



## Gums tuning the rheological properties of modified maize starch pastes: Differences between guar and xanthan



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### ABSTRACT

The experimental setup aimed at gaining a more fundamental knowledge on the effects of two frequently used food gums, guar and xanthan, on the rheological properties of modified waxy starch systems. With regard to starch/non-starch hydrocolloid combinations, chemically modified starches receive much less attention than their native counterparts, despite their commercial and scientific value. The use of cross-linked waxy starches allows to treat the combined systems as dispersions of undisturbed, swollen granules, surrounded by the gum solution. This hypothesis was verified by using confocal scanning laser microscopy with fluorescently labeled gums. The average swollen granule sizes were derived by laser light scattering, and the corresponding total volume fraction occupied by the swollen starch ( $\Phi$ ) was calculated. The specific effects of the gums appeared to strongly depend on the starch content. Both gums, which were effectively increased in concentration due to the starch swelling, dominated the dynamic rheological properties and flow behavior at low  $\Phi$ . At higher  $\Phi$ , granule interactions governed the rheological properties. Whereas both gums could weaken the granule network, they contributed to the elastic behavior by their own entanglements. Moreover, xanthan gum appeared capable of reducing the granule interactions within flow, which results in lower viscosities at high starch volume fractions and high shear rates.

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### 1. Introduction

In nature, starch occurs as granules in cereals (common and durum wheat, maize, rice and rye), in roots and tubers like potato and cassava, and in legumes such as peas. Starch comprises two different glucose polymers: amylose and amylopectin. Amylose is a linear polymer of D-glucopyranose units which have  $\alpha$ -(1,4) linkages. Amylopectin has the same backbone but it is highly branched. On the branching points D-glucopyranose has an  $\alpha$ -(1,6) linkage. The starch granule consists of alternating amorphous and semi-crystalline growth rings. The amorphous shells are less dense and contain amylose and less ordered amylopectin, whereas the semi-crystalline

shells have alternating amorphous and crystalline lamellae (BeMiller & Whistler, 2009; Buleon, Colonna, Planchot, & Ball, 1998; Tester, Karkalas, & Qi, 2004). When heated above the gelatinization temperature in the presence of excess water, the starch granules lose their internal order and absorb the water. This causes a swelling of the granules and a rise of the viscosity. The resulting system is generally denoted as a 'paste'. For non-waxy starches a significant amount of amylose leaches out of the granules (BeMiller & Whistler, 2009; Hermansson & Svegmarm, 1996). For waxy starches – which are essentially free of amylose – amylopectin molecules remain initially inside the granules during swelling, however upon prolonged heating, the majority of the granules breaks up, and the system is converted to a macromolecular amylopectin solution (Schirmer, Höchstötter, Jekle, Arendt, & Becker, 2013). Chemically cross-linked starches are commercially very popular, because the strengthened granules can much better withstand the elevated temperatures and high shear forces encountered in production processes (Singh, Kaur, & McCarthy, 2007; Tharanathan, 2005). For numerous industrial food applications chemically modified starches

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and non-starch hydrocolloids or gums are combined in order to improve their sensory and rheological properties as well as their physicochemical stability. Already at low concentrations, gums are known to significantly increase the viscosity of starch-based systems (BeMiller, 2011). This synergy is largely responsible for their commercial success. When the granules remain intact, mixed starch/gum pastes are generally regarded as discontinuous systems: the swollen starch granules are surrounded by the continuous watery phase containing the dissolved gums (Abdulmola, Hember, Richardson, & Morris, 1996; Alloncle & Doublier, 1991). Due to the water absorption of the starch, the effective concentration of the polysaccharides present in the external phase is raised. The latter is believed to cause the often observed synergistic effects in the rheological behavior of starch/gum systems. When amylose leaching takes place in the case of non-waxy starches, or when granules disintegrate during heating, starch polymers could interact with the gum molecules in the continuous phase, and the microstructural interpretation of the rheological behavior becomes more complicated (BeMiller, 2011; Christianson, Hodge, Osborne, & Detroy, 1981; Shi & BeMiller, 2002).

