



High pressure processing (HPP) of pea starch: Effect on the gelatinization properties



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ABSTRACT

High pressure processing (HPP), an emerging technology, can be used to promote gelatinization of starch granules. This phenomenon is highly dependent on the source of starch, pressure level, time and temperature applied as well as the dispersion medium. This work evaluated the effect of HPP (up to 600 MPa/15 min/25 °C) on particle size distribution, optical microscopy, differential scanning calorimetry and pasting properties of pea starch. Results showed no difference between control samples and processed ones up to 400 MPa (water dispersion) or all samples dispersed in ethanol, except for the thermal properties at 400 MPa that showed 31% of gelatinization in water dispersion. Samples processed at pressures higher than 500 MPa showed changes on particle size and distribution (increase at 500 MPa and a slight reduction at 600 MPa), and no detected gelatinization enthalpy at DSC. The optical microscopy observation indicated that HPP (>400 MPa) caused the loss of birefringence. Regarding the pasting properties, the initial viscosity increased from 8 cP at 0 MPa to 34 cP at 600 MPa. All results indicated that HPP can be used to promote “cold gelatinization” on pea starch water dispersion, achieving a specific technological profile and possibly leading to new ingredients.

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1. Introduction

Starch is the main carbohydrate reserve in higher plants and the most important source of energy for humans. Industrially, it has been widely used for numerous applications in various industries, including food and non-food, due to its functional properties, such as dispersion of ingredients, texturizing agent, fat replacer, and mouth feel enhancer (Adebawale, Afolabi, & Olu-Owolabi, 2005; Perez-Pacheco et al., 2014; Wang & Copeland, 2013).

Starch structure and properties of phase transitions are important once they influence viscosity, appearance, texture, water-holding capacity and enzyme digestibility of processed starch based food products (Wang & Copeland, 2012). The physicochemical properties of the starch and thus its applications depend on factors such as the amylose/amylopectin ratio, granule size and shape, degree of polymerisation, diffraction pattern and the differences in crystalline/amorphous regions of granules, as well as its

botanical origin and source (Błaszczak et al., 2003; Sankhon et al., 2014).

Pea starch is mainly available as a by-product from pea protein extraction, being a relative cheaper source of starch compared to wheat and potato. Pea starch is characterized by its high amylose content (35–65%), fast retrogradation, resistance to shear thinning and high resistant starch content (Wang & Copeland, 2015). Due to its high amylose starch percentage, pea starch is mainly industrially used to obtain flexible films with good mechanical properties and gas barrier (Ratnayake, Hoover, & Warkentin, 2002).

Chemical and physical modifications of starch are commonly employed to obtain starches with special functional properties, increasing its range of use. Although chemically modified starches are available for industrial uses, most industries (especially food and pharmaceutical industries) prefer starches that have been physically altered (heat, moisture, shear, radiation, high pressure processing) due to their relative safety (Adebawale et al., 2005).

High pressure processing (HPP) is a non-thermal emerging technology that subjects a product to high pressures (up to 1000 MPa) for a controlled time and temperature. HPP affects only non-covalent bonds and can cause serious structural damage to biopolymers,

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including protein denaturation and starch gelatinization (Balny, Masson, & Heremans, 2002; Hu et al., 2011). In this context, HPP has been employed to gelatinize or physically modify different types of starch dispersions (Li et al., 2011, 2012; Yang, Gu, & Hemar, 2013). Starch granules could be gelatinized completely at room temperature by HPP and the impact of the process in starch gelatinization depends markedly on starch type, treatment pressure, temperature, time and water content (Bauer & Knorr, 2005; Li et al., 2011, 2012). All data relative to HPP on starch were obtained in aqueous media; being not previously established if the HPP can induce changes on starch dispersed in other dispersion media, or if water (hydration) has a central role in the modifications observed in starches after HPP.

Several studies have been carried out with starch processed by HPP (Li et al., 2011, 2012; Yang et al., 2013), however, nothing has been done to verify its effects on Particle Size Distribution (PSD), optical microscopy, Differential Scanning Calorimetry (DSC) and pasting properties on the same study, which disrupts the well understanding of the phenomenon on the starch. Additionally, there are studies on the structure and functionality of starches from peas (Bogacheva, Morris, Ring, & Hedley, 1998; Chung & Liu, 2012; Ratnayake, Hoover, Shahidi, Perera, & Jane, 2001; Wang, Sharp, & Copeland, 2011), but little information is available for starches from peas processed by HPP. In respect to that, Le Bail et al. (2013) observed that HPP (500 MPa; 20 min; 20 or 40 °C) induced gelatinization of several starches including pea starch, lacking a deep evaluation about the process impact on the pea starch molecular structure. Considering the lack of information about HPP effects on pea starch and on starches dispersed in non-aqueous media, this work aimed to evaluate the impact of HPP on pea starch dispersed in water and ethanol by using structural and functional evaluation of the processed sample.

