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Chemical composition and functional properties of native chestnut starch (*Castanea sativa* Mill)

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ABSTRACT

Starch isolation methods can change their physico-chemical and functional characteristics hindering the establishment of a starch-food functionality relation. A simple high yield and soft isolation method was applied for chestnut (*Castanea sativa* Mill) starch consisting in steeping and fruit disintegration in a 25 mM sodium bisulfite solution and purification by sedimentation. Starch integrity, physico-chemical composition, morphology and functional properties were determined, being observed significant differences from previous described methods for chestnut starch isolation. The X-ray pattern was of B-type, with a degree of crystallinity ranging from 51% to 9%, dependent on the starch moisture content. The onset, peak, and conclusion gelatinization temperatures were 57.1 °C, 61.9 °C and 67.9 °C, respectively. Total amylose content was 26.6%, and there was not found any evidence for lipid complexed amylose. Swelling power at 90 °C was 19 g/g starch, and the amount of leached amylose was 78% of the total amylose content. Native chestnut starch presents a type B pasting profile similar to corn starch but with a lower gelatinization (56.1 °C) and peak viscosity (79.5 °C) temperatures, making native chestnut starch a potential technological alternative to corn starch, especially in application where lower processing temperatures are needed.

1. Introduction

Chestnut kernels (*Castanea sativa* Mill.) are a highly appreciated seasonal nut in the Mediterranean countries. They are mainly consumed fresh, after cooking, with roasting, boiling or frying being the most common cooking methods. Although being a highly perishable product, nowadays chestnuts can be found on the market all around the year due to the availability of frozen and boiled frozen chestnuts. Other important chestnut products are available on the market, among them the high added value and highly appreciated "Marrons Glacés" (Comba, Gay, Piccarolo, & Aimonino, 2009), and chestnut flour obtained by grinding dried chestnuts, used for valorization of small chestnuts or chestnuts with double embryos (Sacchetti, Pinnavaia, Guidolin, & Rosa, 2004). Chestnut flour is then used as a confectionery paste for producing desserts and jams. It is evident that a step of heat treatment of whole chestnuts or chestnut flour is always used before consumption. The heat treatments, like cooking, change considerably the sensorial and nutritional properties of chestnuts, many of these changes being directly or indirectly related with starch gelatinization. For example, chestnut cooking results in large changes in the macromolecular structure of starchy material and these are correlated with changes of digestibility (Pizzoferrato, Rotilio, & Paci, 1999). For cooked chestnuts, besides its sweetness and color, their texture like firmness and elasticity are important quality attributes (Mellano, Beccaro, Bounous, Trasino, & Barrel, 2009). Starch is one of the main components of chestnut kernels (*C. sativa*), accounting for approximately 50% of the chestnut kernel dry matter (Borges, Gonçalves, de Carvalho, Correia, & Silva, 2008; Vasconcelos, Bennett, Rosa, & Ferreira-Cardoso, 2009; Pereira-Lorenzo, Ramos-Cabrer, Díaz-Hernández, Ciordia-Ara, & Rios-Mesa, 2005), so it is expected that the quality attributes and behavior during industrial processing and transformation will be related to the physico-chemical and functional properties of starch from the different chestnut cultivars available, as observed for other starchy foods. For example, the quality of cooked rice is related with its starch chemical composition and properties, with cooked rice with low amylose content being soft and sticky, while rice with high amylose content being firmer and fluffy (Juliano, 1985). Also starch gelatinization during hydrothermal treatment of cassava may play an important role in defining the final characteristics of the cooked product (Beleia, Butarelo, & Silva, 2006).

Chestnut starch has been previously isolated from oven-dried chestnut flours either from *C. sativa* (Correia & Beirão-da-Costa, 2010; Demiate, Oetterer, & Wosiacki, 2001), *Castanea crenata* S. (Yoo, Lee, Kim, & Shin, 2012) and *Castanea mollissima* Bl. (Zhang,

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Chen, & Zhang, 2011), and in this last work, also from freeze-dried chestnut flour. The drying method clearly affected the properties of the Chinese chestnut starches, both in composition, physico-chemical, thermal, pasting, and functional characteristics (Zhang et al., 2011). Furthermore the starch isolated from *C. sativa* flours and characterized, was obtained from an oven dried chestnut flour at 60 °C during approximately 24 h (Correia & Beirão-da-Costa, 2010, 2012; Correia, Leitão, & Beirão-da-Costa, 2009) using different isolation methods (Correia & Beirão-da-Costa, 2010). The drying process at 60 °C changed significantly the chemical composition and functional characteristics of the chestnut starch (Correia & Beirão-da-Costa, 2010, 2012; Correia et al., 2009), as also observed previously by other authors (Attanasio, Cinquanta, Albanese, & Matteo, 2004). The starch chemical composition and functional properties of the chestnut starch previously described are therefore not representative of the native chestnut starch. Moreover the main method used in all purification procedures of chestnut starch has been the alkaline steeping method (Correia & Beirão-da-Costa, 2010; Demiate et al., 2001; Yoo et al., 2012). There are several reported methods for starch isolation and purification in the literature (Liu, 2005, chap. 7). The preferred method for a particular source is dependent on the easiness of releasing the starch granules from the plant cell matrix without damaging their structure, the amount and nature of contaminants present, and the simplicity and economy of the procedure. Although the alkaline stepping method is superior for rice starch isolation due to the presence of insoluble protein (Adoracion, Li, Okita, & Juliano, 1993), it is also known that the alkaline extraction of starch can significantly change the physico-chemical properties of the isolated starch (Cardoso, Putaux, Samios, & da Silveira, 2007; Thys et al., 2008). The objective of this work was to apply a simple and soft isolation and purification method for chestnut (*C. sativa*) starch in order to maintain its native physico-chemical composition, a requirement needed to study native chestnut starch functional properties aiming in the near future to correlate the physico-chemical and functional properties of starch with the quality attributes of processed chestnuts from different cultivars. Functional properties of native chestnut starch were compared with commercial potato and corn starches in order to evaluate potential technological advantages of the native chestnut starch.

