



Low acyl gellan gum fluid gel formation and their subsequent response with acid to impact on satiety



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ABSTRACT

Fluid gels have shown potential for use in numerous applications including foods. One such application is in the production of self-structuring food formulations that take advantage of natural digestive processes to increase satiety, potentially helping to combat obesity. The formation and properties of low-acyl gellan gum fluid gels, produced by applying shear during the gelation process are discussed. The acid gelation process of the low-acyl gellan gum fluid gels was investigated through the direct addition of hydrochloric acid, inducing a range of pH environments and also their response to a prolonged exposure to an acidic environment, similar to the conditions found in the stomach. Quiescent LA gellan gum gels were also exposed in this way for comparison to the fluid gels.

Rheology was performed on the fluid gels after their formation to determine structure development. Using these methods, the influence of applied shear and acid concentration on the transition temperature, viscosity and molecular ordering in the fluid gel systems has been studied.

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1. Introduction

Polysaccharides are widely used as replacements for fats and sugars in low fat and reduced calorie foods or within enhanced satiety products (Brown, Cutler, & Norton, 1996; Garrec, Fransch-Melnik, Henry, Spyropoulos, & Norton, 2012; Norton, Frith, & Ablett, 2006; Tang, Lelievre, Tung, & Zeng, 1994). The use of hydrocolloids only as simple gelling and thickening agents does not offer any specific benefit to health, convenience or quality in a formulated product (Gabriele, Spyropoulos, & Norton, 2009). This has led to the development of hydrocolloid fluid gels, which can be designed to achieve a wide range of structural properties (Cassin, Appelqvist, Normand, & Norton, 2000; Norton, Smith, Frith, & Foster, 2000) to impart specific advantages to formulated foods.

Fluid gels are formed when shear stress is applied to a solution undergoing gelation; as a result particles are formed within the solution rather than the entire solution forming a homogenous gel. These particles of gel exist as a highly concentrated (high volume fraction) suspension, giving rise to an entirely different set of material properties to the quiescently formed gel (Brown et al., 1996). The application of fluid gels in food formulations has been investigated, and many studies manipulate the ability of some

polysaccharides to form solid, brittle gels under quiescent cooling. One such polysaccharide is Gellan. Gellan gum (Caggioni, Spicer, Blair, Lindberg, & Weitz, 2007; Sworn, 2000, 2009; Sworn, Sanderson, & Gibson, 1995; Valli & Miskiel, 2001) is a multifunctional hydrocolloid which is already available for use in a wide variety of food products as a gelling and stabilising agent (Gibson, 1992; Kelco Division of Merck and Co, 1993; Sanderson, 1990). The gellan polymer consists of monosaccharides b-D-glucose, b-D-glucuronic acid and α -L-rhamnose in molar ratios of 2:1:1 (Sanderson, 1990) linked together to form a primary linear structure. The biopolymer is produced with two acyl substituents present on the 3-linked glucose, L-glycerol positioned at O(2) and acetyl at O(6). Direct recovery of the polysaccharide from the fermentation broth yields the high acyl form whereas deacylation by alkali treatment results in a low acyl counterpart. Gellan gum is currently commercially available in both the high acyl and the low acyl form. When hot solutions of gellan gum are cooled in the presence of gel-promoting cations, gels ranging in texture from brittle to elastic are formed, principally through cation-mediated helix–helix aggregation (Gibson & Sanderson, 1997). This paper reports results obtained with low acyl gellan gum since it forms clear gels, while high acyl gellan yields opaque gels with less thermal stability. It is important to note however that clarity is not a natural attribute of low acyl gellan gum, it is the result of an additional clarification process that is not applied to the high acyl gellan gum commercial product. The gel phase transition

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temperatures of low acyl gellan (30–50 °C) are lower than those of high acyl gellan gum (70–80 °C), which also makes the formation of fluid gels less complex.

To form low acyl gellan gum fluid gels with deionised water, four steps must be followed: (a) dispersion, (b) hydration, (c) addition of either salt or acid and (d) the cooling process, which leads to a quick set as soon as the gelling temperature is reached (Valli and Clark, 2010). The traditional mechanism proposed for the sol–gel transition of gellan gum is based on a random coil to double helix transition followed by helix to helix aggregation, which involves weak interactions such as Van der Waals forces and Hydrogen bonds. Gel-promoting ions reduce the effect of electrostatic repulsions among helices, augmenting the development of a network. In addition, ionic bridges among carboxylic groups of neighbouring chains have been reported to occur when divalent cations are used (Nickerson, Paulson, & Speers, 2003; Tang, Tung, & Zeng, 1997). The properties of gellan gum gels, such as texture, setting temperature and melting temperature, are dependent upon the types of ions present and their concentrations (Sanderson, Bell, Clark, & Ortega, 1988).

The ordering of helices in the production of gellan gum fluid gels is restricted by the shear force applied to small volumes during the cooling step (Valli & Miskiel, 2001), the greater the shear, the smaller the volumes in which ordering can occur, the smaller the resultant fluid gel particles. Weak gels with a similar consistency to fluid gels have also been produced, by allowing gelation to occur in quiescent conditions, provided specific gellan and gel-promoting ion concentrations are used (Rodríguez-Hernández, Durand, Garnier, Tecante, & Doublier, 2003; Sworn et al., 1995). These are structurally different however, as Rodríguez-Hernández et al. (2003) visualised the heterogeneous microstructure of Na-low acyl gellan gum weak gels by confocal scanning laser microscopy (CSLM), showing a network structure rather than the particulate structure of a fluid gel.

