



Preparation and characterisation of whey protein fluid gels: The effects of shear and thermal history



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ARTICLE INFO

Article history:

Received 8 July 2014

Accepted 24 November 2014

Available online 4 December 2014

Keywords:

Whey protein isolate/WPI

Fluid/shear gels

Particulate gels

Thermal history

Shear history

Rheology

ABSTRACT

With the potential use of fluid gels in structuring fluids, whey protein isolate (WPI) was used as a prerequisite to prepare fluid gels: owing to its high nutritional value and thermal stability on gelling. Fluid gels were prepared via heat-induced gelation of a 10 wt% protein solution under controlled temperature and shear. Physical properties of the resulting gel particles (e.g. size and particle–particle interactions) were found to be dependent on the combination of shear and thermal history. Discrete large aggregates ($>120\ \mu\text{m}$) were obtained at low shear, with aggregate size decreasing ($<40\ \mu\text{m}$) at higher shear. Such microstructural changes in the particles led to the control of the suspension rheology. All suspensions showed a marked shear thinning behaviour associated with particle break-up which was observed to be more apparent for larger aggregates, originally made at low shear. The viscoelastic properties of the particulate systems, once in intimate contact (e.g. high volume fraction) resembled a pseudo solid material. In addition, it was shown that at a given volume fraction, the elasticity of the suspension varied dependant on their original processing conditions, owing to the degree of particle–particle interactions. A qualitative model has therefore been presented for the formation of particles by which the rheology is determined.

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1. Introduction

Particulate gel suspensions with particles in the micron-scale have received increasing interest in recent years, especially within the food industry where they have been applied to structure fluids (Fernández Farrés, Moakes, & Norton, 2014; Sullo, Watson, & Norton, 2014). Further to this their textural role as fat mimetics has been recently proposed, as the presence of small, soft and smooth particles lowers the friction between oral surfaces providing enhanced fat perception (Fernández Farrés, Douaire, & Norton, 2013). Additionally, it was shown that the rheology of full fat mayonnaise could be simulated by replacing the majority of oil droplet content with spherical agar gel particles (J. E. Norton & Norton, 2010).

An essential feature regarding their application is the novel rheological properties of fluid gels, since they behave in a solid-like fashion at rest, but flow above a critical value of applied stress (I. T. Norton, Jarvis, & Foster, 1999). The rheological properties can be finely controlled to meet requirements for desired applications by

varying the composition (e.g. polysaccharide, protein), cross-link density (e.g. salt, concentration), particle size and/or degree of particle–particle interactions. Both particle size and degree of interactions are directly related to the processing conditions used in production (Gabriele, Spyropoulos, & Norton, 2009; Garrec & Norton, 2012).

Fluid gels are formed when a separation process is applied to a biopolymer system undergoing its sol–gel transition (I. T. Norton et al., 1999). Amongst all the existing methods for the preparation of gel particles (e.g. emulsion route or biopolymer mixtures), shear induced fluid gels are typically more attractive due to relatively easy modulation of resulting properties, by varying two parameters: shear and thermal history. On the other hand, particles produced via a phase separation route (e.g. emulsion route) require additional steps; due to the transfer of produced particles into an aqueous phase, lowering efficiency.

The mechanism proposed for the formation of shear gels is based primarily on nucleation and growth (I. T. Norton et al., 1999). It has been proposed that thermodynamically driven spherical gel particles form mainly through growth of the initial gel nuclei via enrichment, when the applied shear is comparable to the time taken for polymer to diffuse through the system and order (Garrec, Guthrie, & Norton, 2013). Thus the rate of ordering is key to particle

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growth. Rapid ordering forces particle growth through particle–particle structuring; this causes particles to rapidly reach sizes that become affected by the applied shear forces. Hence particle size becomes determined by the applied mechanical break-up and large (>100 μm) anisotropic particles are formed (Gabriele, Spyropoulos, & Norton, 2010).

Such changes in the microstructure led to varying flow properties, which fall in between the behaviour of colloidal particles and polymeric gels (Altmann, Cooper-White, Dunstan, & Stokes, 2004; Gabriele et al., 2010; Garrec et al., 2013). At low volume fractions ($\phi \sim 0.2$) polysaccharide fluid gels showed high viscosities and strong shear thinning behaviour as expected for highly aggregated suspensions, mainly dominated by colloidal forces. The prevention of a continuous gel network in this way leads to incomplete gelation at the particle surface. As a result, significant particle–particle interactions can occur, reported to give rise to both yield stresses and subsequent shear thinning behaviour (Berli & Quemada, 2000; Garrec et al., 2013).

At high phase volumes where particles are in contact and so their movement becomes sterically confined, the fluid gels behave as a suspension of soft particles with rheology dependant on particle elastic modulus as well as particle–particle interactions (Fernández Farrés et al., 2013; Gabriele et al., 2009). The deformability of such soft gel particles, particularly at higher concentrations has been suggested as an explanation for effective volumes exceeding those characteristic of hard sphere suspensions (Adams, Frith, & Stokes, 2004).

