



Reduction of particle size based on superfine grinding: Effects on structure, rheological and gelling properties of whey protein concentrate



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ABSTRACT

The overall objective of this study was to determine the influence of altering pH on the rheological and gelling properties of whey protein concentrate (WPC) and superfine grinding-treated whey protein concentrate (sWPC). Superfine grinding could produce a narrow and uniform particle size distribution in powders. The rheological and gelling properties of five types of protein powders with mean particles size of 48.0 μm , 27.5 μm , 21.5 μm , 11.9 μm and 8.8 μm were investigated. With the decrease of powder particle size, the lightness, gelling properties and WHC of gels at pH 4.5 were significantly improved. Thus, the operations of superfine grinding rendered WPC into an ingredient with excellent gelling properties at pH 4.5, and which can be of practical utility in production of microparticulated whey proteins. Correspondingly, the longer of superfine grinding processing time, the thinner of gels and higher of gelation temperature (from 73.5 °C to 85.6 °C) at pH 6.5.

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1. Introduction

Whey protein concentration (WPC) is a by-product of the cheese-making process. The varied functional properties of WPC (such as water solubility, water absorption, viscosity, gelation, emulsion) have generated much interest in the food industry (Ngarize et al., 2005). An important functional property of WPC is their ability, under appropriate conditions, to form heat-induced viscoelastic gels capable of immobilizing large quantities of water and other food components (Ikeda and Foegeding, 1999). The use of whey proteins in food systems as gelling and thickening agents and texture modifiers has been extensively reviewed in the literature (Morr and Ha, 1993).

However, WPC forms a gel at a moderate concentration (about 9%, w/w). A higher whey protein content in a product might increase the firmness of the product, which could result in rubbery mouth-feel and undesirable texture changes in time (hardening) (Childs et al., 2007). To circumvent these problems, modification of whey protein is proposed as an interesting solution (Purwanti et al.,

2012).

Non-thermal technologies such as superfine grinding technology are of interest to the food industry, because they do not only provide alternatives to conventional methods of thermal processing but also offer opportunities for creating new ingredients and textures.

Nano-ball-milling is the traditional equipment for the superfine grinding. Superfine grinding technology is a new technology that is a useful tool for making superfine powders with good surface properties like dispersibility and solubility (Zhu et al., 2015). To date, the superfine grinding technology is widely used in metallic materials and medicines (Song et al., 2002). Nowadays, superfine grinding technology has also been applied in biotechnology and foodstuffs and has shown a high potential for many other commercial applications (Fu et al., 2011). Nevertheless, so far little information is available on the properties of superfine grinding-treated WPC (sWPC).

Hayakawa et al. (1993) produced microparticulated protein particles (<3 μm) from egg white, casein and soybean hull by Jet Mill Grinding and found that hydrophobicity of casein increased by grinding to superfine particles. Our previous study also showed that the solubility, protein surface hydrophobicity, oil binding capacity, emulsifying properties, foaming properties and thermal

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stability of WPC are increased by superfine grinding (Sun et al., 2015b).

Rheological and gelation are very important functional properties of protein in food applications. Many factors influence the flow curve and formation of protein gels, including protein molecular weight, protein concentration, solubility, and pH (Wang et al., 2015). The investigation of rheological and gelling properties will be helpful in the applying of sWPC in food product.

The main objective of this study was to investigate the influence of superfine grinding treatment and altering pH on the structure, rheological and gelling properties of WPC. In addition, information about the modification of the rheological and gelling properties of WPC will be helpful in understanding their structural and functional properties, and also in improving the rheological and gelling properties of WPC and sWPC dispersions for further application in product development.

2. Materials and methods

2.1. Materials

Commercial WPC (whey protein concentrate) was purchased from Fonterra Commercial Trading (Shanghai) Co., Ltd. The compositions of the WPC were 80.1% protein (dry basis), 3.8% fat, 5.1% moisture 3.6% lactose monohydrate, and 3.2% starch and less than 4.0% ash. All chemicals (HCl, NaOH, sodium dihydrogen phosphate and disodium hydrogen phosphate) used in the experiments were purchased from Tianjin chemical reagent factory and of analytical grade.

2.2. Production of the superfine WPC by superfine grinding process

Superfine whey protein concentrate powders (sWPC) were ground in a multidimensional swing high-energy nano-ball-milling (CJM-SY-B Qinhuangdao Taiji Ring Nano-Products Co., Ltd., Hebei, China). In the following article, sWPC-2h stands for superfine grinding-treated for 2 h; sWPC-4h stands for superfine grinding-treated for 4 h; sWPC-6h stands for superfine grinding-treated for 6 h; sWPC-8h stands for superfine grinding-treated for 8 h.

