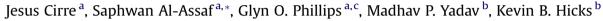
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ABSTRACT

Corn fibre gum (CFG) in the solid state (milled powder form) was subjected to a maturation treatment by heating under atmospheric pressure at 110 °C for 5 (CFG5) and 24 h (CFG24). The treatment reduced the solubility and aggregation of the proteinaceous component increased with heating time. The protein content of CFG is comparable to that of gum arabic but has a different amino acids profile. Glutamine and proline are the most abundant in CFG compared with hydroxyproline and serine in gum arabic. The control and matured CFG samples have been characterized by gel permeation chromatography coupled on line to a multi angle laser light, refractive index and UV detectors. The weight average molecular weight (M_w) values for control, CFG5 and CFG24 were 4.2, 5.8 and 5.7 × 10⁵ g/mol respectively with corresponding Rg values of 29, 44 and 41 nm. High pressure homogenization treatment of the control gum. A three times increase in the proportion of the adsorbed fraction on to the oil–water interface accounts for the improved emulsification. A model to describe the changes following maturation and the improved emulsion stability is proposed.

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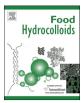
1. Introduction

Corn fibre arabinoxylan, also called hemicellulose B (Hemi. B) is isolated from the fibrous portions (pericarp, tip cap, and endosperm cell wall fractions) of corn kernels by alkaline solution treatment, often in the presence of hydrogen peroxide. It is commonly referred to as "Corn fibre gum" (CFG) (Yadav, Johnston, & Hicks, 2009). The emulsification behaviour of CFG extracted from corn fibre obtained from 3 different milling processes: wet milled pericarp fibre (WPF), dry milled pericarp fibre (DPF) and wet milled pericarp and endosperm fibre (WPEF) has been reported by Yadav, Johnston, Hotchkiss, and Hicks (2007). Two different fractions, CFG-1 and CFG-2 from each of these starting materials were isolated by (a) sequential alkaline extraction and alkaline hydrogen peroxide bleaching and (b) an additional alkaline hydrogen peroxide treatment of the alkali treated residue, respectively. The carbohydrate portion of these fractions contained approximately 50% xylose, 32% arabinose, 6.6% galactose, 4.6% glucuronic acid (mole %) and traces of rhamnose, glucose and fucose. They also investigated the emulsification behaviour of these two CFG fractions and found that the CFG-2 fraction had a better emulsification and stabilizing ability than the CFG-1 fraction. Possible reasons for the superior performance of CFG-2 could be its higher protein and lipid contents, higher average molecular weight, more compact structure and lower viscosity in comparison to CFG-1. It was also found that CFG extracted from wet milled pericarp fibre had a higher protein content than CFG extracted from dry milled pericarp fibre and was also a better emulsifier. The investigators suggested that it could be the presence of phenolic acids, lipids, and protein as well as the high molecular weight and branching of CFG which accounted for its superior emulsification properties. Additionally, they reported that the emulsification behaviour was better than native and modified acacia gums (Yadav et al., 2007).

Gum arabic, is another complex polysaccharide which consists mainly of carbohydrates and small amount ($\sim 2\%$) of protein which is an integral part of the structure (Akiyama, Eda, & Kato, 1984). Controlled thermal processing (maturation treatment) greatly







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improves its ability to emulsify oil in water emulsions (Aoki, Al-Assaf, Katayama, & Phillips, 2007; Al-Assaf, Phillips, Aoki, & Sasaki, 2007; Castellani et al., 2010). The new products designated as *Acacia (sen) SUPERGUM*TM differs, not in the amount of protein it contains, but by having more of the high molecular weight fraction termed arabinogalactan protein complex (AGP) (Al-Assaf et al., 2007). The AGP fraction has been demonstrated to determine the functionality and flow properties of gum arabic (Al-Assaf et al., 2007; Aoki, Al-Assaf, et al., 2007; Castellani et al., 2010; Li et al., 2011) and can be disaggregated following filtration and high pressure homogenisation (Al-Assaf, Sakata, McKenna, Aoki, & Phillips, 2009).

The objective of this study is to examine the effect of maturation treatment on CFG solution properties and emulsification behaviour with direct comparison to gum arabic.

2. Experimental

2.1. Materials

Unless indicated otherwise, all chemicals and reagents were of analytical grades obtained from Fisher Scientific, UK.

2.2. Isolation of corn fibre gum

To prepare lipid and starch free CFG, first "corn fibre" was extracted with hexane to remove oil and treated with thermostable α -amylase at 90–95 °C to hydrolyse the starch present in the corn fibre. CFGs were extracted from de-oiled and de-starched corn fibre according to the alkaline hydrogen peroxide technology (Yaday et al., 2007) with some modifications. In brief, de-oiled and destarched corn fibre was mechanically stirred into an alkaline solution containing 0.088 M NaOH and 0.026 M of Ca(OH)₂ in the presence of 0.63% aqueous H₂O₂ at boiling temperature for 30 min with maintaining pH about 11.5 by adding more NaOH. The reaction mixture was centrifuged at 6000 g for 30 min after cooling it by stirring at room temperature and the supernatant was separated from the residue by decantation. The residue was re-suspended in water and boiled for 5 min with mechanical stirring. The reaction mixture was centrifuged as described above and the supernatant was decanted and mixed with the first supernatant. The pH of the combined supernatants (alkaline H₂O₂ extract) was then adjusted to 4.0-4.5 by adding HCl to precipitate Hemicellulose A ("Hemi. A"), which was collected by centrifugation at 10,000 g for 1 h. Two volumes of ethanol were gradually added to the supernatant with stirring to precipitate the Hemicellulose B, or "Hemi. B", (CFG), which was collected and dried.

