



## Short communication

## Shear rheology and filament stretching behaviour of xanthan gum and carboxymethyl cellulose solution in presence of saliva

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## ABSTRACT

The objective of the work reported in this paper is to determine if saliva addition has an effect on the rheology of xanthan gum solutions. The reasons for the interest was that it has been previously reported that flavour release from high viscosity xanthan thickened foods is not reduced in the same way as foods thickened by other hydrocolloids at comparable viscosities. It was previously postulated that this could be due to an interaction between saliva and xanthan that could change the microstructure and rheology of xanthan solutions. In this work the effect of saliva on the rheology of CMC and xanthan solutions was compared. Solutions of molecularly dissolved xanthan gum and CMC mixed with water or human whole saliva at a ratio of 5:1 showed little impact of the presence of saliva on steady shear or dynamic viscosity for the two hydrocolloids. In filament thinning experiments saliva addition significantly increased filament break-up time for xanthan gum while it had little effect on the break-up time of the CMC filament. Also, filament thinning appeared a lot less even and was not as reproducible in the case of xanthan gum. Addition of CMC and hydroxypropyl methylcellulose (HPMC) to xanthan gum solutions showed a similar increase in break-up time to saliva, but to see this effect the viscosity of the added CMC or HPMC solution had to be very much higher than the viscosity of saliva. The results are discussed in the context of the structure of xanthan gum and the reported extensional rheology of saliva.

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## 1. Introduction

For many years there has been substantial interest in the taste and flavour perception from viscous solutions. It is universally observed that increase in viscosity inhibits taste and flavour perception due to increasingly poor mixing with saliva and therefore slow transport of tastant and flavour molecules to the taste buds (Baines & Morris, 1987; Morris, 1993). Mixing efficiency has also been linked with the good taste and flavour release from starches, which retain their granular form when swollen, compared to a molecular solution of hydroxypropyl methylcellulose (HPMC) that was of the same shear viscosity at  $50 \text{ s}^{-1}$  (Ferry et al., 2006). Rheologically, the two types of solution differ in droplet break-up behaviour as measured rheologically (Desse, Mitchell, Wolf, & Budtova, 2011) and by mixing a solution containing a marker dye with water (Ferry et al., 2006). However, a recent study on model

soups thickened with swollen particles of physically modified xanthan gum and molecularly dissolved xanthan gum revealed that superior mixing behaviour and shorter filament break-up times did not coincide with enhanced taste and flavour release (Abson et al., 2014). The sensory panel was also not able to distinguish taste and flavour perception between the tomato flavour intensity in xanthan and modified starch thickened tomato soup. Morris (1993) found that xanthan gum gives better taste perception at high viscosity than solutions thickened with other molecularly dissolved polysaccharides. The cited observations could suggest that predicting the sensory properties of hydrocolloids from measurements in water could be misleading and that the appropriate fluid to predict sensory behaviour based on flow properties would be saliva.

In this paper the rheological properties of xanthan gum solutions following mixing with water and saliva were analysed in steady shear, oscillatory shear and in uniaxial extensional flow. For the scope of this initial study behaviour in extensional flow was quantified by filament break-up times acquired in a capillary break-up extensional rheometer. The results were compared to solutions of carboxymethyl cellulose (CMC). This was chosen partly because

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like xanthan it is not only a polyelectrolyte with a cellulose backbone, but also because it is frequently used in the formulation of artificial saliva (Hahnel, Behr, Handel, & Buergers, 2009). Unusual strand-like phase separation has been found for xanthan polyelectrolyte blends, but is not seen in mixtures of xanthan with non-polyelectrolytes (Boyd et al., 2009). Thus, the addition of solutions of CMC and the uncharged hydroxypropyl methylcellulose (HPMC) to xanthan gum solutions was included to see if either hydrocolloid gave similar effects to saliva addition.

## 2. Materials and methods

### 2.1. Materials

Xanthan gum (KELTROL®-T) and carboxymethyl cellulose (CEKOL®-20000) with a degree of carboxymethyl substitution in the range 0.75–0.85 (manufacturers data) were provided by CP Kelco UK Ltd (Leatherhead, UK). Hydroxypropyl methylcellulose (METHOCEL™ K4M) was obtained from Dow Wolff Cellulosics (Bomlitz, D). The methoxyl degree of substitution and the hydroxypropyl molar substitution were 1.4 and 0.21 respectively (manufacturers data). The aqueous solutions were prepared with double distilled water and also contained sodium chloride (Sigma Aldrich, Gillingham, UK) and the antimicrobial sodium azide (Sigma Aldrich, Gillingham, UK) at a level of 0.2%w/w and 0.05%w/w respectively. Human whole saliva was used following collection from a healthy volunteer. All protocols involving saliva were previously approved by the local ethics committee and the biological safety officer.

