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Preparation, characterization and *in vitro* digestibility of gellan and chitosan–gellan microgels

Joice Aline Pires Vilela, Fabiana de Assis Perrechil, Carolina Siqueira Franco Picone, Ana Carla Kawazoe Sato, Rosiane Lopes da Cunha *

Department of Food Engineering, School of Food Engineering, University of Campinas (UNICAMP), R. Monteiro Lobato, 80, 13083-862 Campinas, SP, Brazil

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ABSTRACT

Gellan microgels with potential application in delivery systems were obtained by physically cross-linked gellan gum. The microgels were produced by atomization followed by ionotropic gelation using $CaCl_2$ (gellan/Ca) or KCl (gellan/K) as hardening agent and part of them were coated with chitosan in order to improve their resistance to gastric digestion. Size distribution, morphology and zeta potential of microgels were evaluated before and after *in vitro* digestion process. The long term stability was also evaluated. Spherical microparticles were obtained at gellan concentration above 0.6% w/w, showing average size among 70–120 μm. Most of the coated and uncoated microgels showed stability in aqueous media, except the uncoated gellan/K microgel. The *in vitro* digestion evaluation showed that all particles maintained their size and shape after the gastric digestion step. However, the enteric digestion caused disintegration of microgels indicating their potential application for enteric delivery systems. The chitosan-coated microgels showed lower degree of fragmentation when compared to the uncoated microgels, indicating that the coating process enable a better control of microgels releasing properties during the enteric digestion.

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1. Introduction

Microgels are gel particles of micrometric dimensions (Oh, Lee, & Park, 2009) which present several applications as encapsulation matrix of bioactive compounds as well as texture modifier agents. Microgels based on biopolymers such as polysaccharides can be added to food, cosmetics and pharmaceutical products without risk to health, since most of these compounds are nontoxic and biocompatible. Polysaccharides microgels can be formed by physical cross-linking, in which hydrophobic and electrostatic interactions are mainly involved in the gel network formation (Das, Zhang, & Kumacheva, 2006). The greater advantage offered by microgels is their flexibility, since the final properties as well as their response to the environmental conditions can be easily tuned.

Microgels properties depend on their composition and the process conditions selected for their manufacturing. Several methods can be used to produce microgels (Poncelet et al., 1992; Das et al., 2006; Herrero, Del-Valle, & Galán, 2006; Li, Rouaud, & Poncelet, 2008; Perrechil, Sato, & Cunha, 2011; Perrechil, Vilela, Guerreiro, & Cunha, 2012). For biopolymer-based microgels, emulsification and extrusion processes are the most used for droplets formation before the gelation step. Among the extrusion-based methods, the atomization process allows the production of micrometer size droplets through a simple apparatus and without the use of drastic process conditions such as intensive heating or organic solvents. The droplets are collected in a bath containing gelling agents, like salts, that can diffuse into the polysaccharide droplets to form the microgels (Herrero et al., 2006; Perrechil et al., 2011).

The gellan gum is a negatively charged polysaccharide produced by the microorganism *Sphingomonas elodea*. Gellan is composed of repeating tetrasaccharide (1,3-β-D-glucose, 1,4-β-D-glucuronic acid, 1,4-β-D-glucose, 1,4-α-L-rhamnose) units containing one carboxyl side group (Jansson & Lindberg, 1983). It has the ability to form gel by the addition of salts or acids, and the gelation can occur even at low gellan concentration. The gelation occurs in two steps: first there is the formation of double helices during cooling, followed by the cation-mediated aggregation of the double helices leading to gelation (Miyoshi, Takaya, & Nishinari, 1996). The bulk gels of gellan have been widely studied and a wide variety of textures/mechanical properties is found depending on the gelation conditions (Sworn, Sanderson, & Gibson, 1995; Tang, Tung, & Zeng, 1996; Yamamoto & Cunha, 2007; Evageliou, Karantoni, Mandala, & Komaitis, 2010; Vilela, Cavallieri, & Cunha, 2011; Picone & Cunha, 2011; Norton, Cox, & Spyropoulos, 2011). Gellan gum can also form

* Corresponding author. Tel.: +55 19 3521 4047; fax: +55 19 3521 4027.
E-mail address: rosiane@fea.unicamp.br (R.L.d. Cunha).

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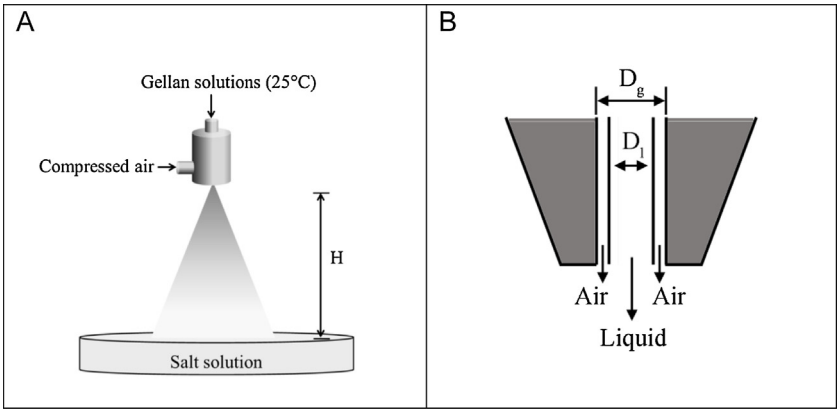

Fig. 1. (A) Extrusion for microgels formation and (B) Atomizer nozzle. H = height from the atomizer nozzle to the salt solution, D_l = diameter of the fluid nozzle exit and D_g = diameter of gas nozzle exit (Adapted from Perrechil et al., 2011).

polyelectrolyte complexes with oppositely charged polymers such as chitosan (Amaike, Senoo, & Yamamoto, 1998; Ohkawa, Kitagawa, & Yamamoto, 2004).

