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Effects of drying temperatures on physico-chemical properties of dried and rehydrated chestnuts *(Castanea sativa)*

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Abstract

This work evaluated the effects of two drying temperatures (40 and 60 °C) on some physical and chemical properties of chestnuts, both dried and after their rehydration using steam at 100 °C. The morphological characteristics of fresh starch granules were characterized by a round or oval shape, with diameter length ranging from 3.0 to 15.0 μm. After drying and rehydration, the granules appeared more shapeless and the surfaces were quite rough. The changes also caused more of the open pore volume fraction in samples dried at 60 °C, than in those dried at 40 °C and, much more, than the fresh samples. Also, calorimetric behaviour, starch and simple sugar changes after chestnut drying and rehydration, were studied.

Keywords: Chestnut; Drying; Porosity; Rehydration

1. Introduction

Chestnuts are characterized by a limited shelf-life because of their high water activity and sugar content. The drying process is the oldest method for the conservation of these fruits (Breisch, 1996), but studies in the literature focus mainly on raw materials (varieties) and only a few assess the impact of different heat treatments on chestnut composition. For instance, the effects of boiling, steaming (107 °C) and roasting in a 200 °C oven (Shin, Oh, & Kim, 1981) and roasting at 220 °C (Künsch, Schärer, Patrian, & Höhn, 2001) were studied. During the drying process, foods undergo volume changes, either by shrinkage due to moisture loss, or by expansion due to gas generation or pore formation. The open pore volume fraction plays an important role in determining the structural properties of a product as well as its rehydration characteristics (Donsì, Ferrari, & Nigro, 1996). Rehydration usually follows the drying process and aims at restoring the raw material's properties when it comes into contact with water. Classic rehydration by dipping is difficult, because of the long time it requires and the un-homogeneous distribution of the absorbed water, so the use of steam can improve the process (Lewicki Piotr, 1998). The evaluation of the objective quality level of rehydrated products is still a matter of interest because of the physical and chemical parameters involved, such as: drying kinetics, open pore volume fraction (Karathanos, Kanellopoulos, & Belessiotis, 1996) and starch behaviour. Starch granules can be physically altered during the drying process, with the result that the morphological granular characteristics and the sensory properties of the chestnuts are modified. Starch and modified starch have been studied by many authors in a variety of vegetables, such as waxy maize, wheat, rice and potato (Paredes-Lopéz, Bello-Pérez, & Lòpez, 1994; Singh, Singh, Kaur, Singh Sodhi, & Singh Gill, 2003), but specific studies providing information on starch modification after drying and rehydration of chestnuts are scarce (Pizzoferrato, Rotilio, & Paci, 1999). The aim of this work is to evaluate the effects of two drying temperatures (40 and 60 °C) on some physical and chemical properties of dried

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chestnuts, and again after their rehydration using steam at 100 °C. The chestnut drying process was studied by monitoring morphological starch changes by scanning electron microscopy, chestnut porosity and calorimetric behaviour by differential scanning calorimetry (DSC), and also starch and simple sugar changes. The same analyses were carried out on rehydrated samples, with the exclusion of DSC and porosity.

2. Materials and methods

2.1. Drying and rehydration experiments

Samples of "*Marrone di Roccadaspide* " chestnuts (*Castanea sativa*), were obtained from local farms in the Campania region, Italy. Drying experiments were carried out on fruits with epicarp and weight ranging from 11.5 to 12.5 g, at 40 and 60 °C in a convection oven, with an air speed of 0.5 m/s, so as to reduce the average moisture of chestnuts to about 20% w/w. The dried products were then rehydrated by steam at 100 °C for about 8 h. During the drying process, the variations in moisture content were monitored. All drying and rehydration tests were carried out on 10 chestnuts and replicated three times. The fresh, dried and rehydrated chestnuts were evaluated by means of scanning electron microscopy (SEM), DSC and porosimeter, and by monitoring starch, amylose, amylopectin and simple sugar contents.

2.2. Total starch determination

The quantitative analyses of total starch were assessed by an enzymatic procedure (AA/AMG 9/97, Megazyme, Ireland), according to the AOAC method, (1997). In compliance with McCleary, Solah, and Gibson (1994), 100 mg of milled product were washed with aqueous ethanol and pre-treated with dimethyl-sulphoxide at 100 °C to disperse starch before analyses. Then the samples were incubated with thermostable α-amylase and amyloglucosidase to hydrolyse the starch (50 °C, 30 min, sodium acetate buffer, pH 4.5) (McCleary, Gibson, & Mugford, 1997). Glucose was determined by spectrophotometric measurement at 510 nm (Spectrophotometer UV–VIS, Perkin Elmer, mod. Lambda Bio 40, USA).

