



# Influence of mixed gel structuring with different degrees of matrix inhomogeneity on oral residence time



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## ABSTRACT

The aim of this study was to examine the influence of structuring mixed biopolymer gels with different degrees of inhomogeneity on oral residence time. Ten model gels with varying mechanical and structural properties were prepared using  $\kappa$ -carrageenan and sodium alginate at concentrations ranging from 0 to 4 wt%. In few of the mixed gel systems, structural inhomogeneity was introduced by incorporation of calcium alginate beads of different sizes, later made by syringe extrusion or spraying techniques. The gels were characterized by dynamic oscillation, fracture behaviour and the structural details were evidenced in different length scales by cryo-scanning electron microscopy (cryo-SEM) and transmission electron microscopy (TEM). In parallel, gels were characterized by quantitative descriptive analysis (QDA™). Oral processing behaviour was assessed in terms of oral residence time, number of chews and difficulty perceived by eleven young participants. A decrease in the gel fracture point with the addition of calcium alginate beads was attributed to the interruption of the continuous  $\kappa$ -carrageenan gel network, as revealed in the Cryo-SEM and TEM images and with narrower linear viscoelastic region. When the mixed gel network included  $\kappa$ -carrageenan with sodium alginate, the linear viscoelastic range was extended, but the gel strength was lower than  $\kappa$ -carrageenan alone highlighting the incompatibility between the biopolymers. Oral residence time was highly dependent on the number of chews and to a certain extent on the difficulty perceived. Oral residence time and number of chews were positively correlated with gel strength, degree of network inhomogeneity in terms of particle size of the beads.

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## 1. Introduction

Swallowing is a vital part of oral processing, it is a complex act that involves functional coordination of the mouth, pharynx, larynx and oesophagus (Palmer, Drennan, & Baba, 2000). Swallowing disorders may occur due to functional as well as physiological inabilities (Matsuo & Palmer, 2008). Chronic swallowing disorders such as dysphagia are common among the elderly population (Roy, Stemple, Merrill, & Thomas, 2007). They are associated with different pathological conditions, such as Parkinson's disease, Alzheimer's disease, dementia, throat cancer (Ekberg, Hamdy, Woisard, Wuttge–Hannig, & Ortega, 2002; Kumlien & Axelsson, 2002), or with the natural body age-linked degeneration, that tend to increase the risk of aspiration and thus, pneumonia (Nishikubo et al., 2015). In the elderly population, swallowing disorders may lead to malnutrition, which is a severe geriatric

syndrome related to risk of infections, impaired recovery and mortality (Norman, Pichard, Lochs, & Pirlich, 2008).

Clinical researchers have approached swallowing disorders by studying anatomic structures and flow of the food bolus (Palmer et al. 2000) through videofluoroscopy (Langmore, 2003; Palmer et al., 2000), fiberoptic endoscopic (Dua, Ren, Bardan, Xie, & Shaker, 1997) or ultrasound equipment (Koshino, Hirai, Ishijima, & Ikeda, 1997) among others. In parallel, food scientists have investigated the role of precise optimization of viscosity of food biopolymers with an objective of manipulating the swallowing process. One of the main conclusions of previous researches is that increasing viscosity of food and thereby increased oral residence time is an effective strategy to combat swallowing disorders (aspiration) (Logemann, 2007). Hydrocolloids have been commonly used as thickeners in food for swallowing disordered and/or dysphagia patients (Zargaraan, Rastmanesh, Fadavi, Zayeri, & Mohammadifar, 2013) these thickeners were conventionally xanthan gum or starch based (Leonard, White, McKenzie, & Belafsky, 2014; Seo & Yoo, 2013). Garcia, Chambers, Matta, and Clark

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(2005) concluded that in comparison to a thin bolus, a thicker bolus will be residing in mouth for a relatively longer time. This sensory feedback of slow bolus flow through the oropharynx will protect airways (Nicosia & Robbins, 2001). Thickened diets have shown to improve the nutritional status of the patients as well as their hydration level due to lower chances of aspiration and pneumonia (Rofes et al., 2010).

Using hydrocolloid based test fluids and gels have been effective model systems to study the influence of rheological properties in the food oral processing (Hayakawa et al., 2014; Hori et al., 2015; Ishihara, Nakauma, Funami, Otake, & Nishinari, 2011; Kohyama et al., 2015; Moritaka & Nakazawa, 2010), with the aim to design food for people suffering from dysphagia and/or at risks of swallowing disorders. Other advantage of working with model hydrocolloids is that they are not emotionally linked and excludes the postprandial satisfaction and flavour experience, which occurs when testing with well-known real food-products (Prescott, 2012; Yeomans, 2012). It was found that the difficulty associated with an increment of time in the mouth (Moritaka & Nakazawa, 2010) was linked with sensory attributes such as resistance to fracture (Hayakawa et al., 2014; Laguna, Barrowclough, Chen, & Sarkar, 2016), which further highlights the influence of viscosity and/or gel strength of the liquid or semi-solid food on oral processing.

Although the influence of consistency on time in mouth is well researched, there has been scant literature on the complex interplay between structural properties of gels and oral processing. In this study, we hypothesize that not only viscosity but also the degree of structure can increase time in mouth. Hence, this study aims to explore different factors to increase the time in mouth: the gel strength, structural complexity, or the interaction between gel strength and complexity. To achieve this objective, we have created a series of edible  $\kappa$ -carrageenan gels without or with sodium alginate or inclusion of calcium alginate beads with diverse mechanical and oral processing properties via precise manipulation of structural inhomogeneity (i.e. different concentrations and particle size of the beads).

