuring is a more detailed method to record the process of powder dissolution (Jean-Marie et al., 2013; Moughal et al., 2000). Mimouni et al. sampled a rehydrating solution at regular time intervals and measured its particle size distribution (PSD) by the Mastersizer (Mimouni et al., 2009). The recent studies developed the method which included a pre-mixing step in a beaker prior to continually measuring the particle size over time in the Mastersizer (Jean-Marie et al., 2013; Ji et al., 2016a). The measurement vessel of the Mastersizer acted not only as a particle size measuring environment but also as a rehydrating environment. The pre-mixing step enables MPI powder to be properly wetted and dispersed prior to measurement in the Mastersizer. A longer pre-mixing time also allows for a shorter time requirement in the Mastersizer to observe the liberation of casein micelles from MPI powder particles, and this approach was applied in this study.

The objective of this paper is to investigate the influence of pH solution on the rehydration behaviour of MPI powder. To the best of our knowledge, this is the first time to study the effect of pH on the wetting of MPI powder by contact angle and Washburn methods. The developed method of static light scattering with a suitable pre-mixing step, was used to record the influence of pH on the dissolution behaviour of MPI powder in real-time.

2. Materials and methods

2.1. Materials

The MPI powder was obtained from Kerry Ingredients (Kerry, Ireland). The powder comprised of 86% milk protein, 1.5% fat, 6% ash, 5.2% moisture and less than 1% lactose. The d50, D [4, 3] and D [3, 2] of the MPI powder were 55.4 μm, 62.1 μm and 44.5 μm respectively. All other chemicals (specifically sodium hydroxide and hydrochloric acid) used were of AR (analytical reagents) grade and obtained from Sigma-Aldrich. Ultrapure distilled water was obtained from a Milli-Q apparatus (Millipore Corp.).

2.2. pH measurement

The pH of the solution was set at 5.4 and 6.4 by HCl addition (0.1 mol/L) and 7.4 and 8.4 by NaOH addition (0.1 mol/L) respectively. The pH of the solutions was measured by a pH meter (S220 Seven Compact, Mettler Toledo).

2.3. Wettability

2.3.1. Contact angle

200 mg of MPI powder was compressed into a tablet with a 2000 kg loading by a compressor (Perkin Elmer, Buckingham-shire, UK). A 12 μL solution droplet was gently dropped onto the surface of the powder tablet at room temperature of 20 °C. The contact angle measurement was performed using an optical tensiometer (Attension Theta, Biolin Scientific Ltd., Espoo, Finland).

2.3.2. Washburn method

The Washburn method measures the wettability of powder based on capillary rise (Ji et al., 2015). 2 g of powder was shaken 100 times into a cylindrical tube (stainless steel, diameter 2.4 cm) where the bottom was covered by filter paper fixed with parafilm, and the weight of whole tube was measured. The tube was slowly brought down to just above the water surface and capillary rise was allowed to occur for 10 min. The tube was then weighed again on a balance to calculate the mass of absorbed water by the MPI powder.

2.4. Dissolution by static light scattering

Static light scattering has developed into a prominent method in dairy science for measuring particle size and the particle size distribution (Jean-Marie et al., 2013; Mimouni et al., 2009). This technique provides a rapid and reproducible measurement of particle size ranging over the nanometre to millimetre range. This technique can be used to monitor the breakage and dissolution of dairy powders (Ji et al., 2016b). Consequently, a Malvern Mastersizer 3000 (Malvern Instruments Ltd, Worcestershire, UK) was used to measure the PSD in this study. It was equipped with a 4 mW He-Ne laser operating at a wavelength of 632.8 nm. The obscurity was set at around 8% in the 120 mL measuring vessel of the Mastersizer.

For poor wetting powders, it is recommended that the powder is initially pre-mixed into the solution so as to produce a dispersion prior to transfer into the particle size analyser, as illustrated in Fig. 1. A mass of 0.8 g of MPI powder was pre-mixed with 200 mL of solution using a 5 cm long magnetic stirrer rotating at 450 rpm at a temperature of 22 °C. At the end of pre-mixing the powder dispersion was transferred into the Mastersizer. The dispersion was continuously passed through a chamber with more intense agitation at 2000 rpm, as illustrated in Fig. 1. PSDs were obtained every one minute during a 50 min time duration in the Mastersizer.

2.4.1. Powder rehydration in distilled water

Initial experimental trials were conducted in distilled water to investigate the progression of the rehydration over time by varying the pre-mix time while maintaining the subsequent time of 50 min in the Mastersizer. Four pre-mix times of 30 min, 90 min, 150 min and 48 h were undertaken, with the 48 h trial being carried out at a lower temperature of 4 °C to inhibit microbial growth. The distilled water had a pH of 6.6.

2.4.2. Powder rehydration in pH-controlled dispersions

The MPI powder was added to water with pHs ranging from pH 5.4 to 8.4. Pre-mixing of the different pH dispersions in the beaker was performed for 90 min. It was found, as highlighted in the Results and Discussion, that 90 min pre-mixing followed by 50 min in the Mastersizer were suitable times for showing any differences in the

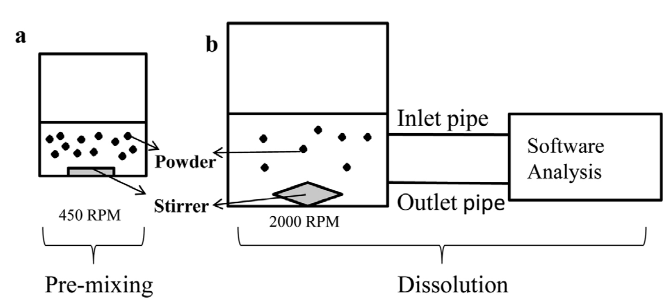

Fig. 1. Schematic illustration of dispersion and dissolution of MPI powder: (a) Pre-mixing in a beaker followed by; (b) Dissolution and particle size-measurement in Mastersizer.