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Effect of High Hydrostatic Pressure, alkaline and combined treatments on corn starch granules metal binding: Structure, swelling behavior and thermal properties assessment

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ABSTRACT

Modification of corn starch granules to transform them into carriers for molecules of nutritional interest was assayed by alkaline and high pressure treatments. Changes in the granular structure of starches were revealed by porosimetry and swelling power studies, while confocal microscopy evaluated the mode of disruption of the granules. Both alkali and high pressure treatments increased the number of pores in the granules while the combined procedures led to a central disruption and an increased deep pore number. The high pore volume found in alkali treated starches resulted in a high swelling power and created more binding sites to load high amounts of minerals, such as zinc and magnesium, as determined by atomic emission spectrometry. The extent of gelatinization process was affected differently in treated starches. The reduction in temperature and enthalpy, associated to high pressure granular starch suspensions, was moderated by the creation of new charged interactions due to alkali addition.

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1. Introduction

High Hydrostatic Pressure (HHP) is an emerging non-thermal technology that, while meeting consumer requirements for fresher taste, minimally processed foods, can cause an important reduction of their microbial load. Although complete spore destruction solely by hydrostatic pressure has not been achieved and combination with heat or other physical or chemical treatments has been proposed (Molina-García, 2002), the effect of pressure on vegetative microbial cells is considered as largely equivalent to that of heat pasteurization, as

spores also survive this process (Considine et al., 2008; Drake et al., 1997; Tauscher, 1995). This technology involves the use of liquids (water is the usual pressure transmission medium) to convey pressure at 100–800 MPa (generally no less than 100 MPa) to treat food materials; most pressure levels used in commercial applications range from 200 to 600 MPa, depending on the product (Mújica-Paz et al., 2011).

Corn starch, a typical cereal origin product, can be completely gelatinized under High Hydrostatic Pressure treatment for a relatively short time at or near room temperature. Buckow et al. (2007) found that at 30 °C, maize starch was gelatinized completely after 30 min at 650 MPa. However, corn starch still retained some degree of integrity after being

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treated under 600 MPa, for 30 min at 20 °C (Oh et al., 2008). Also, a pressure of 600 MPa for 30 min led to a complete gelatinization of rice starch granules (Li et al., 2012). Meanwhile, when low pressure levels are employed, gelatinization is not complete and starch granular integrity and independence are partially preserved. Corn starch treated at 400 MPa, for 30–40 min at mild temperatures between room conditions and 40 °C, presents a mixed character preserving many of the native granule properties (granular integrity, reduced inter-granular interactions) while acquiring some of those of gelatinized starch (such as the partial substitution of internal hydrogen bridges by those with water) (Fernández et al., 2008; Pei-Ling et al., 2010). These last properties can be observed as a reduction in the enthalpy of gelatinization determined by differential scanning calorimetry (DSC) (Deladino et al., 2015). The decrease in enthalpy is attributed to the non-gelatinized starch fraction, which can also be related to its digestibility or resistant character (Papathanasiou et al., 2015).

Pores at the surfaces of intact starch granules, internal cavities at the granule hilum and channels connecting both, are architectural features that could influence granule reactions (Huber and BeMiller, 2000). Porous modified starches possess ligand slow-release characteristics compared with native starch, due to their adsorbent characteristics (Nagata et al., 2001; Weirong and Huiyuan, 2002). In a previous work, porosimetry analysis helped understand the effect of different high pressure treatments on corn starch (Deladino et al., 2015). Porosity of starch carriers was higher in HHP treated samples compared to the native starches, thus an increased exposed surface area suggested higher potential binding sites, which could be occupied by active compounds. The ability of these modified starch granules as carriers for small molecules of nutritional interest, such as oligoelements ions and antioxidant compounds has been successfully tested (Deladino et al., 2015; Fernández et al., 2008; Teixeira et al., 2015). The amount of yerba mate antioxidant compounds, an herbal aqueous extract rich in chlorogenic acid (isomers and esters), caffeic acid and rutin, or metals ions that could be bound increased substantially over native starch with large differences in the incorporation level depending on the pressure treatment and ligand addition sequence (Deladino et al., 2015; Teixeira et al., 2015). It was shown that adding ligands to previously HHP treated starch is less efficient than to bind them to starch during pressure treatments (Deladino et al., 2015). However, the former method is considered more suitable for industrial application, as pressure treatment and binding become separate operations and the modified starch can be dried and stored before proceeding to binding.