Within the food industry, gellan gum fluid gels have numerous applications as suspension and emulsion stabilisers, working at lower concentrations than standard thickeners (García, Alfaro, Calero, & Muñoz, 2011). They also find uses in paint formulations, ophthalmic drug delivery systems (Sultana, Aqil, & Ali, 2006; Suri and Banerjee, 2006), and in medicine for the preparation of artificial lacrimal fluids (García et al., 2011). Gellan gum fluid gels exhibit highly pseudoplastic flow, which provides stable suspension of large included particles combined with low viscosity at higher rates of shear. When used in foods, this results in low viscosity in the mouth, so they can be used to great effect in beverages because of the low viscosity under shear, whilst still allowing for suspension of fruit pulp or jelly pieces. This suspension can be achieved without adversely affecting mouthfeel.

In this work, we aim to investigate the influence of processing conditions during low acyl gellan gum fluid gel formation using two common production methods, and determine how an acid environment affects the structural properties of the resultant fluid gels. This will enable improved understanding of their structure and material responses for their potential application in self-structuring satiety improving food products.

2. Materials and methods

2.1. Sample preparation

Low acyl (LA) gellan gum (Kelcogel F, CPKelco, UK) was used to prepare the model acid-sensitive hydrocolloid system in this study, with no further purification or modification to its properties. The water used for the prepared hydrocolloid solutions was passed through a reverse osmosis unit and then a milli-Q water system.

Hydrochloric acid was purchased from Fisher Scientific (Loughborough, UK) and was used for the acid bath soaks and direct acidification of the fluid gels.

2.2. Fluid gel production

Fluid gel production and rheological measurements (both during and after fluid gel production) were carried out in a Gemini HR nano stress-controlled rheometer using a 4° truncated (150 μm) cone (40 mm in diameter) and plate geometry. This approach allows for the precise control of the temperature, rate of cooling and the applied rate of shear, which determine the structure of the produced fluid gels. Solutions of 2 wt. % LA gellan gum were transferred via pipette to the rheometer plate at 90 °C. The sample temperature was allowed to reach equilibrium (2 min) after the geometry was lowered and excess material removed. All experiments were carried out using a thin film of silicon oil, around the outer edge of the geometry, to control evaporation. Cooling was then performed whilst a constant shear between 50 and 1000 s⁻¹ was applied, with a cooling at a rate of 3 °C/min. By conducting these sheared cooling profiles with LA gellan gum solutions, fluid gels are formed and their viscosities during formation can be measured. Note that due to the cooling constraints of the rheometer equipment, the maximum cooling rate possible is 6 °C/min.

A pin-stirrer was used to produce the LA gellan gum fluid gels on a larger scale than the rheometer method. The pin-stirrer consists of a rotating shaft (13 mm diameter) with 8 pairs of pins evenly distributed along its length, which is inserted into a jacketed vessel with 8 pairs of stator pins along the length of the inside wall. The rotating pins pass between the stator pins as they rotate, increasing the anisotropy of the flow field. The average shear rate within the pin stirrer apparatus has been shown to be about 50 s⁻¹ with these process conditions (Gabriele, 2011). The hot LA gellan gum solution was fed into the pin-stirrer. The internal pin-stirrer volume (with shaft inserted) was approximately 140 ml. The temperature of the fluid entering (T_{in}) and exiting (T_{exit}) the pin-stirrer were recorded using thermocouples, and T_{exit} was controlled via a recirculating water bath that ran through the cooling jacket of the pin-stirrer. Silicon tubing was used to connect all of the units within the process, and material flow was induced via a peristaltic pump (Masterflex® L/S®). A shaft rotation speed of 1500 rpm was used, with the flow rate maintained at 100 ml/min. Samples were then collected, refrigerated (3 °C) and stored for at least 24 h, before testing, to allow post processing ripening effects to fully take place (Gabriele et al., 2009).

2.3. Direct and post-production exposure to acidic environment

Both LA gellan gum fluid and acid fluid gels were assessed post-production in terms of their response to a prolonged exposure to an acidic environment that simulates the conditions found in the stomach during digestion. Quiescent LA gellan gum gels were also exposed in this way for comparison. The fluid gels (1 wt. %) were produced using the jacketed pin-stirrer and were placed into visking dialysis tubing (23 mm ID, 14 kDa pore size, Medicell International, Ltd.) immediately after production, which was then allowed to soak in a 0.5% HCl acid solution (corresponding to ~pH 1) for 24 h. The acid soaking time of 24 h was hugely exaggerated to take into consideration exposure extremities and the possible effects of chemical breakdown. Texture analysis of these systems was then performed to assess their structural properties. Acid exposure (after approximately 1 h of soaking) caused the gel strength of the samples to increase and for them to change state from a fluid to a solid. The strengths of the solid gel samples formed enabled compression testing to be performed. After 24 h of soaking no

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