2. Materials and methods

2.1. Materials

Chestnut (*C. sativa* Mill var. Longal) fruits were collected from Castanha da Terra Fria, a Protected Designation of Origin region of Portugal. Chestnut fruits harvested at a mature stage, were dehulled, chopped, freeze-dried and then milled. Commercial grade corn (Sigma, S-4126) and potato (Ramazzotti, S. A., Portugal) starches were used as standards when necessary.

2.2. Starch extraction

Chestnut starch was isolated from chestnut flours obtained from freeze-dried chestnuts by steeping in a 25 mM sodium bisulfite aqueous solution followed by sedimentation in water. The chestnut flours (25 g) were steeped in a 25 mM sodium bisulfite solution (250 mL) for 1 h at room temperature and further disintegrated using a Waring blender for 3 min. The starch was separated from cell debris by sedimentation, and the top brown mucilaginous layer was scrapped off the surface. The sedimentation process in water was repeated eight times after re-suspension of the starch slurry in 250 mL of water. The starch precipitate obtained was filtered, washed with water (250 mL) followed by washing with ethanol (250 mL) and dried in the air at room temperature.

2.3. General composition of chestnut starch

Total and damaged starch were determined according to the AACC methods 76.13 and 76-31, respectively, using K-TSTA and K-SDAM kits of Megazyme (Megazyme International Ireland Ltd., Co.). Total amylose and free amylose contents were determined by iodine binding according to Chrasil (1987), with and without lipid extraction with 85% aqueous methanol, respectively. Lipid-complexed amylose was calculated as the difference between total and free amylose content. Nitrogen and phosphorous content of the starch was determined after acid digestion according to the method of Novozamsky, Houba, van Eck, and van Vark (1983), by the methods described by Houba, Novozamsky, and Temminghoff (1994) and Coutinho (1996). Protein content was obtained by multiplying the nitrogen content by 6.25. Water content was determined by oven drying of the starch at 104 °C until constant weight (AOAC, 2000).

2.4. Determination of granule structure and size of chestnut starch by scanning electron microscopy

Chestnut starch morphology was analyzed using the FEI Quanta 400 Scanning Electron Microscope (FEI Company, USA) in environmental mode at 6 mbar using a Large Field Detector (LFD). Starch samples were suspended in water during 24 h with occasional stirring. One drop of the starch suspension was applied on carbon glue and let dry at room temperature. An accelerating voltage of 30 kV was used. The granule size (diameter) was obtained using the Image Tool software Version 3.0 for windows (UTHSCSA, 2002).

2.5. Color evaluation

Chestnut starch color was evaluated with a Chroma Meter CR-300 Minolta (Osaka, Japan). CIE Lab coordinates were obtained using D65 illuminant a 10 observer as reference system. L^*, a^* and b^* parameters were calculated from the average of five color measurements. The equipment was calibrated with a white standard ($L^* = 97.71$; $a^* = -0.59$ and $b^* = 2.31$). For reference, commercial corn (Sigma, S-4126) and potato (Ramazzotti, S. A., Portugal) starches were used. From these values, chroma was calculated using Eq. (1):

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (1)$$

saturation difference (ΔC^*) between chestnut and reference standards was calculated as $C^*_{\text{chestnut starch}} - C^*_{\text{standard}}$. Hue difference ($\Delta H^*$) was calculated according to Eq. (2):

$$\Delta H^* = \sqrt{(\Delta E^*)^2 - (\Delta L^*)^2 - (\Delta C^*)^2} \quad (2)$$

where ΔE^* is the total color difference calculated according to Eq. (3):

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (3)$$

2.6. X-ray pattern and relative crystallinity

Powder X-ray diffraction (XRD) data were collected at room temperature with the PANalytical X'Pert Pro diffractometer, equipped with X'Celerator detector and secondary monochromator in $\theta/2\theta$ Bragg–Bentano geometry. The measurements were carried out using a CuKα radiation ($\lambda_{\alpha 1} = 1.54060$ Å and $\lambda_{\alpha 2} = 1.54443$ Å) in a 4–60° 2θ angular range, a step width of 0.017° and a counting time of 100 s/step.

The relative degree of crystallinity was determined following the method previously reported by Huang et al. (2007) using the following equation (Eq. (4)):

$$X_c = \frac{\sum_{i=1}^{i=n} A_{ci}}{A_t} \times 100 \quad (4)$$

where A_c is the crystallized area on the X-ray diffractogram and A_t is the sum of the crystallized and amorphous area on the diffractogram. For the analysis of the effect of the water content on the relative crystallinity of starch, the water content of the starch was adjusted by equilibration with saturated salt solutions (NaCl – %ERH/100 = 0.7541; $Mg(NO_3)_2 \cdot 6H_2O$ – %ERH/100 = 0.5447 and NaOH – %ERH/100 = 0.0698) or water in a close environment, until constant weight, and analyzed as described above.