Alkali-treated starches are promising as metal binders because of the partial ionization of hydroxyl groups. The treatment would turn alkali starch into a cation exchanger: the sodium counter ions have less affinity to the resultant starch than multivalent metal ions and are easily replaced. Tang and Liu (2012) studied the acidity of caustic digested starch and its role in starch adsorption on mineral surfaces. They measured the zeta potentials of starch digested with 0.25% NaOH, 0.25% KOH or distilled water at room temperature and found that the isoelectric points of the causticized starch shifted towards more acidic pH than the native starch treated by distilled water. Moreover as the concentration of sodium hydroxide increases (0.15%, 0.25% and 0.5%), the isoelectric points are increasingly lower. Thus, caustic treatment of starch caused the formation of surface functional groups such as carboxylic groups (–COOH). The negative surface charge of the treated starch probably arose from the dissociation of protons from these functional groups, as also observed by Niemelä (1990).

Several studies of starches from different botanical origins contributed to the knowledge of alkali effects on starch granules structure and how these modifications could alter the swelling power, the gelatinization process and, as a consequence, the rheological properties of suspensions (Maher, 1983; Mondragón et al., 2004; Nor Nadiha et al., 2010; Roberts and Cameron, 2002; Wang et al., 2012; Yamamoto et al., 2006). However, works discussing the combined effects of alkali addition and high pressure on starches are limited (Wang and Copeland, 2012). In the present work, corn starch modified by alkaline treatment was compared with native and high pressure modified starches. In addition, a combination of the two procedures (alkali and pressure treatments) was analyzed. The efficiencies of the different treatments

towards magnesium and zinc binding were explored, as well as several structural observations: porosimetry, swelling power, X-rays structure, calorimetric parameters and confocal microscopy granular image.

2. Experimental

2.1. Materials

Corn starch was purchased from Molinos Río de La Plata (Argentina). Sodium hydroxide (NaOH), magnesium sulphate heptahydrate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) and zinc sulphate monohydrate ($\text{ZnSO}_4 \cdot \text{H}_2\text{O}$) were all purchased from Panreac (Spain). Blue dextran ($\text{MW} = 2 \times 10^6$) and fluorescein isothiocyanate (FITC) were both purchased from Sigma–Aldrich (USA). Nitric acid (65%), chloridic acid (32%), fluorhydric acid (40%) and phosphoric acid (85%) were all purchased from Merck (Germany).

2.2. Preparation of starch carriers

Carrier systems were native corn starch (S), alkaline corn starch (SA), high pressure treated starch (HPS) and alkaline-high pressure treated starch (HPSA).

SA was obtained by suspending 10 g/100 mL of corn starch in a NaOH solution with a final NaOH concentration of 0.1 g/100 g deionized water (Milli-Q, Millipore Inc. Bedford, MA, USA), under agitation for 1 h at 25 °C.

HPS was obtained by suspending 10 g/100 mL of the native corn starch in deionized water (Milli-Q, Millipore Inc. Bedford, MA, USA), this suspension was treated using a High Hydrostatic Pressure (HHP) Pilot Food Processor (Stansted Fluid Power LTD. Model FP 571000:9/2C, UK) operated under 400 MPa, for 35 min, at an initial vessel temperature of 38 °C and reaching a final process temperature of 40 °C. These conditions were selected based on different assays performed in the lab (data not published) and conditions applied in previous works (Deladino et al., 2015; Fernández et al., 2008; Teixeira et al., 2015).

For HPSA samples, the SA suspensions were subjected to the HHP treatment as described above.

All treated starches were dried overnight in a convection oven at 35 °C (Thermo Scientific, Germany). The dried samples were then crushed with a mortar and stored at room temperature in hermetic boxes.

2.3. Preparation of starches loaded with minerals

Mineral salt solutions, MgSO_4 and ZnSO_4 were dissolved at 0.05 M concentration in 10 g/100 mL S, SA, HPS and HPSA starch aqueous suspensions. All suspensions were agitated in an orbital shaker (Orbit Environ Shaker, Lab Instruments, USA) at 25 °C and 180 rpm for 15 h. In the case of samples with both salts, 0.05 M of each one was employed. Then the samples were centrifuged (Rolco, USA, 20 min, 300 g) and the supernatants were discarded. Samples were dried in an oven at 30 °C. The obtained products were powdered in a mortar and stored in hermetic boxes. These samples will be named in general as “active starches”; the specific nomenclature with the detail of the applied treatments and the added minerals will be referred as: native (S_{Zn} , S_{Mg} , S_{ZnMg}), alkali treated (SA_{Zn} , SA_{Mg} , SA_{ZnMg}), HHP treated (HPS_{Zn} , HPS_{Mg} , HPS_{ZnMg}) and alkali-HHP treated (HPSA_{Zn} , HPSA_{Mg} , $\text{HPSA}_{\text{ZnMg}}$) starches.

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