2.3. Maturation process, <u>GPC</u> analysis and rheological properties measurements

The percentage loss on drying, maturation treatment, GPC-MALLS and enzyme digestion was determined according to the methods previously described (Al-Assaf et al., 2007). The maturation treatment on CFG was carried out at 110 °C in an oven at atmospheric pressure for 5 and 24 h. The modified CFG products were labelled as CFG5 and CFG24 respectively. The solubility in distilled water and 0.2 M NaCl was determined by measuring the weight retained on a glass fibre filter (0.7 micron, Fisher Scientific, UK) following the dispersion of 0.5 g of the test materials in 20 ml of the respective solvent as described previously (Gulrez, Al-Assaf, Fang, Phillips, & Gunning, 2012; Li, Al-Assaf, Fang, & Phillips, 2013).

The effect of high pressure homogenisation on the aqueous solutions of control and matured CFG was investigated using Nanomiser (NM2-L100-D07, Penetration type generator, Yoshida Kikai Co. Ltd). The solution of 2 mg ml⁻¹ of control or matured CFG

in 0.2 M NaCl was initially treated by high shear mixing using polytron model PT 2100 (Impeller PTA 21 9 mm diameter) for 3 min at 26,000 rpm followed by high pressure homogenizer using a Nanomizer. Each solution was passed twice through the Nanomizer at 50 MPa and 75 MPa and subsequently diluted to 1 mg ml⁻¹ and injected into the GPC-MALLS system.

2.4. Rheological properties measurements

The shear flow viscosity of control and matured samples was measured at 25 °C using an advanced controlled stress rheometer AR 550 (TA instruments, UK). The respective sample solution (2 wt% in 0.1 M NaCl) was prepared as described above. Two geometries were used: cone (2°, 4 cm diameter) and plate for control gum and parallel plates (6 cm diameter) for CFG5 and CFG24 due to the presence of hydrogel fraction. The effect of shear induced disaggregation was investigated by initially performing the flow measurements with increasing shear rate (up curve) and upon completing the measurements with decreasing shear rate (down curve).

2.5. Emulsification performance and study of emulsifying properties

The emulsification performance and stability were evaluated for control and matured samples using the procedure reported by Aoki, Katayama, Ogasawara, Sasaki, Al-Assaf, & Phillips, 2007. The selected sample was dissolved in distilled water at 6 wt% (moisture compensated) and left on a roller mixer to hydrate overnight. Next 33.33 g of this 6 wt% stock solution was mixed with 0.96 g of 10 wt% aqueous solution of citric acid (to adjust the pH to \sim 4), 1.04 g of 10 wt% aqueous solution of sodium benzoate (as a preservative), 8 g of medium chain triglyceride (MCT) oil and diluted with distilled water to a final weight of 80 g. The final concentrations in the 80 g emulsions were 2.5 wt% gum, 10 wt% MCT oil, 0.12% citric Acid and 0.13 wt% sodium Benzoate. The pre-emulsion was prepared by mixing using high shear mixer (polytron, 3 min at 2600 rpm) and subsequently passed twice through a high pressure homogenizer (Nanomizer) at 50 and 75 MPa. The droplet size distributions of emulsions were determined, using Malvern Mastersizer 2000 (Malvern Ltd., UK), by fitting the data using the general purpose model. Values of 1.45 and 0.001 were used for MCT refractive index and absorption index respectively and 1.33 and 0 for the dispersant (water) respectively. The emulsions were subjected to an accelerated stress testing by incubation at 60 °C. The performance and stability of the emulsion were evaluated by measuring the initial droplet size immediately after preparing the emulsions and after storing them at 60 °C (accelerated stress test) for 3 and 7 days.

2.6. Separation of adsorbed and non-adsorbed CFG fractions

The adsorbed and non adsorbed CFG fractions from the emulsions were separated according to the method reported by Funami et al. (2007). The procedure was carried out on emulsions made using the same method above with the exception of using coldpressed Valencia orange oil supplied by Citrus and Allied Essences Ltd., Belcamp, MD and by passing through EmulsiFlex-B5 high pressure homogenizer (Avestin Inc.) at 20,000 psi pressure 3 times. The concentration of adsorbed gum was calculated using Eq. (1).

$$C_{\rm a} = C_{\rm g} \times \frac{\rm RM_{\rm a}}{\rm RM_{\rm g}} \tag{1}$$

Where C_a is the concentration of the adsorbed gum, C_g of the gum in solution, RM_a and RM_g the mass recovery (measured by GPC-RI) of the adsorbed and non adsorbed gums.

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