2.7. Thermal analysis

Gelatinization parameters of chestnut starch were measured by differential scanning calorimetry (DSC SETERAM 131, France). The instrument was calibrated using indium and deionized distilled water as standards. Samples (7–8 mg dry basis) were placed in a stainless pan at room temperature, added 24 mg of deionized water and sealed in the sample pan press. The sealed pan was left still for 12 h at room temperature to allow complete starch hydration. Then, the pan with the sample was placed in the calorimeter and heated from 25 to 120 °C (the heating rate was 10 °C/min) with a nitrogen flow of 30 mL/min. An empty pan was used as a reference. Onset temperature (T_0), peak temperature (T_p), conclusion temperature (T_c) and gelatinization enthalpy ΔH (J/g of dry starch) were determined. The total gelatinization temperature range (R) and peak height index (PHI) were determined according to Eqs. (5) and (6) (Krueger, Knutson, Inglett, & Walker, 1987):

$$R = 2 \times (T_p - T_0) \quad (5)$$

$$\mathrm{PHI} = \frac{\Delta H}{T_p - T_0} \quad (6)$$

2.8. Chestnut starch swelling behavior

Swelling behavior of the isolated and purified chestnut starch was determined according to the method described by Srichuwong, Sunarti, Mishima, Isono, and Hisamatsu (2005). Briefly, starch suspensions (1%, w/w) were heated at 50, 60, 70, 80 and 90 °C during 30 min with re-suspension every 5 min. After this time, the tubes were rapidly cooled to room temperature by immersion in tap water and centrifuged at 4500 rpm for 15 min. The supernatant was decanted and the swollen starch precipitate was weighted. The total carbohydrate content of the supernatant was determined by the phenol–sulfuric acid method (Dubois, Giles, Hamilton, Rebers, & Smith, 1956), and the solubilized starch (SS) was calculated as the ratio between the total carbohydrates in the supernatant in relation to the starch dry matter. The swelling power was calculated according to the following equation (Eq. (7)):

$$\text{swelling power (g water/g starch)} = \frac{\text{weight of precipitates}}{\text{starch (dry weight)} \times (1 - \%SS/100)} \quad (7)$$

The amount of leached amylose (g/100 g starch dry weight), occurring during starch swelling was determined by the colorimetric method described by Chrasil (1987), using amylose from potato (Fluka, ref. 10130) as standard.

Table 1
Chemical composition of the native chestnut starch isolated from var. Longal.

Component	
Humidity	12.4 ± 0.3%
Total starch	93.2 ± 1.1%
Apparent amylose	24.7 ± 0.9%
Amylose	26.6 ± 1.3%
Damage starch	17.6 ± 1.0%
Phosphorous	0.00110 ± 0.00001%
Protein	0.48 ± 0.02%
L^*	92.88 ± 0.24
a^*	−0.34 ± 0.03
b^*	4.38 ± 0.22

2.9. Pasting properties

The pasting properties of chestnut starch were studied using a starch pasting cell attached to a controlled stress rheometer (AR-1000, TA Instruments, UK). Viscograms of starches were monitored using 12% (w/v) starch-water suspensions. The sample was stirred strongly (160 s^{-1}) for 30 s at 30 °C before switching the shear rate to 60 s^{-1}, which was maintained until the end of the test. The sample was heated from 30 °C to 90 °C at 15 °C/min and the temperature was held at 90 °C for 5 min. Subsequently, the sample was cooled to 30 °C at 15 °C/min and held at 30 °C for 5 min. Viscosity data were recorded over time by the TA data analysis software provided by the instrument's manufacturer. For comparison purposes two commercial starches, one from corn (Sigma, S-4126) and another from potato (Ramazzotti, S. A., Portugal) were also analyzed. To compare the pasting properties of the different starches, gelatinization temperature (GT), taken as the temperature at which viscosity begins to rise; peak viscosity (PV), taken as the highest viscosity achieved during heating; hot paste viscosity (HPV), taken as the viscosity value at the end of the isothermal period at 90 °C; cold paste viscosity (CPV), taken as the viscosity value at the end of the isothermal period at 30 °C; breakdown (PV–HPV) and setback (CPV–HPV) were calculated from the viscosity-temperature versus time curves obtained.

2.10. Statistical analysis

The results obtained were subject to a one-way analysis of variance (ANOVA) test using the Statistica 8.0 software. Significantly different average values were tested using the Fischer least significant differences. The level of significance used for all the statistical tests was 95%.

3. Results and discussion

3.1. Isolation, purification and chemical characterization of native chestnut starch.

The procedure applied for chestnut starch isolation is outlined in Fig. 1 and its chemical composition is shown in Table 1. Although the chestnut starch isolated in this work was obtained from freeze-dried chestnut flours, it can also be obtained from fresh chestnuts without any drying method, but in this case chestnut fruits must be processed as soon as possible due to their high perishability. The use of water instead of the 25 mM sodium bisulfite aqueous solution during steeping and fruit disintegration was excluded due to the development of a brown color throughout the solution, probably due to the action of chestnut polyphenol oxidase on chestnut polyphenols, which yielded a starch precipitate with a brownish color (results not shown). The use of a 25 mM sodium bisulfite aqueous solution during steeping and fruit disintegration was enough for avoiding this undesirable event. This method allowed an almost

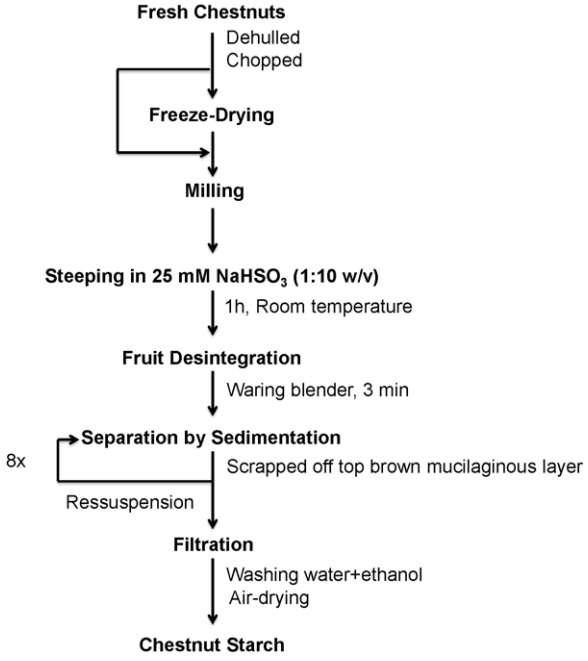

Fig. 1. Isolation and purification scheme of native chestnut starch.

