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Effect of high pressure treatment on the structural, mechanical and rheological properties of glucomannan gels

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ABSTRACT

The influence of high hydrostatic pressure (HHP) of 100 (C100), 200 (C200), 400 (C400) and 600 MPa (C600) on the structural, physicochemical, and thermo-rheological properties of aqueous glucomannan dispersions (AGD) was studied. Each AGD was prepared with a 5% concentration of deacetylated glucomannan (GM) at pH = 11 for use in the preparation of restructured fish products. The control (0 MPa) AGD (C0) exhibited a partially crystalline GM network with a glass transition temperature (T_g) of ~75 °C. C0 gels at 25 °C were rigid, with high breaking force (*BF*), fracture constant (K_f) and complex modulus (G^*) and low *tanδ* values due to the large number of physical junctions produced by complete deacetylation of the GM chains. This structural response was reflected in high water binding capacity (WBC) and colour (L^*) and low cooking loss (CL). Specifically, 200 MPa reduced close packing ability in the crystalline regions, evidenced in the lower T_g~65 °C and resulting in lower gel strength (*S*), K_f, G^* and a higher viscous component (high *tanδ*), thus reducing the L^* value. Conversely, 600 MPa increased the degree of cross-linking in C600, reinforcing the number and extent of crystalline regions, resulting in the broadening of the O–H stretching band in the FT-IR spectrum and producing a thermoset physical network with no glass transition region. Thus, C600 gel was firmer (higher G^*) with greater *S*, and less flexible (lower strain amplitude-γ_{max}). Consequently, depending on the textural properties desired in the final restructured fish product, the most appropriate pressure would be 200 MPa for softer final gels and 600 MPa for firmer and less deformable gels.

1. Introduction

Konjac glucomannan (KGM) is a neutral polysaccharide from the tuber of *Amorphophallus konjac C. Koch*, able to form thermo-resistant gels when deacetylated by adding an alkaline agent (Nishinari, Williams, & Phillips, 1992; Yoshimura & Nishinari, 1999).

The three-dimensional networks formed by glucomannan (GM) are physically cross-linked structures formed by locally ordered regions (junction zones) which are stabilized by physical interactions such as hydrogen bonds and hydrophobic interactions (Huang, Takahashi, Kobayashi, Kawase, & Nishinari, 2002).

Previous research has thoroughly investigated the influence of thermal conditions (Herranz, Borderias, Solas, & Tovar, 2012), GM concentration (Herranz, Borderias, Solo-de-Zaldívar, Solas, & Tovar, 2012) and deacetylation ratio (Solo-de-Zaldívar, Tovar, Borderías, & Herranz, 2014) on the stability of these networks as they relate to the mechanical, rheological and structural properties of aqueous glucomannan dispersions (AGD). These studies were carried out to determine the GM gelation conditions most suited to making gels in combination with minced fish, with the appropriate texture for restructured seafood products (Solo-de-Zaldívar, Herranz, Borderías, & Tovar, 2014; Solo-de-Zaldívar, Tovar, Borderías, & Herranz, 2015). The more elastic and time-stable GM gels were obtained at 0.6 N KOH to achieve a pH value of ~10.7 for AGD at 3% and/or 5% GM concentration; this pH corresponds to a ~95% deacetylation ratio for 3% AGD and a ~58% ratio for 5% AGD (Solo-de-Zaldívar, Tova et al., 2014).

The food industry is becoming increasingly interested in using high hydrostatic pressure (HHP) as a non-thermal process to produce safe foods with different or enhanced functional properties (Cando, Moreno, Tovar, Herranz, & Borderías, 2014; Cao, Xia, Zhou, & Xu, 2012). As has previously been shown, HHP mainly affects hydrophobic and electrostatic interactions and protein conformation in fish gels made from surimi and myofibrillar proteins

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(Moreno, Cardoso, Solas, & Borderías, 2009; Pérez-Mateos & Montero, 1997; Tan, Lai, & Hsu, 2010) but there is little information in the scientific literature about the effect of HHP on hydrocolloids other than starch (Kim et al., 2015; Liu, Hu, & Shen, 2010). Therefore, before applying HHP to the coupled network formed by glucomannan-fish proteins, an in-depth analysis of HHP treatment on GM gels is needed to assess whether HHP could enhance polymer functionality, thus improving the reticular stability of the GM gel-networks and supporting its use in GM gelation.

Consequently, the aim of this paper was to study the effect of HHP on GM gels in terms of the structural, physicochemical, thermo-rheological and viscoelastic properties of 5% aqueous glucomannan dispersions processed under different levels of HHP. This initial study could be applied to fish processing, specifically fish restructuring.

