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Effect of thermomechanical treatment on microstructure of guar gum/acid milk gels

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ABSTRACT

The influence of thermal and shear conditions on the final microstructure and rheological properties of guar gum/acid milk gels was investigated. In an immiscible guar gum/milk mixture, gelation can kinetically trap the sedimenting protein-rich droplets. In order to modify the phase separation and gelation kinetics, acidification procedure, shear and thermal histories were modified. The properties of the set and stirred mixed gels were then discussed in light of the processing conditions applied and the relative phase separation and gelation times. Application of various thermal stages showed an increase in the sediment layer and a modification of the morphology of the micro-gels when delaying both the phase separation and gelation mechanisms. Moreover, a substantial reduction of the phase separation could be obtained by applying a continuous shear until different stages of acidification. The results demonstrated that the size and shape of the micro-gels depended not only on the time interval between phase separation and gelation, but also on thermal and shearing conditions.

1. Introduction

In most industrial processes, food systems are subjected to thermal treatment and shearing. This is particularly the case of dairy products for which the effects of technological process steps is of crucial importance in the final texture (Lee and Lucey, 2004; Xu et al., 2008; Béal et al., 1999).

During the production of acid milk gels, casein micelle destabilization and aggregation lead to the formation of a three dimensional network structure (Heertje et al., 1985). Then, the casein strands can be broken apart by shearing, providing stirred acid milk gels, and the size of the aggregates decreases as the rate of shearing increases (Tamine and Robinson, 2007; Van Marle et al., 1999; Afonso and Maia, 1999). Temperature and pH are the two major parameters that affect acid milk coagulation. It is well established that heat treatment of the milk affects rheological properties of the gel and the gelation kinetics (Lucey et al., 1999). The pH kinetics decrease can be controlled by GDL addition whose progressive hydrolysis is strongly temperature-dependant (Heertje et al., 1985; Lucey et al., 1997b; Lee and Lucey, 2004) and mimics the acidification kinetics obtained by a bacterial culture. In the case of bacterial culture, bacteria growth needs an adapting lag phase, depending on the strain, which is absent when using GDL (Lucey et al., 1998).

Processed food systems such as mixed acid milk gels usually contain proteins and polysaccharides performing both nutritional and structural functions (Tolstoguzov, 1995). Whereas the effects of temperature and shearing processes on aggregation and gelation of casein micelles as a result of acidification have frequently been studied (Lee and Lucey, 2004), the presence of polysaccharides during the acid-induced gelation leads to another degree of complexity in milk protein systems.

Actually, milk protein and polysaccharide such as guar gum are usually incompatible due to a depletion-flocculation phenomenon (Bourriot et al., 1999; Tuinier et al., 2000). These demixed biopolymer solutions have emulsion-like properties and can be treated as 'water-in-water' emulsions (Schorsch et al., 1999; Tolstoguzov, 2003). Under non-equilibrium conditions, droplets behave as conventional emulsions and can evolve through coalescence and sedimentation (Foster et al., 1997). However, it has been shown that when one of the biopolymer is able to form a gel, it freezes the phase separation process at a specific point (Turgeon et al., 2003). This phenomenon has been well illustrated using acid-induced gelation of milk proteins (Rohart et al., 2014), heat-

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treated whey proteins (De Jong et al., 2009; Syrbe et al., 1998) or cold setting gels of carrageenan and gelatin (Norton and Frith, 2001; Wolf et al., 2000; Lorén et al., 1999; Garnier et al., 2003).

Although kinetic aspects are very important when considering immiscible mixtures, the flow and thermal histories have a great influence on the properties of final system (Turgeon et al., 2003). Considering a mixture of gelatin and guar, previous studies have shown that under specific conditions of applied shear and controlled gelatin gelation temperature, it was possible to use gelation to trap the dispersed phase in an anisotropic morphology (Wolf et al., 2002; Wolf et al., 2000). Nevertheless, the effect of the application of flow fields with high shear rates and particularly during the acidification of milk proteins on gels properties has reveived less attention.

The objective of this study was to model the pH kinetics obtained using bacterial culture by using GDL and playing on thermal history and to design a technological pathway to add fibre in an acid milk gel avoiding macroscopic phase separation. The effects of thermal and mechanical treatments on the properties of stirred guar gum/acid milk gels were investigated using the knowledge about phase separating systems in water-in-water emulsions. The gel structures were characterized based on macroscopic behaviour, microstructure and rheological properties. The results were discussed in light of the understanding of the phase separation and gelation kinetics as affected by the thermal and shear rate history.

2. Materials and methods

2.1. Preparation of the mixed gels

Skim milk with 46 g kg^{-1} protein was reconstituted from low-heat skim milk powder (CH low heat, Ingredia, Arras, France) dispersed in Milli-Q water under continuous stirring for 1 h at room temperature. To ensure complete hydration, skim milk was kept overnight at 4 °C. Then the milk was heat-treated in a thermostatically controlled water bath at 80 °C for 7 min, and cooled to 60 °C for dry powdered guar gum addition (Viscogum, Cargill, USA). The mixture was stirred 30 min at 60 °C and finally cooled down to 8 °C or 43 °C in a cold water bath. Guar gum concentrations of 0.25 and 0.3 wt% were studied.