Microparticulated whey protein (MWP), as a result of thermal/shear treatment has been shown to change surface and colloidal properties (Dissanayake & Vasiljevic, 2009). Such particulates have modified foaming and emulsifying activity, however further heating formed gels with much lower gelling temperatures and significantly higher complex moduli (Dissanayake, Kelly, & Vasiljevic, 2010; McClements & Keogh, 1995; Nicolai, Britten, & Schmitt, 2011). Continued processing of the MWP also enhanced flow characteristics through increased uptake of the continuous phase, as aggregate size increased (Chung, Degner, & McClements, 2014). Such increased structural properties have been shown to augment textural and sensory perceptions of dairy products (Meza, Verdini, & Rubiolo, 2010; Torres, Janhøj, Mikkelsen, & Ipsen, 2011).

Fat replacers based on biopolymer particles with controlled size and shape are already commercially available. *Simplesse*[®] is a system of microparticulated, denatured protein spheres between 0.1 and 3 μm in diameter; owing to their size and morphology the protein are able to replicate organoleptic mouth feel. The protein particles are formed using a rapid heating process (up to 120 °C) over a short period of time to denature the whey protein. Reducing the pH of the system below the isoelectric point increasing electrostatic repulsion, aided by aggregate blocking agents (i.e. lecithin) and high shear, led to an incomplete aggregation process resulting in discrete particles (Singer, Yamamoto, & Latella, 1988).

From an application point of view, coupled with the highly controllable rheological properties, thermal stability and formulation simplicity are equally important within industry: owing to many potential thermal processes and cost effectiveness within product production. This study therefore investigates the formation of thermally stable whey protein aggregates by applying shear gel technology to a gelling system. The research uses whey protein isolate (WPI), owing to its irreversible heat induced gelation, as a potential candidate for the preparation of fluid gels without additives. Therefore, the production of WPI fluid gels at native pH are investigated, with emphasis to the effect of shear and thermal history on particle intrinsic properties, and corresponding rheological properties thereafter.

2. Materials and methods

2.1. Materials

Whey protein isolate (WPI) (WPI, W994, S-493391) was obtained from Kerry Ingredients (Listowel, Ireland). The composition of the WPI as stated by the supplier was 91.0% protein, moisture 4.0%, fat 1.0%, ash 3.5% and lactose 0.5%. Mineral content of the WPI was: Ca – 0.50, P – 0.65, Na – 0.10, K – 0.15, Mg – 0.02 and Cl – 0.02%.

2.2. Preparation of stock solutions

Whey protein stock solutions of 10 wt% were prepared by dispersing WPI powder in deionised water. Sodium Azide (0.02 wt %) was added to the solution to prevent bacterial growth. Solutions were then stirred for two hours at ambient temperature until completely dispersed and stored at 5 °C until usage (pH remained unadjusted ca. pH 6.4).

2.3. Fluid gel preparation using a rheometer

A Malvern Kinexus Pro, stress controlled rheometer (Malvern Instruments Ltd, UK) equipped with a couette measuring system, cup diameter 27 mm and vane diameter 25 mm was used to prepare all WPI fluid gels. Aliquots of WPI solution were transferred using a syringe to the rheometer cup set to a temperature of 25 °C. The geometry was then lowered and sample temperature left to equilibrate for 2 min before shear-heating profiles were undertaken. Heating profiles starting at 25 °C and increasing to 80 °C with a 10 min holding period were conducted at various heating rates (1, 3, 5 and 10 °C min^{-1}) under steady shear (200, 400, 600 and 800 s^{-1}) before quiescent cooling at 3 °C min^{-1} to 20 °C. In all experiments a thermal cover was placed over the system to help prevent water loss. Samples were then removed from the geometry and stored at 5 °C for 24 h before being prepared into known volume fractions and further tests carried out (as described in Section 2.7). Each sample was prepared in triplicate with apparent viscosity profiles showing the average of at least three repeats.

2.4. Static light scattering (SLS)

Particle size distributions were determined using static light scattering. A Malvern Mastersizer MS2000 (Malvern Instruments Ltd, UK) attached to a Hydro SM manual small volume sample dispersion unit was used to obtain both surface and volume weighted means, $d_{3,2}$ and $d_{4,3}$ respectfully. Particle sizes are calculated based on the Mie theory as such particles are assumed to be monodisperse homogeneous spheres. Samples were prepared by diluting gel particles in distilled water (RI = 1.33) to avoid multiple scattering. Average distributions based on 3 runs were then taken in triplicate.

2.5. Optical light microscopy

An optical light microscope, Brunel SP300-fl (Brunel Microscopes Ltd, UK) fitted with an SLR camera (Canon EOS Rebel XS, DS126 191) was used to image fluid gel particles with objective lenses up to 40 \times magnification (5 \times , 10 \times , 20 \times and 40 \times). Slides were prepared by firstly diluting fluid gel samples (1:4), then adding one drop onto a microscope slide (VWR, UK) and covered with a coverslip (Thickness No.1, VWR, UK).

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