quantitative recovery (94%) of the starch present in chestnut kernel (chestnut starch content – 51.65 ± 4.80%, de Vasconcelos et al., 2009), with a high purity (93%) and higher integrity of the starch granules (18% of damaged starch) when compared with previous methods used for chestnut starch isolation presenting a high amount (>40%) of damaged starch (Correia & Beirão-da-Costa, 2010, 2012). The purity of the starch isolated can also be confirmed by the low amount of protein present in the starch preparation (Table 1). SEM images (Fig. 2) show that the starch granules are round and oval, presenting a smooth surface, and no fracture being observed. Native chestnut starch granules had a mean diameter of 11 μm, with a range from 4 to 21 μm. These values are in accordance with previous results obtained for starch granules isolated from *C. sativa* (Correia, Cruz-Lopes, & Beirão-da-Costa, 2012), *C. crenata* (Yoo et al., 2012) and *C. mollissima* (Zhang et al., 2011).

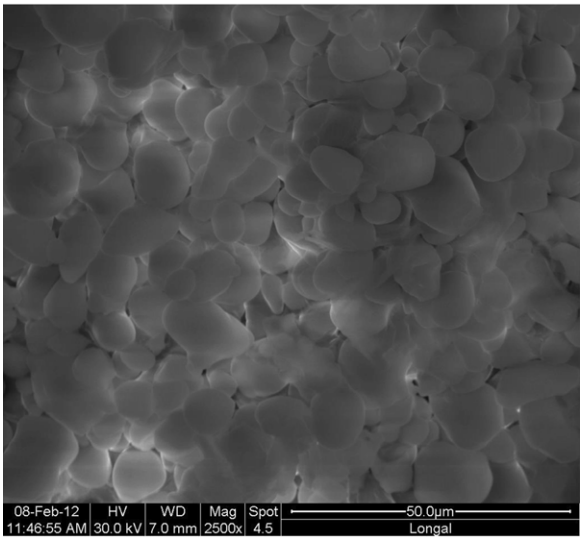

Fig. 2. Scanning electron micrographs of native chestnut starch granules (var. Longal) magnified by 2500×.

The amount of apparent and total amylose of chestnut starch isolated from var. Longal, are not significantly different ($p < 0.05$, Table 1), being no evidence for the presence of amylose complexed with lipids in chestnut starch. The total amylose content of the chestnut starch was 26.6%, a value lower than that found by Correia and Beirão-da-Costa (2010) and Correia, Nunes, and Beirão-da-Costa (2012) for *C. sativa* (32.9% for fresh chestnuts, and 51–57% depending on the extraction method) Yoo et al. (2012) for *C. crenata* (29.6%), higher than that found by Zhang et al. (2011) for *C. mollissima* (18.3%). These differences can be attributed to the different geographical origin of the chestnuts, different methods used for amylose quantification, or due to the different methods used for starch isolation. The amount of phosphorous present in native chestnut starch was low (Table 1).

Chestnut starch presented a white color to the naked eye. The isolated chestnut starch presented a high L^* value (Table 1), although lower than that of commercially available corn ($L^* = 98.3$) and potato starches ($L^* = 95.4$). Chestnut starch is only slightly more saturated ($\Delta C^* = 0.05$) and slightly more yellow ($\Delta H^* = 0.72$) than corn starch, overall chestnut starch presented a slight deeper color. The same is observed when the chestnut starch color is compared to the commercial potato starch color ($\Delta C^* = 1.22$ and $\Delta H^* = 0.18$), although presenting a higher color saturation. Lightness, color and hue angle values obtained for the chestnut starch isolated with the current soft method are close to that reported for chestnut starch isolated by alkaline and enzymatic methods of oven-dried chestnut flours (Correia, Cruz-Lopes, et al., 2012).

3.2. Chestnut starch crystallinity

Starch granules present a semi-crystalline structure corresponding to different polymorphic forms, which are classified into three types, namely A, B and C, based on their characteristic and distinct X-ray diffraction patterns (Buléon, Colonna, Planchot, & Ball, 1998; Zobel, 1988). The crystallinity is exclusively associated with the packing of the amylopectin double helices, while the amorphous regions mainly represent amylose (Zobel, 1988). The packing of amylose and amylopectin within the granules has been reported to vary among the starches, with A-type polymorphs being found mainly in cereal starches, B-type found in tubers and high amylose starches, and with legume starches presenting a C-type, this last type corresponding to the coexistence of A and B-type polymorphs (Liu, 2005, chap. 7). Double helix formation of the amylopectin side chains is dependent on their particular structure, with starches containing longer amylopectin branches showing B-type diffraction pattern, while starches containing amylopectin with shorter branch length presenting A-type crystallinity (Hizukuri, 1986). Moreover, the branch pattern also affects the crystallinity type (Jane, Wong, & McPherson, 1997).