$\kappa$ -Carrageenan is a biopolymer with repeating disaccharide units of 3-linked  $\beta$ -D-galactose 4-sulfate and 4-linked 3,6-anhydro- $\alpha$ -D-galactose.  $\kappa$ -Carrageenan can form thermo-reversible gels at low concentrations and the gelation involves coil-to-helix molecular transition of the  $\kappa$ -carrageenan molecules followed by aggregation that occur upon cooling (Morris, Rees, & Robinson, 1980). Sodium alginate is a linear anionic polysaccharide derived from brown seaweeds, consisting of  $\beta$ -1,4-D-mannuronic acid (M-block) and  $\alpha$ -1,4-L-glucuronic acid (G-block). Sodium alginate can undergo ionic crosslinking upon contact with calcium ions in aqueous solution to form an “egg-box model” gel structure (Yoo, Song, Chang, & Lee, 2006). The divalent calcium displaces the sodium ion and due to the physical crosslinking or chelation between the carboxylate anions of guluronate units in alginate and the calcium ions, the calcium-alginate gel beads are formed. Mixing of these distinct macromolecules may result in the formation of two microscopic layers, with each containing most of one constituent and little of the other. The phenomenon is known as phase separation and, in the gel state, the phase morphology of the mixture determines the overall structure (Goh, Sarkar, & Singh, 2008, 2014) and thus may have an influence on the oral processing behaviour. Furthermore, incorporation of food-grade calcium alginate beads in a  $\kappa$ -carrageenan “continuous” biopolymer matrix may increase/decrease the mechanical strength of the mixture depending upon the interaction, which might further influence the oral residence time.

To our knowledge, this is the first study that generates insights on impact of mixed gel structuring on oral residence time by employing a holistic combination of characterization of these mixed gels using structural, mechanical (small and large

deformation rheology), sensory and oral processing techniques.

## 2. Materials and methods

### 2.1. Sample preparation

$\kappa$ -Carrageenan and sodium alginate were both obtained from Special Ingredients (Sheffield, UK). Calcium chloride was obtained from Mineral Water (Purfleet, UK). All three ingredients were food grade and used without any further purification. The concentration of the biopolymers is summarized in Table 1.

*Calcium alginate beads production (CAI)*. Firstly, sodium alginate solutions were prepared by slowly adding the exact quantity of the powder in distilled water. The obtained dispersion were then heated and stirred for 1 h at 90 °C to ensure complete solubilisation. Calcium chloride solutions (2 M) were prepared by dissolving the required quantity in distilled water. For the preparation of big beads, sodium alginate (Na alginate) solution was extruded using a 0.8 mm nozzle syringe (Terulo, Neolus) into the calcium chloride bath. For the small beads, sodium alginate solution was sprayed at 50–55 mL/min over the calcium chloride bath using jet sprayer (0.45 mm nozzle diameter). The Na-alginate beads were cross-linked by  $\text{Ca}^{2+}$  ions to form sprayed Ca-alginate beads. Both beads (big and small i.e. sprayed, particle size is summarized in Table 2) remained in the  $\text{CaCl}_2$  bath for 30 min; the prepared beads were removed and washed with deionized water twice to remove any non-cross-linked  $\text{Ca}^{2+}$  ions.

*$\kappa$ -Carrageenan gel production ( $\kappa$ )*. 1–4 wt% of  $\kappa$ -carrageenan (as indicated in Table 1) was prepared by dissolving appropriate quantities of  $\kappa$ -carrageenan in distilled water and mixed by magnetic stirring for a few hours at 80 °C to facilitate hydration.

*$\kappa$ -Carrageenan and sodium alginate gel production (M- $\kappa$ SAI)*. Binary gel preparation involved dry blending of appropriate quantities of  $\kappa$ -carrageenan and sodium alginate and dissolving in distilled water (1.0 and 2.0 wt%) followed by magnetic stirring for a few hours at 80 °C.

*$\kappa$ -Carrageenan and calcium alginate bead production (B- $\kappa$ CAI/S- $\kappa$ CAI)*. Small (spray) or big beads were added to tray (12 × 7.5 × 1.5 cm length, width, depth), then,  $\kappa$ -carrageenan solution of 1–2 wt% concentration (80 °C) was poured in to the tray in 1:1 w/w. After storage at 4 °C for 24 h, gels were cut in a circular shape (2.0 × 1.0 cm; diameter × height).

### 2.2. Rheological measurements

#### 2.2.1. Small deformation rheology

The rheological properties of the mixed gels were analysed by dynamic oscillatory measurement in a Kinexus rheometer (Malvern, UK). Gel cylinder of 30 mm diameter were placed into a pre-heated plate (37 °C), the rheometer was equipped with a 30 mm of parallel plate. Considering that the gap between the plates should be larger than the biggest bead, a gap of 3 mm was selected. A strain sweep test from 0.01 to 100% was carried out to determine the linear viscoelastic region at constant angular frequency of 1 Hz. Frequency sweeps were conducted from 0.01 to 100 Hz at constant strain of 0.05%. The elastic (storage modulus,  $G'$ ) and viscous modulus (loss modulus,  $G''$ ) and complex moduli ( $G^*$ ) were recorded. Experiments were replicated three times.

#### 2.2.2. Large deformation rheology

To characterize the mechanical properties, fracture mechanics of mixed gels were conducted by both penetration test using upper Volodkevich Bite Jaw and compression test using 75-mm diameter aluminium plate (P/75) (Texture analyser, Stable Micro Systems, Godalming, UK). Since human frontal teeth are around 8–9 mm,

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