### 2.2. Polysaccharide solution preparation

Each polysaccharide was added in appropriate quantity to an aqueous solution of 0.2%w/w NaCl and 0.05%w/w sodium azide to make a 1%w/w stock solution while mixing on a magnetic stirrer hot plate at 70 °C. The dispersion was stirred continuously for 1 h and kept at room temperature until further use but at least overnight. The desired concentration of each polysaccharide solution was obtained by mixing the stock solution with 0.2%w/w sodium chloride solution on a roller mixer for at least 2 h at room temperature.

### 2.3. Saliva sample preparation

Mechanically stimulated saliva was collected from a healthy volunteer between 10 am and 12 pm. The volunteer was asked to not eat and drink, with the exception of water, for 2 h prior to donation. The donor chewed on a small piece of laboratory self-sealing film (Parafilm, Bermis Flexible Packaging, Neenah, USA) (approximately 5 cm × 5 cm) to stimulate flow of saliva over a period of 10 min. Saliva was expectorated every 30 s into an ice-chilled container whereby the saliva from the first 30 s was discarded as it was likely to contain food and other debris that may have been present in the oral cavity. The collected saliva sample was gently swirled, transferred to a centrifuge tube and centrifuged at 10,000g for 30 min at 4 °C to remove cell debris. The supernatant of the centrifuged sample was stored in a –80 °C freezer and used for experiments within two weeks of collection.

### 2.4. Mixing of polysaccharide solution with saliva

The frozen saliva was defrosted in an ice-chilled water bath just before experiments. 1 g of saliva was added to 5 g of 0.5%w/w xanthan gum solution or 0.65%w/w CMC solution in a 50 mL conical

tube. These solutions were prepared by diluting the stock solution with 0.2% NaCl. Mixing was carried out on a roller mixer for 2 h at 4 °C. Control samples were prepared by replacing saliva with double distilled water under the same conditions.

### 2.5. Steady shear and oscillatory shear rheology

All rheological measurements were carried out in duplicate and at 20 °C with a stress controlled rotational rheometer (MCR 301, Anton Paar, Graz, A) using a cone and plate geometry (0.5° angle, 50 mm diameter). Steady shear viscosity data were collected at shear rates between 0.1 and 1000 s<sup>-1</sup>. Strain sweeps were performed from 0.1 to 1000% strain at the angular frequency of 1.59 Hz. Frequency sweeps were conducted from 10 Hz to 0.1 Hz at 0.5% strain which was in the linear viscoelastic domain.

### 2.6. Filament break-up

Extensional rheology tests were carried out at 25 °C using a capillary break-up extensional rheometer (CaBER-1, Thermo Haake GmbH, Karlsruhe, D) equipped with 6 mm diameter plates. Samples were carefully loaded to ensure the absence of air bubbles between the upper and lower plates with an initial gap of 3 mm. The upper plate was moved up rapidly (within 20 ms; shorter times were not feasible as then the sample would de-wet the upper plate) to a pre-set height of 19 mm to form a filament. The thinning of the filament diameter was monitored at the mid-point of the filament length which corresponds to the standard set-up of the equipment used. Due to the difficulty in loading the sample in the shape of a perfect cylinder and almost certain inhomogeneity of the samples at molecular level, at least 20 replicate measurements were performed and representative results were chosen for analysis.

### 2.7. Experimental design and data analysis

The concentration of the xanthan gum solution was set to 0.5%w/w to correspond to a value typically found in thickened foods. Similarly, the concentration of sodium chloride of 0.2%w/w is found in popular brands of tomato soup. To gain insight into the role charge has to play in the interaction with saliva, CMC was chosen as a negatively charged food hydrocolloid as it is often used as food thickener. A 0.65%w/w concentration of the CMC solution was selected to be shear viscosity matched at a shear rate close to 50 s<sup>-1</sup> at 20 °C with 0.5%w/w xanthan gum following dilution of five parts of polysaccharide solution with one part of water. The shear rate of 50 s<sup>-1</sup> was chosen as the shear rate often used to assess the viscosity of liquid foods in correlation with sensory behaviour (Christensen, 1980; Ferry et al., 2006; Koliandris et al., 2010; Moskowitz & Arabia, 1970) and it corresponds to the value applied in the paper motivating this study. The mixing ratio of 5:1 was chosen based on the maximum flow rate of stimulated saliva reported by Humphrey and Williamson (2001), an estimated serving size of 10 mL of liquid food and an in-mouth residence time of roughly 15 s.

To aid the discussion of the main study data further solutions of CMC at lower concentration and HPMC solutions at two concentrations typically found in food products were included. HPMC is uncharged and mixing xanthan solution with either CMC or HPMC, maintaining the mixing ratio of five parts of xanthan to one part of diluent, filament break-up experiments of these two types of mixtures were conducted. The aim was to gain insight into the role of viscosity and charge of the diluent. Shear viscosity of these CMC and HPMC solutions was also measured and viscosity at 50 s<sup>-1</sup> is reported.

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