The X-ray diffraction pattern of chestnut starch is presented in Fig. 3. On the same figure it is also shown the effect of water content on the degree of crystallinity of chestnut starch. Chestnut starch X-ray diffraction pattern was characterized by peaks at diffraction angles of 5.62°, 9.81°, 10.57°, 14.20°, 14.83°, 16.97°, 19.35°, 22.02°, 23.77° and 25.78°. This X-ray diffraction pattern is characterized as B-type, like tuber starches (Buléon, Bizot, Delage, & Pontoire, 1987; Hizukuri, Kanebo, & Takeda, 1983; Zobel, 1988) and no reflections corresponding to A-type starches, reported to be present at 15.3°, 17.0°, 18.0°, 20.0° and 23.4° 2θ angles (Buléon et al., 1987; Hizukuri et al., 1983; Zobel, 1988) were observed in the X-ray diffraction pattern obtained for chestnut starch. The X-ray diffraction pattern of chestnut starch has been previously described to be of the C-type (Correia, Cruz-Lopes, et al., 2012; Yang, Jiang, Prasad, Gu, & Jiang, 2010; Yoo et al., 2012). As the proportion of A- and B-polymorphs in C-type starches and the total crystallinity in starches largely depends on the starches water content (Bogracheva, Wang,

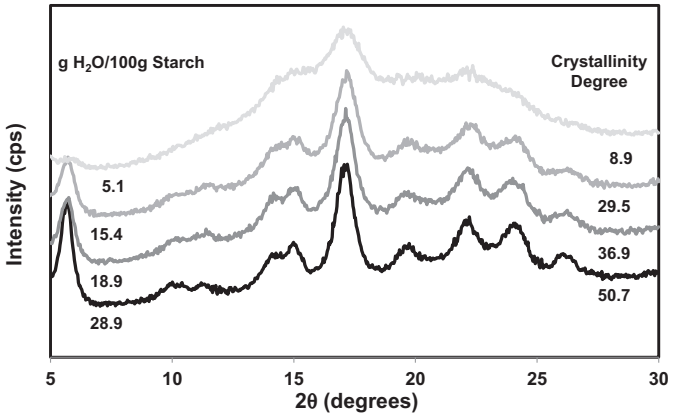

Fig. 3. X-ray diffraction pattern and effect of starch water content on the degree of crystallinity of native chestnut starch (var. Longal).

& Hedley, 2001; Bogracheva, Wang, Wang, & Hedley 2002), the X-ray diffraction patterns of chestnut starch was obtained in a wide range of water contents (Fig. 3). The lack of A-type reflections were observed for all X-ray diffraction patterns of chestnut starch with water contents ranging from 5.1 to 28.9%, excluding the possibility of the observed differences being due to the starch water content between the our and previous studies.

Starch crystallinity is an important factor for the overall granule properties. For starches with A-type polymorphs, gelatinization temperature generally tends to increase with increasing crystallinity, while the reverse being observed for starches with B-type polymorphs (Pérez, Baldwin, & Gallant, 2009, chap. 5). Chestnut starch relative crystallinity increase linearly (degree of crystallinity (%) = 1.766 * %water content (g/100 g) + 1.35; $r = 0.994$, $p < 0.0058$) with starch moisture content (Fig. 3). Water is essential for the crystallinity of starch granules. The B-type crystallites consist of left-handed parallel stranded double helices packed in hexagonal unit cells. Double helices are connected through a network of hydrogen bonds that form a channel inside the hexagonal arrangement of six double helices, with 36 water molecules per unit cell, some of which located in the channels. Water plasticizes the amorphous region of starch granules allowing the rearrangement of double helixes, and the buildup of crystalline hydrate lattices (Buléon et al., 1987, 1998; Hizukuri et al., 1983; Imberty, Buleon, Tran, & Perez, 1991; Liu, 2005, chap. 7; Rappenecker & Zugenmaier, 1981; Zobel, 1988). The degree of crystallinity for chestnut starch equilibrated at an equilibrium relative humidity of 75% was 36.9%. This value is in the range of the values found (20–35%) for other starches equilibrated at moderate equilibrium humidity's (Gidley & Bociek, 1985; Hartley, Chevance, Hill, Mitchel, & Blanshard, 1995).

3.3. Thermal properties of chestnut starch

Starch granules when heated in excess of water undergo significant structural and morphological changes including starch swelling due to water absorption, loss of crystallinity due to the amylopectin double helix dissociation, and amylose leaching to the water phase. These set of changes are generally refereed as starch gelatinization, and occur in a temperature range dependent on the starch botanical origin (Buléon et al., 1998; Charles, 2004; Liu, 2005, chap. 7; Miles, Morris, Orford, & Ring, 1985; Morris, 1990; Ring, 1985). Chestnut starch gelatinization properties are shown in Table 2. The onset (57.1 °C), peak (61.9 °C) and conclusion (67.9 °C) temperatures are lower than that reported previously for chestnut starch isolated from oven dried chestnut flours (Correia, Nunes, et al., 2012), and chestnut starches isolated from *C. mollissima* (Yang et al., 2010) and *C. crenata* (Yoo et al., 2012), although higher than

Table 2
Gelatinization properties of chestnut starch isolated and purified from var. Longal.

T_0 (°C)	T_p (°C)	T_c (°C)	ΔH_{gel} (J/g)	R (°C)	PHI (J/g °C)
57.1 ± 0.1	61.9 ± 0.5	67.9 ± 0.5	10.0 ± 0.2	9.6	2.08

that described for *C. mollissima* in another study (Zhang et al., 2011). The gelatinization enthalpies are obtained are closer to that found for starches isolated from *C. mollissima* (Yang et al., 2010; Zhang et al., 2011) and *C. crenata* (Yoo et al., 2012), but higher than that described for *C. sativa* starch (Correia, Nunes, et al., 2012).

The low gelatinization temperature range ($R = 9.6$ °C) and high PHI (2.08 J/g °C) suggest the presence of a high degree of molecular order for chestnut starch (Sandhu, Singh, & Kaur, 2004). The gelatinization temperature range and PHI obtained in this study are lower and higher, respectively, than that previously described for *C. sativa* starch (Correia, Nunes, et al., 2012) and *C. mollissima* (Zhang et al., 2011), and close to the values found for the starches isolated from *C. crenata* (Yoo et al., 2012) and another study of *C. mollissima* (Yang et al., 2010).