2. Materials and methods

2.1. Pea starch and dispersions preparation

Pea starch (33% of amylose) was obtained from Emsland-Stärke Group - Food Division, Germany. Starch samples were prepared using 4% (w/w) of starch dispersed in distilled water or in ethanol (99.9%), stirred at 25 °C (using magnetic mixer) within 24 h prior to processing.

2.2. High pressure processing (HPP)

A high pressure equipment (QFP 2L - 700 Avure Technologies, OH, USA) with a chamber of 2 L volume, maximum pressure work of 690 MPa and temperature controlled from 10 to 90 °C was used in the assays. Samples were packaged in sealed LDPE-Nylon-LDPE bags (16 µm thickness - TecMaq, Brazil). Processes were performed at 300, 400, 500 and 600 MPa for 15 min at 25 °C. The pressurization rate was about 6 ± 0.5 MPa/s; the depressurization time was almost instantaneous. After process, all samples were distributed in glass petri dishes, cooled down quickly, lyophilized (water dispersion) or air dried (ethanol dispersion) and stored before analysis. All processes were carried out in triplicate.

2.3. Visual observation and optical microscopy observation

Visual observation was conducted in graduated cylinders of 25 mL, all samples were shaken for 1 min, and placed on a table to rest during 1 min, and a photo was taken showing samples side by side, for comparison purpose.

Optical microstructure was observed using an optical microscope (Carl Zeiss Jena, Carl Zeiss Microimaging GmbH, Germany) with an 20x, 40x or 100x objective and 10x optovar, coupled to a

digital camera and software (EDN2 Microscopy Image Processing System). Before the observation, a small amount of sample was carefully placed on a glass slide, and placed a droplet of distilled water and covered with a cover glass.

2.4. Particle Size Distribution (PSD) analysis

PSD was evaluated by light scattering (Malvern Mastersizer 2000 with Hydro 2000s, Malvern instruments Ltd, UK). A little amount of dry sample was slowly added into a sample compartment, previously filled with ethanol (99.9%; room temperature), until obscuration reaches values around 10. The mean diameter was evaluated based for both particle volume ($D[4,3]$; Equation (1)) and the particle surface area ($D[3,2]$; Equation (2)). This is useful since the particles are not ideal spheres, and $D[3,2]$ is more influenced by smaller particles, whilst $D[4,3]$ is more influenced by larger ones (Bengtsson & Tornberg, 2011; Lopez-Sanchez et al., 2011). These analyses were carried out in triplicate.

$$D[4,3] = \frac{\sum_i n_i d_i^4}{\sum_i n_i d_i^3} \quad (1)$$

$$D[3,2] = \frac{\sum_i n_i d_i^3}{\sum_i n_i d_i^2} \quad (2)$$

2.5. Differential Scanning Calorimetry (DSC)

Thermal transition of starch samples was evaluated using a DSC (TA Instruments, New Castle, DE, USA). Samples (control and processed) were weighed (3 µg) in aluminium pans and water (7 µL) was added. Then, the pans were sealed, rested for 30 min at room temperature and heated at temperatures from 30 °C to 95 °C at the rate of 10 °C min⁻¹. An empty pan was used as reference, and the DSC equipment was calibrated with indium for temperature and heat capacity. From the DSC data it was obtained the thermal transitions of starch dispersions - according to the parameters T_0 (onset), T_p (peak gelatinization) and T_c (conclusion) - ΔH value (referred to enthalpy of gelatinization), temperature range ($\Delta T = T_c - T_0$) and degree of gelatinization (%G), that was calculated using the following Equation (3) (Błaszczak et al., 2007):

$$\%G = \left(\frac{\Delta H_{cs} - \Delta H_{ps}}{\Delta H_{cs}} \right) \cdot 100\% \quad (3)$$

Where ΔH_{cs} and ΔH_{ps} were the gelatinization enthalpies of control starch and pressurized starches, respectively.

2.6. Pasting properties

The pasting properties of pea starch samples were evaluated according to the method 162 of ICC (1996) using a Rapid Visco Analyser (RVA) (model RVA 4500, Peter Instruments, Warriewood, Australia) and the curves were analysed by the software TCW3.15.1.255 through the profile 'Extrusion-1' and 3.0 g (db) of starch sample. The parameters analysed were the viscosity (cP) and the pasting temperature (°C). The 'Extrusion-1' profile was chosen by analysing the cold viscosity.

2.7. Statistical evaluation

When relevant, the effect of pressure was evaluated by using the

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