2.3. Determination of the amylose and amylopectin

Quantitative analysis of the amylose and amylopectin fractions was carried out by enzymatic analysis (AM/AMP 01/96, Megazyme, Ireland). The concentration of amylose in the starch sample was estimated by spectrophotometric absorbance at 510 nm (Yun & Matheson, 1990).

2.4. Determination of sugar

Simple sugars were determined by HPLC (Hewlett Packard, mod. 79852, USA). Extraction and analysis of sugars were carried out according to the AOAC method (1989). The HPLC system was equipped with 4.6×250 mm (60 Å, 4 μm) Carbohydrate-Cartridge column (Waters, USA). The mobile phase was an acetonitrile–water solution (75:25), with a flow rate of 1.4 ml/min and a column temperature of 60 °C. Peaks were detected by a refractive index detector (Hewlett Packard, mod. 100, USA), and concentrations were calculated with external standards.

2.5. Measurement of pore size and pore size distribution of samples

The pore size of samples and their distributions were measured using a mercury porosimeter (Thermofinnigan, Pascal, mod. 140 and mod. 240, USA), trough and the following equation:

$$Pr = 2\gamma \cos \delta,$$

where the pore size was related to the applied pressure in the mercury porosimeter through the Washburn equation (Adamson, 1990); P is the pressure applied on the mercury in order to enter into the sample pores of radius r or larger; γ is the surface tension of mercury, taken as $\gamma = 0.18$ N/m, and δ is the contact angle of mercury on food, taken as $\delta = 140°$. The samples analyzed were dried until to reach the equilibrium moisture content, equal to about 8.3% for chestnuts dried at 40 °C and 6.4% for chestnuts dried to 60 °C.

2.6. DSC measurements

Differential scanning calorimetry measurements were performed on a Mettler Toledo calorimeter (mod. TC 15 TA Controller, Switzerland). The samples (20 mg) were weighed directly in DSC aluminium pans. The samples were scanned at a rate of 10 °C/min from 10 to 150 °C.

2.7. Scanning electron microscope

Metalization was achieved by covering the sample with a thin layer of gold and gold/palladium. The process was carried out by an AGAR Auto Sputter Coater mod.108 A (England), for 150 s. The operation was carried out in argon- and vacuum-sealed atmosphere. The scanning electron microscope, LEO 420, mod. 2.04, was supplied by ASSING (Italy).

2.8. Chemicals

All reagents, from Sigma-Aldrich (Sydney, Australia), were of analytical or HPLC grade as required.

2.9. Statistical analysis

All values reported were calculated on a dry weight (d.w.) basis and represent the average value of the analysis of 3 different samples. The data from the chemical analyses were subjected to a variance analysis, and the significance of differences between means was determined with Student–Newman–Keuls's multi-range test.

3. Results and discussion

3.1. Drying and rehydration of chestnuts

The experimental data regarding chestnut moisture variations during drying at 40 and 60 °C showed that the time needed to reach about a 20% moisture content was about 51 h for chestnuts dried at 40 °C and 37 h for chestnuts dried at 60 °C (Fig. 1). Afterwards, the chestnuts were rehydrated using steam at 100 °C, to at least a 30% moisture content, that was reached after 8 h for samples dried at 40 °C. The samples dried at 60 °C (at the same rehydration time) showed moisture values of 35.3%, which were significantly higher than values obtained for chestnuts dried at 40 °C. The mathematical model for the dehydration of chestnuts, as well as for their rehydration, constitutes the object of another research paper under study.

3.2. Starch, amylose and amylopectin contents

The drying temperatures had little effect on chestnut starch content, equal, in fresh samples, to about 58.3% (d.w.), since it did not vary with the different temperatures used. Instead, the starch content significantly decreased after rehydration, with no differences between the tested temperatures (Table 1). This behaviour can be explained, not only by starch thermal modification, but also by endogenous amylase activities, as consequence of dry-heat treatments followed by steam rehydration. The thermal treatments caused the formation of modified starch, which is not detectable by the enzymatic test (Voragen, 1998); in the chestnut, the enzymatic activities