Chitosan is a positively charged polysaccharide obtained by the alkaline deacetylation of the chitin. The chitosan structure is composed by units of *N*-acetyl-2-amino-2-deoxy-D-glucopyranose and 2-amino-2-deoxy-D-glucopyranose linked by (1 → 4)-β-glycosidic bonds (Dash, Chiellini, Ottenbrite, & Chiellini, 2011). Particles coated with chitosan show enhanced protective capacity (Zou et al., 2011; Colinet, Dulong, Mocanu, Picton, & Cerf, 2010; Cook, Tzortzis, Charalampopoulos, & Khutoryanskiy, 2011) and notably increase their mucoadhesion properties, since the mucus layer of the gastrointestinal tract show negative charge (Allen, Flemström, Garner, & Kivilaakso, 1993). The mucoadhesion is a relevant property for encapsulation matrix, because it is related to the residence time of the particles in the gastrointestinal tract (Gåserød, Jolliffe, Hampson, Dettmar, & Skjåk-Bræk, 1998; Harding, 2003) and to the absorption rate of the bioactive compound.

Therefore, the purpose of this study was to investigate the potential as encapsulating matrix of the gellan microgels produced by atomization followed by ionotropic gelation using mono (K^+) or divalent (Ca^{2+}) cations as gelation agents. The effect of the chitosan coating on the particles properties and stability was also studied.

2. Material and methods

2.1. Materials

Deacylated gellan gum (Kelcogel) was kindly donated by Kelco Biopolymers (San Diego, CA) and it was used without further purification. Chitosan (deacetylation degree ~86%) was purchased from Primex Ingredients SA (Iceland). The other reagents were of analytical grade and purchased from Sigma-Aldrich Corporation (St. Louis, USA).

2.2. Preparation of biopolymers solutions

Gellan solutions were prepared by the dispersion of the powder in deionized water, followed by heat treatment at 70 °C for 30 min under magnetic stirring. After that, these solutions were cooled to 25 °C using a water bath. The chitosan solution (0.25% w/w) was obtained by dissolving the polysaccharide in sodium acetate buffer 0.2 M (pH = 3.0 ± 0.1) under magnetic stirring at room temperature for 12 h.

2.3. Gellan microgels production

The extrusion process used for the microgels production was adapted from the Perrechil et al. (2011). The gellan solutions (25 °C) at different concentration (0.4, 0.6, 0.8, 1 and 1.2% w/w) were extruded by an atomizer nozzle (D_l = 0.7 mm and D_g = 2.5 mm) into KCl or $CaCl_2$ solutions to produce the microgels. The height from the atomizer nozzle to the salt solutions (Fig. 1) was H = 200 mm, the feed flow rate was 0.18 L/h and the compressed air (absolute pressure of 200 kPa) flow rate at the nozzle was 1628 L/h (~150 m/s). The extruded particles were maintained in the salt solution under magnetic stirring for 30 min and then filtered through a sieve with opening of 0.037 mm. The concentration of salt solutions was 2.28% (w/v) KCl or 1.1% (w/v) $CaCl_2$, which resulted in the same ionic strength (0.3 M). Morphology, particle size distribution and zeta potential of these microgels were evaluated, as well as their long term stability.

2.4. Chitosan coating

Gellan microgels (0.6, 0.8, 1 and 1.2% w/w) formed by ionic gelation with $CaCl_2$ (gellan/Ca) or KCl (gellan/K) were dispersed into a solution of chitosan under magnetic stirring for 30 min. The addition of the microgels into the solution of chitosan was done in two steps to prevent the formation of agglomerates. First, the gellan particles were dispersed in sodium acetate buffer 0.2 M (pH 3.0) and then, chitosan solution (0.5% w/w) was added gradually, resulting in a final dispersion containing 20% w/w of particles in 0.25% (w/w) chitosan solution. The dispersions were filtered through a sieve opening of 0.037 mm, and the morphology, particle size distribution and zeta potential of these microgels (gellan/Ca/chitosan and gellan/K/chitosan) were evaluated. The long term stability of these microgels was also determined in order to compare with uncovered microgels.

2.5. Stability

To check for microparticle stability, suspensions were prepared by dispersing 10% (w/w) microgels (1% w/w gellan) in distilled water or in sodium acetate buffer 0.2 M (pH 3). These microgel suspensions were stored at 10 °C and fractions were collected to evaluate their mean size, D_{32} (Section 2.7.1), during 15 days. The stability evaluation was done considering the ratio between the mean size measured at final time (D) to the initial mean size (D_0).

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