3.4. Swelling power, starch solubility and amylose leaching

As stated previously, the heating of starch in excess of water results in extensive swelling of starch granules. The swelling ability of starch contributes to important characteristics of most starchy food products, such as pasting and rheological behaviors. For example starch swelling has been reported to be a promising method for predicting the eating quality of Japanese white salted noodles (Konik, Miskelly, & Gras, 1993). Also the soluble amylose leached from starch granules during swelling can be determinant to the textural properties of the product (Doublier, 1981), and has been also related with the cohesiveness in cooked tubers (Moorthy, 1985).

Table 3 shows the changes in swelling power, solubilized starch and leached amylose of chestnut starch granules from 50 °C to 90 °C. As expected the temperature had a significant effect on starch swelling (ANOVA, $p < 0.000003$). The highest increment in swelling power was observed when the temperature increased from 50 °C to 60 °C, corresponding to the onset temperature of starch gelatinization observed by DSC. The maximum swelling power of chestnut starch was observed at 90 °C, corresponding to 19.24 g/g starch, a value close to that observed for corn (~21 g/g starch; Lan, Hihua, Yun, Bijun, & Zhida, 2008; Srichuwong et al., 2005) and wheat starches (18–27 g/g starch, Sasaki & Matsuki, 1998), but lower than that observed for potato (35–40 g/g starch; Singh, McCarthy, & Singh, 2006; Srichuwong et al., 2005) and cassava starches (42–71 g/g starch, Rickard, Asaoka, & Blanshard, 1991; Srichuwong et al., 2005).

The amount of solubilized starch significantly increased with the increase of temperature (ANOVA, $p < 0.00018$). The amount of solubilized starch increased linearly with the increase of temperature ($r = 0.994$, $p < 0.00056$) reaching a maximum value of 21 g/100 g

Table 3
Effect of temperature on solubilized starch, swelling power and amylose leached for native chestnut (var. Longal) starch.

Temperature (°C)	Solubilized starch (g/100 g starch)	Swelling power (g/g starch)	Amylose leached (g/100 g starch)
50	1.7 ± 0.7[a]	5.45 ± 0.15[a]	0.32 ± 0.01[a]
60	5.6 ± 0.4[b]	10.66 ± 0.06[b]	4.3 ± 0.02[b]
70	9.8 ± 1.8[c]	11.84 ± 0.44[c]	7.9 ± 0.3[c]
80	17 ± 0.5[d]	15.30 ± 0.23[d]	13 ± 0.2[d]
90	21 ± 0.8[e]	19.24 ± 0.35[e]	19 ± 0.3[e]

For each column, values with different superscripts represent significantly different means ($p < 0.05$) by post-hoc Fischer least significant differences.

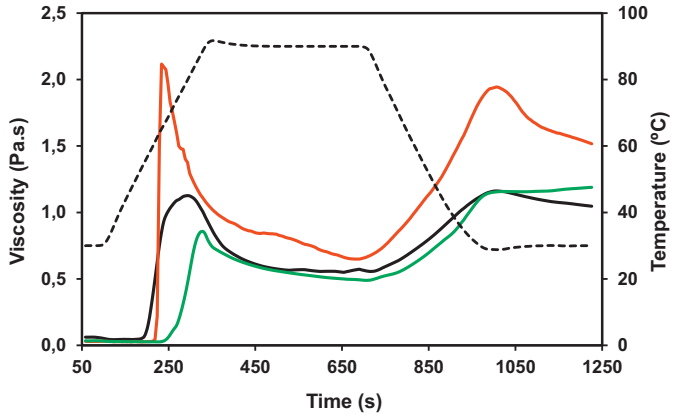

Fig. 4. Starch pasting cell-viscograms (12% [w/v]) of native chestnut (▬), potato (▬) and corn (▬) starches; (▬ ▬) temperature.

starch at 90 °C (Table 3). The amount of solubilized starch in chestnut starch is in the range of that found for corn (14–20 g/100 g starch; Lan et al., 2008; Srichuwong et al., 2005) and potato starch (~18 g/100 g starch, Srichuwong et al., 2005) and lower than that observed for cassava starch (~70 g/100 g starch, Srichuwong et al., 2005).

With the increase in temperature there is observed a linear increase (ANOVA, $p < 0.000001$; $r = 0.994$, $p < 0.00053$) in the amount of leached amylose from chestnut starch granules (Table 3). The amount of amylose leached from chestnut starch at 90 °C represents 78% of the amylose content of starch. The amount of amylose leached during starch swelling is highly correlated with the amount of solubilized starch during starch swelling (solubilized starch (g/100 g starch) = 0.9144 ± 0.06248 amylose leached (g/100 g starch) − 0.01173 ± 0.008196; $r = 0.993$, $p < 0.0007$). In fact the variation in the amount of solubilized starch during starch swelling explains 98.6% of the variation of the amylose leached, that together with the slope of the regression line (IC95% = 0.7156; 1.113) suggests that amylose is the main, if not the only carbohydrate leached during chestnut starch swelling.

The differences observed between the swelling power and solubilized starch of chestnut starch and that observed for the starches from other botanical sources may be the result of different extent of non-covalent interaction between starch molecules within the amorphous and crystalline domains, related with structural differences in the amylopectin and amylose polysaccharides and granule starch organization as for example different amylose–amylopectin ratio, chain length and molecular weight distribution, degree/length of branching and conformation (Rickard et al., 1991).

3.5. Pasting properties

The heating of a starch-water dispersion under shear above its gelatinization temperature yields starch pastes. The pasting profiles of a starch is an effective method for relating starch functionality with its structural features and access the potential industrial application in products dependent on the viscosity and thickening behavior of starch.