Gelation of the mixed systems was induced by the addition of glucono-δ-lactone (GDL) (Sigma Chemicals, St Louis, MO, USA) or using starter culture. The pH was measured in the meantime with a pH-meter Consort D130 multiparameter analyser (Turnhout, Belgium).

For GDL-induced acidification, 1.5 wt% GDL was added to the milk at 22 °C under stirring during 1 min before rapid cooling to 8 °C in an ice bath. Thermal stages were applied to the milk during acidification in order to modulate the kinetics of the pH decrease of the milk. For bacterial fermentation, a mother culture was prepared by adding 5 g of freeze-dried culture (mixture of *Lactobacillus delbrueckii* subsp. *bulgaricus* and *Streptococcus thermophilus*) to 10 mL sterilized reconstituted skim milk. Preheated milk was inoculated at 43 °C and kept at this temperature for 5 h and during fermentation, a continuous stirring at 500 rpm using a blade was performed until a desired pH value for some samples (it will be mentioned every time it was the case).

The acidification was carried out until the samples achieved a pH of 4.6 (±0.05). Set acid milk gels were made in plastic cylinders (30 mm diameter × 65 mm height) and they were immediately cooled down in cold water bath to 20 °C in order to slow down drastically the pH decrease (less than 0.1 pH unit between ending acidification process and stirred gel analysis).

For stirred acid milk gels preparation, the gels were broken and stirred by forcing the gels with a Masterflex® peristaltic pump (Cole Parmer, Vernon Hills, USA) at 380 mL min^{-1} through two successive plastic pipes (length: 40 cm and internal diameter: 7 mm; length: 100 cm and internal diameter: 3 mm) with a mesh of 500 μm holes at the end. Gels containing large particles were stirred in the two pipes to obtain a homogeneous stirred gel.

2.2. Instrumental characterization

The phase separation process was observed by a light backscattering technique using Turbiscan LAB® (Formulaction, France). Turbiscan LAB® apparatus was equilibrated at room temperature or at 43 °C before measurement to follow phase separation at 8 °C or 43 °C, respectively. A non-acidified guar gum/skim milk mixture was poured in glass tubes (diameter 27.5 mm) and was maintained at 8 °C in a temperature controlled water bath or it was immediately placed in the apparatus at 43 °C. Backscattering values at 880 nm were acquired along a 40 mm height of tube with one acquisition every 40 μm. Measurements were performed every minute.

Epifluorescence and optical microscopies were used to visualize the microstructure of the stirred mixed gels using an Olympus BX51 microscope (Olympus America, Lake Success, NY). For epifluorescence microscopy, proteins were stained by addition of Nile Red (25 μL in 1 g of sample). Then the sample was placed on a microscope slide with a glass coverslip on the surface. All the observations were carried out 1 day after mixed gels manufacture.

Rheological measurements of stirred acid milk gels were performed using a stress-controlled rheometer (Carri-Med CSL^2 100, TA Instruments, UK) fitted with a stainless steel plate and cone (diameter 60 mm, 4° angle) geometry. Frequency sweep tests were performed in the range of 0.01–10 Hz within the linear domain (maximum shear stress of 1 Pa). All rheological measurements were carried out 1 day after stirred acid milk gels manufacture. Repeated measurements were performed at least twice.

3. Results and discussion

3.1. Kinetics of phase separation and gelation

When studying polysaccharides/acid milk gels, it is important to characterize the two processes that are likely to occur, *i.e.* phase separation and gelation.

3.1.1. Phase separation of guar gum/milk mixtures

Guar gum/milk systems were investigated prior to acidification. Exceeding a given polysaccharide concentration, the phase-separated biopolymer solutions have emulsion-like properties and can be treated as 'water-in-water emulsions' (Foster et al., 1997). In these systems, a macroscopic phase separation has been reported (Bourriot et al., 1999; Rohart et al., 2014), which in general lead to the formation of two phases, one top phase enriched in polysaccharide while the bottom phase contained mainly proteins. Due to its non-thermodynamic equilibrium, this process is highly dependent on experimental time. Therefore, it is of high interest to study the phase separation kinetics of a guar gum/skim milk mixture.

Fig. 1a shows the variation of Δ Backscattering for a non-acidified 0.3 wt% guar gum/milk mixture at 43 °C over different times along the height of the sample. Compared to the control corresponding to time 0 h, a change in Δ Backscattering intensity profiles appeared from the first minutes of measurement. Then after 0.5 h, the Δ Backscattering level increased drastically at the bottom of the tube (left part of the graph) while it decreased at the top (right part of the graph). At longer time scales, the Δ Backscattering level remained constant at the bottom whereas the