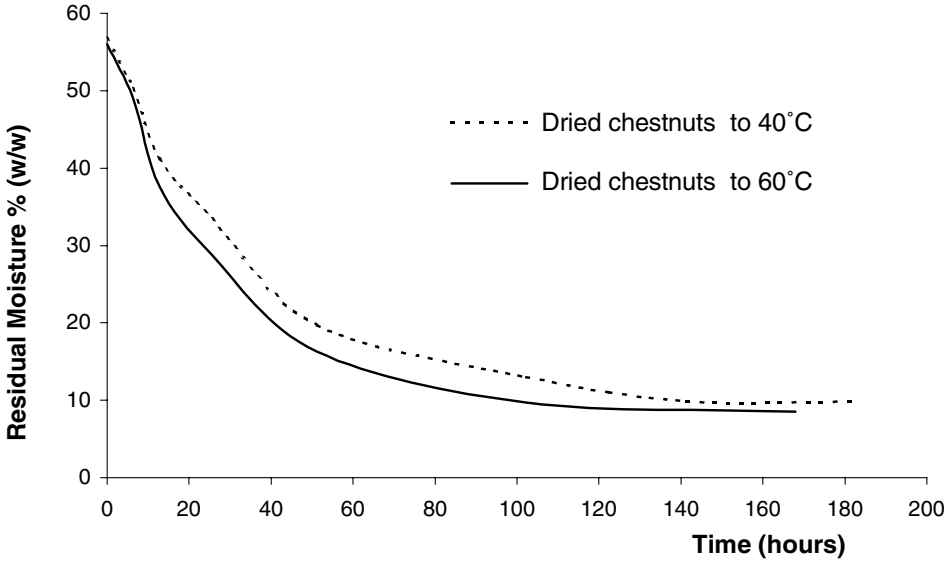

Fig. 1. Moisture content of chestnuts dried at different temperatures.

Table 1
Percentages of total starch, amylose and amylopectin of chestnuts

	Starch % (dry wt. basis)	Amylose (% of starch) (dry wt. basis)	Amylopectin (% of starch) (dry wt. basis)
Fresh chestnuts	58.3 ± 1.8 a	32.9 ± 2.2 a	67.2 ± 2.4 a
Dried chestnuts at 40 °C	56.2 ± 2.4 ac	43.3 ± 4.0 b	56.7 ± 2.6 b
Dried chestnuts at 60 °C	53.9 ± 2.3 ac	57.8 ± 1.2 c	42.2 ± 2.0 c
Rehydrated chestnuts after drying at 40 °C	50.7 ± 2.6 bc	53.1 ± 1.0 d	47 ± 2.0 d
Rehydrated chestnuts after drying at 60 °C	51.5 ± 2.2 bc	62.7 ± 0.9 e	37.3 ± 2.0 e

Results are the means of three determinations ±standard deviation.
Means with different letters are significantly different ($p \leq 0.05$).

were mainly attributed to β-amylase (Nomura, Ogasawara, Uemukai, & Yoshida, 1995). However, even though the role of β-amylase in the degradation of starch was not clarified at all, β-amylase does not hydrolyse the 1–4 glycogen bonds that are located 2 or 3 units from the branch point of amylopectin. Residue molecules, known as "limits dextrins", are formed. Their amounts, with the formation of malto-oligosaccharides molecules, could explain the starch decrease after steam rehydration; thus simple sugars did not increase at all (Table 2). The percentages of amylose and amylopectin in fresh chestnuts were about 32.8% and 67.2% of starch (d.w.), respectively, and these play an important role in starch functionality. The amylopectin decreased to values of about 56.7%, when dried at 40 °C, and 42.2% when dried at 60 °C; after rehydration the amylopectin content further decreased to 46.9% in samples dried at 40 °C, and to 37.3% in samples dried at 60 °C. The magnitudes of these notable changes seem to be partially affected by the chosen method for our analyses; thus they cannot distinguish dextrins content from amylose. Consequently, in processed chestnuts, the amylose content could be an overestimate.

3.3. Sugar content

Sucrose was the most representative sugar in fresh chestnuts (Künsch et al., 2001; Senter, Payne, Miller, & Anagnostakis, 1994), with values of about 29.7% (d.w.) in fresh product, and only minor amount of fructose (1.9%), maltose (1.5%), glucose (1.4%) and xylose (0.4%), were detected (Table 2). The sucrose content decreased significantly, in both dried samples, to a greater extent after drying at 60 °C, owing to thermal degradation; while the maltose content remained more stable after drying. After chestnut rehydration the sucrose concentration, responsible for the sweet taste, remained at high values (>22%), and was closely related in both samples, as were fructose and glucose content.