The pasting properties of the native chestnut starch dispersion with a concentration of 12% (w/v) and that of commercial potato and corn starch dispersions at the same concentrations are presented in Table 4. To compare the pasting properties of the different starches, gelatinization temperature (GT), peak viscosity (PV), hot paste viscosity (HPV), cold paste viscosity (CPV), breakdown (PV–HPV) and setback (CPV–HPV) were calculated from the viscosity-temperature versus time curves obtained (Fig. 4). For all starches analyzed, in the initial phase there was observed a gradual increase in viscosity with the increase in temperature (Fig. 4). Chestnut starch showed a GT of 56.1 °C, close to the onset temperature observed for the analysis of chestnut starch by DSC (57.1 °C). The GT obtained for the native chestnut starch isolated in this study was lower than that described previously for the starches isolated from *C. sativa* (Correia & Beirão-da-Costa, 2010; Correia, Nunes, et al., 2012), *C. crenata* (Yoo et al., 2012) and *C. mollissima* (Zhang et al., 2011). The chestnut starch GT was lower than that observed for potato and corn starches (Table 4). Pasting results in a significant change in the viscosity of the starch-water dispersions, as during gelatinization and under shear conditions the dispersed phase changes from a state of rigid semi-crystalline starch granules, for temperatures below the gelatinization temperature, to a dispersed phase consisting of swollen granules, partially disintegrated granules and ultimately molecularly dispersed granules, above the gelatinization temperature (Liu, 2005, chap. 7), resulting in a dispersed phase of swollen granules embedded in, and reinforcing, a continuous matrix of entangled leached amylose molecules (Ring, 1985). The initial increase in the viscosity of the starch-water suspension is explained by the loss of free water and restricted flow of water due to the increased volume of the swollen granules occupying more space.

Chestnut starch shows a peak viscosity of 1.127 Pa s, a value lower than that observed for potato starch, but higher than that observed for corn starch (Table 4). The peak viscosity often correlates with the quality of the end-product, providing additionally an indication of the viscous load likely to be encountered by a mixing cooker (Ragaee & Abdel-Aal, 2006). Peak viscosity of chestnut starch occurred at 79.5 °C, a value higher than that obtained for potato starch (65.5 °C) and lower than that observed for corn starch (89 °C). Peak viscosity and temperature are indicative of the water binding capacity of starches (Liu, 2005, chap. 7).

The decrease in viscosity observed during the high temperature holding period (breakdown viscosity) for chestnut starch was between that of potato and corn starch. The breakdown viscosity is the result of the disintegration and alignment of swollen starch granules due to the continuous shear stress at high temperatures. Breakdown viscosity can be used to predict the capacity of starch pastes used as thickeners to resist severe processing conditions (Adebowale, Sanmi, & Awonorin, 2005). High breakdown values are associated with high peak viscosities, which are related to the degree of swelling of the starch granules during heating. This is observed for the three starches studied, as the potato starch presenting a higher peak viscosity also shows a high breakdown value when compared to chestnut and corn starches.

Cold paste viscosity (CPV) for chestnut starch paste was close to that observed for corn starch paste and lower than that observed for potato starch paste, showing that chestnut starch has an equivalent capacity to corn starch to form a viscous paste. This increase in viscosity during cooling is due to the formation of an amylose network resulting in a gel structure (Miles et al., 1985). The final viscosity gives an indication of the stability of the cooled, cooked paste under low shear (Liu, 2005, chap. 7).

Setback viscosity (CPV–HPV), a measure of synaeresis of starch upon cooling of the cooked starch pastes, was lower for chestnut starch paste when compared to corn and potato starch pastes. Setback (SB) viscosity is a measure of the degree of reassociation during cooling among the starch molecules involving amylose (Charles, 2004), leached from swollen starch granules, and is generally used as a measure of the gelling ability or retrogradation tendency of starch (Singh, Bawa, Singh, & Saxena, 2009).

Starch swelling and amylose leaching are determinants for the rheological properties of starch pastes. Amylopectin contributes to the swelling of starch granules, and amylose and lipids inhibit the swelling (Loh, 1992; Morris, 1990; Rani & Bhattacharaya, 1989;

Table 4
Pasting properties of starches from chestnut (var. Longal), potato and corn.

	GT (°C)	PV (Pa s)	HPV (Pa s)	CPV (Pa s)	PV–HPV breakdown	CPV–HPV setback
Chestnut	56.1 ± 0.1[a]	1.127 ± 0.007[a]	0.551 ± 0.003[a]	1.047 ± 0.017[a]	0.576 ± 0.008[a]	0.496 ± 0.017[a]
Potato	61.1 ± 0.6[b]	2.117 ± 0.020[b]	0.649 ± 0.005[b]	1.516 ± 0.012[b]	1.468 ± 0.020[b]	0.867 ± 0.013[b]
Corn	69.9 ± 0.4[c]	0.857 ± 0.006[c]	0.491 ± 0.002[c]	1.189 ± 0.006[c]	0.366 ± 0.006[c]	0.698 ± 0.006[c]

For each column, values with different superscripts represent significantly different means ($p < 0.05$) by post-hoc Fischer least significant differences.

Yoo et al., 2009), so the differences in the structural features of amylopectin and amylose that influence the swelling power discussed earlier, will ultimately influence the starch pasting profile.

Chestnut starch, as well corn starch, presents a type B pasting behavior, with a lower peak viscosity than that observed for potato starch (presenting a type A pasting behavior) and less thinning during cooking. This behavior is the result of the presence of moderately swelling starch, leading to a lower peak viscosity but making them less fragile to the shear stress applied (Schoch & Maywald, 1968). Comparing the pasting behavior of the native chestnut starch with that found for the commercial corn starch, it can be observed that it has a similar thickening behavior but with a lower gelatinization and peak viscosity temperatures, making native chestnut starch a potential technological alternative to corn starch, especially in application needing lower processing temperatures.