2.2.1. Determination of particle size distribution for powders

The particle size distributions of powders (WPC and sWPC-2h, sWPC-4h, sWPC-6h and sWPC-8h) were determined by BT-2001 Laser analyzer (Dandong Bettersize Instruments Ltd. China) and air was used as dispersion medium. The Laser analyzer is based on Dynamic Light Scattering (DLS). Stokes-Einstein equation was used to calculate the particle size and distribution.

2.2.2. Scanning electron microscopy (SEM) of powders

The samples were attached to double-sided adhesive tape attached to SEM stubs, covered with a carbon layer of 10 nm and then a mix of gold by sputtering with E-1510 (Hitachi, Science Systems, Ibaraki, Japan). The samples were then examined with a SU-1510 instrument (Hitachi, Science Systems, Ibaraki, Japan) operated at 10 Kv voltage, and photomicrographs were obtained.

2.2.3. Chromaticity measurement of powders

The chromaticity of the powders was determined using a colorimeter (DC-P3 automatic colorimeter). The instrument was standardized using a white calibration plate. A CIE Lab color scale was used, and L^* (Lightness), a^* (related with the red (positive values) – green (negative values) opposition) and b^* (related with the yellow (positive values) – blue (negative values) opposition) were determined.

2.3. Fourier transform infrared chromatography

All spectra were recorded using an FT-IR spectrophotometer (IS50, Nicolet, Thermo Fisher) at a resolution of 4 cm^{-1} and an average of 20 scans from 4000 to 1000 cm^{-1} at room temperature ($25 \text{ }^\circ\text{C}$). The spectra were transformed and peaks were detected (curve-fitting algorithm) by OMNIC. In order to correct for background, spectrum of the air was recorded prior to each sample.

2.4. Flow curve analyses

Flow curve analyses were carried out using a serrated plate-and-plate geometry (25 mm diameter, 1 mm gap) on the 0.12 g/mL, pH 6.5 model dispersions using Rheometer Haake MARS-III (Thermo-electron, Karlsruhe, Germany) at a controlled temperature of $25 \text{ }^\circ\text{C}$. Shear stress against the increasing shear rates from lowest value of 0 s^{-1} – 600 s^{-1} .

The shear rates versus shear stress were interpreted using the rheometric computer program according to power law expression:

$$\tau = k\gamma^n \quad (1)$$

Where τ is the shear stress (Pa); γ is the shear rate (s^{-1}); n is the flow behavior index, and k is the consistency coefficient index (Pa s^n).

The n and k values were obtained from plots of log shear stress versus log shear rate, according to the power law equation:

$$\log\tau = \log k + n\log\gamma \quad (2)$$

Apparent viscosity (η) was calculated using Newtonian law, in addition with linear least square method for regression analysis.

$$\tau = \eta\gamma \quad (3)$$

Then we can obtain a formula like this:

$$\eta = k\gamma^{n-1} \quad (4)$$

2.5. Dynamics of gelation and viscoelasticity

Dynamic oscillation measurements were carried out using a serrated plate-and-plate geometry (25 mm diameter, 1 mm gap) on the 0.12 g/mL, pH 6.5 model dispersions using Rheometer Haake MARS-III (Thermo-electron, Karlsruhe, Germany). Simethicone was applied to the exposed surfaces of the sample to prevent evaporation. During gelation experiments, the frequency was 1 Hz and the stress was kept constant at 1 Pa, a value found to be in the linear viscoelastic region. The samples were heated from 20 to $100 \text{ }^\circ\text{C}$ at a rate of $4 \text{ }^\circ\text{C}/\text{min}$. The temperature at which the storage modulus (G') and the loss modulus (G'') crossed over was taken as the gelation temperature (T_{gel}).

2.6. Gel properties

The concentration of the protein dispersions were 0.12 g/mL, and pH was respectively adjusted to 4.5, 6.5 and 8.5 using 0.1 mol/L HCl and 0.1 mol/L NaOH solutions. The gels were prepared by keeping the dispersions at $90 \text{ }^\circ\text{C}$ for 25 min (Mishra et al., 2001) in water bath and then transferred to refrigeration temperature ($4 \text{ }^\circ\text{C}$ for 16 h).

Lightness of gels was determined using a colorimeter (DC-P3 automatic colorimeter). The instrument was standardized using a white calibration plate and black plate. A CIE Lab color scale was

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