When studying starch/gum combinations, the effects of guar gum and xanthan are frequently compared because both are high molecular weight polymers that induce high viscosities in solution. Molecularly and structurally both polysaccharides strongly differ. Guar gum is derived from the ground endosperm of guar seeds (*Cyamopsis tetragonoloba*). The molecular weight is generally in the order of  $10^6$  g/mol. This non-ionic polysaccharide has a main chain of (1 → 4)-linked  $\beta$ -D-mannopyranosyl units with (1 → 6)-linked  $\alpha$ -D-galactopyranosyl side groups (Imeson, 2010). Xanthan gum is an anionic polysaccharide, produced by fermentation of the bacterium *Xanthomonas campestris*. The molecule consists of (1 → 4)-linked  $\beta$ -D-glucose backbone. The trisaccharide side chains are (3 → 1)- $\alpha$ -linked D-mannopyranose, (4 → 1)- $\beta$ -D-mannopyranose and (2 → 1)- $\beta$ -D-glucuronic acid (Born, Langendorff, & Boulenguer, 2005; Imeson, 2010). The molecular weight of commercial xanthan gum is usually in the range of  $10^6$  g/mol. In the presence of salts, and at low temperatures, xanthan is known to exist in a double helical conformation (Sato, Norisuye, & Fujita, 1984). Upon heating above the transition temperature  $T_m$ , xanthan loses its helical conformation and converts to a random coil. This transition temperature depends on the salt content, the molecular weight and the acetyl and pyruvate content of the molecules (Capron, Brigand, & Muller, 1998; Liu, Sato, Norisuye, & Fujita, 1987; Smith, Symes, Lawson, & Morris, 1981). When the denatured xanthan molecules are cooled, the helical conformation is restored. At low xanthan concentrations and in the presence of salt, this renaturation is believed to be an intramolecular process. At higher xanthan concentrations, intermolecular interactions can be established as well (Matsuda, Biyajima, & Sato, 2009). Due to these phenomena, the rheological properties of xanthan solutions are strongly affected by their thermal history (Capron et al., 1998; Rochefort & Middleman, 1987). Xanthan gum exhibits very unique rheological behavior. Its rigid nature on the one hand and its ability to engage in non-permanent intermolecular associations on the other hand, result in weak gel solutions with unusually strong shear thinning behavior (Choppe, Puaud, Nicolai, & Benyahia, 2010; Cuvelier & Launay, 1986). Its rheological behavior depends strongly on the applied concentration and the corresponding molecular organization. The critical overlap concentration  $c^*$  marks the start of the semidilute regime. Above the entanglement concentration  $c^{**}$ , molecules start to engage in intermolecular interactions (Meyer, Fuller, Clark, & Kulicke, 1993; Wyatt & Liberatore, 2009). At more elevated concentrations ( $\sim 2\%$  w/v), anisotropic phases are observed due to the parallel alignment of the stiff molecules, and the rheological properties depend less strongly on the xanthan

concentration (Allain, Lecourtier, & Chauveteau, 1988; Lee & Brant, 2002; Oertel & Kulicke, 1991; Sato, Kakihara, & Teramoto, 1990).

In this paper, the rheological synergy between the gums (either guar or xanthan) and modified starch was investigated. More specifically, it was evaluated to which extent the unique rheological behavior of xanthan is manifested in mixed xanthan/starch systems. For that reason, results of the xanthan-starch systems are thoroughly compared with guar-starch and gum-free starch pastes. In contrast with the frequently studied native starch systems, preference was given to chemically cross-linked and acetyl substituted waxy maize starch. Native granules are very sensitive to shear and heat and tend to degrade very easily. This behavior greatly complicates the microstructural interpretation of the obtained results. The use of modified starch allowed a comprehensive study of the rheological properties as a function of different starch volume fractions. A better insight in the effects of gums on the rheological properties of modified starches can not only result in a more sensible use of both components in commercial applications, but it might also assist in the search for native starch alternatives that can mimic the rheological behavior of their modified counterparts.

## 2. Materials and methods

### 2.1. Materials

Xanthan gum (Satiaxane CX911, pyruvic acid content >1.5%) and guar gum (Viscogum MP41230) were acquired from Cargill Texturizing Solutions (Vilvoorde, Belgium). For the covalent labeling, a clarified version of the xanthan gum was used (Satiaxane CX931) to avoid complications during purification and noise within the micrographs due to residual cell debris and other fermentation impurities. Adipate crosslinked and acetyl substituted waxy maize starch (Resistamyl 347) was provided by Tate & Lyle Benelux.

### 2.2. Preparation of gum solutions

Xanthan gum powder was dispersed in deionized water, whilst continuously stirring with a magnetic stirrer. NaCl was added in a concentration of 0.01 M. Next, the premix was put in an Ekato Unimix LM3 laboratory mixer (EKATO Rühr- und Mischtechnik GmbH, Schopfheim, Germany), a mixing apparatus equipped with a temperature control system, paravisc agitator with revolving blades and a colloid mill homogenizer. To fully dissolve the xanthan gum, the premix was homogenized at room temperature for 15 min at 5000 rpm and stirred at an agitation speed of 150 rpm. During homogenization, the system was placed under vacuum to limit air inclusion. The resulting solution of xanthan (0.8% w/w) was heated to 85 °C by means of the unimix system (10 min, stirred at 150 rpm). Guar gum (0.8% w/w) was dissolved in deionized water by heating to 70 °C and continued magnetic stirring at room temperature for 4 h. NaCl was added at 0.01 M. Both hydrocolloid stock solutions were allowed to rest overnight (at 5 °C) before use.

### 2.3. Pasting experiments

The starch powder was suspended either in a 0.1 M NaCl solution (for preparation of the gum free pastes), or in a diluted gum stock solution (adjusted to 0.1 M NaCl). In this manner, starch slurries were obtained with three different gum concentrations in the continuous phase: 0%, 0.2% and 0.4% (w/w). For the last part of the setup, systems were also prepared at a NaCl content of 0.01 M. As it will be remarked repeatedly throughout the text, the effective xanthan concentration in the systems is raised due to starch granule swelling in the heating step. When reference is made to

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