2. Materials and methods

2.1. Sample preparation

Konjac glucomannan used for this study was purchased from Guinama (Valencia, Spain) and had its average molecular weight of about 11×10^5 Da. 5% (w/v) aqueous glucomannan dispersions (AGD) were prepared with this type of konjac glucomannan by continuous stirring for 30 min at 50 rpm in a vacuum homogenizer (Stephan UM5, Stephan u. Söhne GmbH & Co., Hameln, Germany) at 60 °C. Then, 4 ml/100 ml (v/v) of alkali coagulant: 0.6 N KOH (Panreac Química, S. A., Barcelona, Spain) was added to AGD to produce pH = 11 following the method described in Herranz, Borderias, Solo-de-Zaldívar et al. (2012). The gels were removed from Petri dishes and cylindrical containers (diameter 3 cm × height 3.5 cm) after setting for 1 h at 30 °C and 5 h at 5 °C, and were placed in a citrate-phosphate buffer at pH 5 (1:10 gel:-buffer proportion) for 20 h at 5 °C to lower the pH of the gels to neutral values. Gels were then vacuum packed in double plastic bags to prevent contact with pressurization fluid. They were then immediately subjected to different degrees of HHP: 100 (C100), 200 (C200), 400 (C400) and 600 MPa (C600) for 10 min at 10 °C (Stansted Fluid Power CTD, FPG 7100:-2C. Stansted, UK.). A control sample (C0) (unpressurized gel) was also used. Analyses were conducted after 1 day of chilled storage. All samples were warmed to 25 °C before analysis and exhibited high moisture content (94.8–95.5%).

2.2. Analyses

2.2.1. FT-IR spectroscopy analysis

FT-IR analysis was done by transmission measurement using a Perkin–Elmer Spectrum 400 Infrared Spectrometer (Perkin–Elmer Inc., Waltham, MA, USA).

Samples were freeze-dried and then dispersion was achieved using Fluorolube reagent, since its absorption bands do not interfere in the observation of the sample bands. In a subsequent step during spectra processing Fluorolube bands are subjected from the samples spectra (Solo-de-Zaldívar, Tovar et al., 2014). A small quantity of the paste produced by homogenization with Fluorolube was placed between two CaF_2 crystals. Then, IR spectra were recorded for all samples by accumulating 32 scans with a resolution of 2 cm^{-1} in a frequency range of 4000–100 cm^{-1}. Measurements were carried out in triplicate and processed using Spectrum 400 software (Perkin–Elmer Inc., Waltham, MA, USA). To facilitate a quantitative discussion of the spectra from each spectrum, absorption intensity was assumed to be proportional to the concentration of the adsorbing species (Wilkson, Slack, Appleton, Sun, & Belton, 1993).

2.2.2. Proximate analysis

A model 9165BNWP pH probe (Analítica Instrumental, S.A., Barcelona) was inserted in the gel to measure pH. The pH meter was an Orion model 720 A (Analítica Instrumental, S.A., Barcelona). The analyses were performed in triplicate.

Water content was determined by drying samples to constant weight at 110 °C, results expressed as a percentage (AOAC, 2000). Measurements were carried out in triplicate.

2.2.3. Physicochemical properties

2.2.3.1. Water binding capacity (WBC). Gels were cut into small pieces (2 g) and placed in a centrifuge tube (diameter 10 mm) with sufficient filter paper (2 Whatman no. 1 diameter 90 mm filters). Samples were then centrifuged in a Jouan MR1812 centrifuge (Saint Nazaire, France) for 10 min at 3000 g at room temperature. WBC, determined in triplicate, was expressed as the percentage of water retained per 100 g of water present in the sample prior to centrifuging.

2.2.3.2. Cooking loss (CL). The sample (40 g) was cut into small pieces and placed in a plastic bag with small holes for drip draining. This bag containing the sample was then placed inside a second bag, hung with the holes at the bottom and cooked in that position in an oven (Rational Combi-Master CM6) for 20 min at 100 °C. The sample was then cooled and weighed. CL was expressed as g/100 g by weight difference between uncooked and cooked samples. Measurements were performed in triplicate.

2.2.3.3. Colour. Lightness (L*), was analysed using a CIELab scale. Measurements were made using a colorimeter (Minolta Chroma Meter Cr-200, Japan). The colour was measured five times on the surface of the gel before and after the HHP treatment. Before the measurements, the colorimeter was standardized using a white calibration plate. Determinations were carried out on five surface points of each sample.

2.2.4. Puncture tests

Cylindrical samples (diameter 3 cm × height 3.5 cm) were pierced to breaking point using a TA-XTplus Texture Analyser (Stable Micro System Ltd., Surrey, UK) with a 5 mm-diameter round-ended metal probe at room temperature. Crosshead speed was 1 mm/s, and a 5 kg load cell was used. The load was recorded as breaking force (BF) and the depth of depression (i.e. the point at which the GM gels lose their strength and rupture) as breaking deformation (BD). Measurements were carried out at least in sextuplicate.

2.2.5. Dynamic rheometry measurements

Small amplitude oscillatory strain (SAOS) tests were performed using a Bohlin CVO controlled stress rheometer (Bohlin Instruments, Inc. Cranbury, NJ). Measurements were made using parallel-plate geometry (20 mm diameter and 1 mm gap). Before measurement the samples were tempered at ambient temperature and cut from Petri dishes into disk-shaped slices 20 mm in diameter and 1 mm thick using a 570 S.T.E slicer (Germany). They were then placed on the lower plate of the rheometer and measured at 25 °C. Samples were allowed to rest for 15 min before analyses to ensure both thermal and mechanical equilibrium at the time of measurement. Samples were coated with a trap solvent to avoid evaporation during measurement. The temperature was controlled to within 0.1 °C by a Peltier element in the lower plate. The dynamic measurements were carried out at least in quintuplicate.

2.2.5.1. Stress sweep tests. To determine the linear viscoelastic (LVE) region, stress sweeps were run at 6.28 rad/s at 25 °C with the