3.6. Effect of the extraction and isolation protocols on the properties of chestnut starch

The chemical, physical and functional properties of the native chestnut starch isolated and purified in this work presents some significant differences from those of previously described reports on the literature on chestnut starch. Although many of the differences described can be due to chestnut species used for starch isolation, among them *Castanea mollissima* Bl. (Yang et al., 2010; Zhang et al., 2011) and *Castanea crenata* S. (Yoo et al., 2012) or to the different chestnut varieties of *C. sativa* used to isolate the starch or chestnut grown in different edapho-climatic conditions (Correia & Beirão-da-Costa, 2010, 2012; Correia, Cruz-Lopes, et al., 2012; Correia et al., 2009; Correia, Nunes, et al., 2012; Demiate et al., 2001), some of the differences are related to the sample preparation and method used for starch isolation (Correia & Beirão-da-Costa, 2010, 2012; Correia, Cruz-Lopes, et al., 2012; Correia et al., 2009; Correia, Nunes, et al., 2012; Demiate et al., 2001; Yoo et al., 2012). The most significant differences are related with the X-ray diffraction pattern, described to be of the C-type (Correia, Cruz-Lopes, et al., 2012; Yang et al., 2010; Yoo et al., 2012), amylose content (Correia & Beirão-da-Costa, 2010; Correia et al., 2009; Correia, Nunes, et al., 2012), swelling power and solubility (Correia, Nunes, et al., 2012), gelatinization temperature (Correia, Nunes, et al., 2012; Yang et al., 2010; Yoo et al., 2012) and pasting properties (Correia & Beirão-da-Costa, 2010; Correia, Nunes, et al., 2012; Demiate et al., 2001). These differences can be attributed to the heat treatment applied in order to dehydrate the chestnuts, to obtain the chestnut flour used to isolate the starch (40 °C during 24 h followed by 60 °C during ~24 h; Correia & Beirão-da-Costa, 2010, 2012; Correia, Cruz-Lopes, et al., 2012; Correia et al., 2009; Correia, Nunes, et al., 2012; oven dried but temperature and time not specified, Demiate et al., 2001), or used to dry the chestnut starch obtained (40 °C, time not specified; Yang et al., 2010). In the works were *C. sativa* starch was isolated and characterized (Correia & Beirão-da-Costa, 2010, 2012; Correia, Cruz-Lopes, et al., 2012; Correia et al., 2009; Correia, Nunes, et al., 2012) the sample pre-treatment at 60 °C induced a significant chemical change of the chestnut starch, with an increase in the amylose content and amount of damaged starch and a decrease in the amount of total starch and an increase in reducing sugars present in the chestnut flour (Correia & Beirão-da-Costa, 2012; Correia et al., 2009). This sample pre-treatment can be classified as a heat-moisture treatment, as it involved the incubation of starch granules at low moisture levels during a certain period of time, at a temperature above the glass transition temperature but below the gelatinization temperature (Jacobs & Delcour, 1998). Also the modification of starch structure due to the enzymatic activity present in the chestnut kernels, enhanced at 60 °C, is very likely (Attanasio et al., 2004; Correia et al., 2009).

Heat-moisture treatment of starch can induce the evolution of the X-ray diffraction pattern from the B- to the A- (or C-) type (Hoover & Vasanthan, 1994a; Kawabata et al., 1994; Stute, 1992). B-type polymorphs are much more susceptible to heat moisture treatments than A-type polymorphs, as the A-type pattern of cereal starches is unchanged after heat-moisture treatment (Franco, Ciacco, & Tavares, 1995; Hoover & Manuel, 1996; Hoover & Vasanthan, 1994b). Other significant changes in the physical, chemical and functional properties induced by the heat-moisture treatment include the increase of the gelatinization temperature and broadening of the gelatinization temperature range (Hoover & Manuel, 1996; Hoover & Vasanthan, 1994b; Stute, 1992) and decrease in the gelatinization enthalpies (Hoover & Vasanthan, 1994a; Stute, 1992). There is also reported a decrease in the starch swelling power and carbohydrate leaching as a result of heat-moisture treatment (Hoover & Manuel, 1996; Hoover & Vasanthan, 1994a, 1994b). The changes induced in the starch organization due to the heat-moisture treatment result in a significant change in the pasting properties of the starch, being observed a higher onset temperature of viscosity development, a lower peak viscosity, and, depending on the treatment conditions, a higher or lower end viscosity (Franco et al., 1995; Hoover & Manuel, 1996).

Also the alkaline stepping method for starch isolation has been the preferred method for chestnut starch isolation (Correia & Beirão-da-Costa, 2010, 2012; Correia, Cruz-Lopes, et al., 2012; Correia, Nunes, et al., 2012; Demiate et al., 2001; Yoo et al., 2012). Alkaline treatment of starch granules can induce the change of starch polymorphism (Cardoso et al., 2007), again with the B polymorph being more susceptible to alkaline conditions than the A polymorph (Thys et al., 2008).

4. Conclusions

Native chestnut (*C. sativa*) starch isolated in this work presented significantly different physico-chemical and functional properties than those previously described. These differences are related with the temperatures used during chestnut processing and also starch isolation methods employed. The overall procedure applied is simple and allowed to obtain chestnut starch with low damaged in a high yield. From the physico-chemical results obtained and, additionally, the soft conditions used throughout the procedure like temperature and pH, no significant changes in starch physico-chemical properties are expected. The ability to have a physio-chemical characterization of starch present in fresh chestnut fruits is the first step for relating its physico-chemical properties with chestnut quality like its texture after cooking. Also the high amounts of starch present in chestnut fruits and their interesting functional properties makes chestnut a possible alternative

source of starch. Native chestnut starch presented a swelling power and pasting profile similar to corn starch, but with a lower pasting gelatinization and a peak viscosity temperatures, making native chestnut starch a potential technological alternative to corn starch, especially in application needing lower processing temperatures. Although only used at laboratory scale, the low price of the reagents employed, the simple procedures used for isolation, with proper adaptation, could be easily applied at industrial scale.

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