3.4. Porosity

Porosity refers to the volume fraction of void space, that can be actual space filled with air or merely space. For hygroscopic materials, porosity changes with moisture content. The water in the fruits may be visualized as being within a solid matrix that contains closed

Table 2
Values of simple sugars in chestnuts (g/100 g dry wt. basis)

	Xylose	Fructose	Glucose	Sucrose	Maltose
Fresh chestnuts	0.4 ± 0.02 a	1.9 ± 0.2 a	1.4 ± 0.3 a	29.7 ± 1.2 a	1.5 ± 0.1 a
Chestnuts dried at 40 °C	–	1.0 ± 0.1 b	1.1 ± 0.2 a	27.9 ± 1.0 b	1.8 ± 0.2 a
Chestnuts dried at 60 °C	–	1.9 ± 0.3 a	1.4 ± 0.2 a	22.3 ± 1.1 c	1.0 ± 0.2 a
Rehydrated chestnuts after drying at 40 °C	0.1 ± 0.01 b	2.1 ± 0.2 a	1.3 ± 0.1 a	22.9 ± 0.7 d	0.6 ± 0.1 b
Rehydrated chestnuts after drying at 60 °C	0.1 ± 0.01 b	2.3 ± 0.1 a	2.3 ± 0.1 a	22.8 ± 0.8 d	0.8 ± 0.1 b

Results are the means of three determinations ±standard deviation.
Means with different letters are significantly different ($p \leq 0.05$).

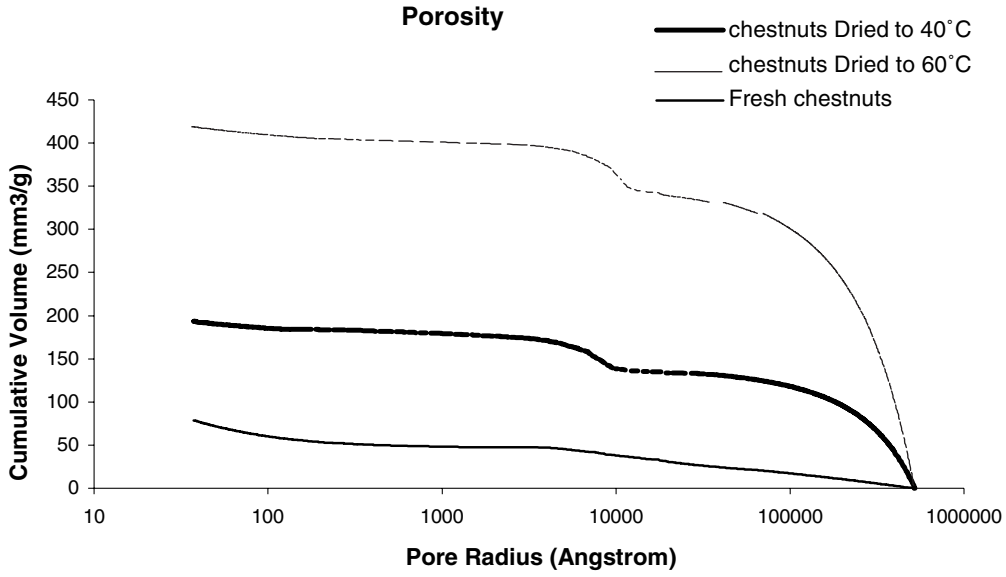

Fig. 2. Cumulative volume of pores of fresh, dried to 40 °C and dried to 60 °C chestnuts as a function of the pore size, as determined by the mercury porosimetry.

or interconnected entities of liquid water. During the drying process these entities are reduced in size due to water evaporation (Karathanos et al., 1996). The actual size of these entities cannot be measured directly, but a highly porous network would be formed. The specific volume of the porous structure, which is accessed by mercury as a function of pore size of fresh and dried chestnuts, is given in Fig. 2, from which the total volume of pore, contained in unit mass of sample, can be found by addition of volumes of pores with sizes from the largest down to that of the specific pore size. Chestnuts dried at 60 °C gave a total open volume of 415.3 mm^3/g, the majority of pores were larger than 10 μm; chestnuts dried at 40 °C gave a total open volume of 193.4 mm^3/g while, for fresh chestnuts, the total open volume was 78.9 mm^3/g and most pores were smaller than 10 μm. These data confirmed that the porosity of hygroscopic materials normally increases as moisture content decreases (Marousis & Saravacos, 1990); however, available data on porosity changes with heating of hygroscopic materials are very limited.

3.5. Gelatinization parameters

Thermograms of fresh chestnut displayed a typical endothermic peak (Fig. 3), owing to the starch transformation. The gelatinisation temperature ($T_p = 82.5$ °C) was higher than that of other starches, as occurs for

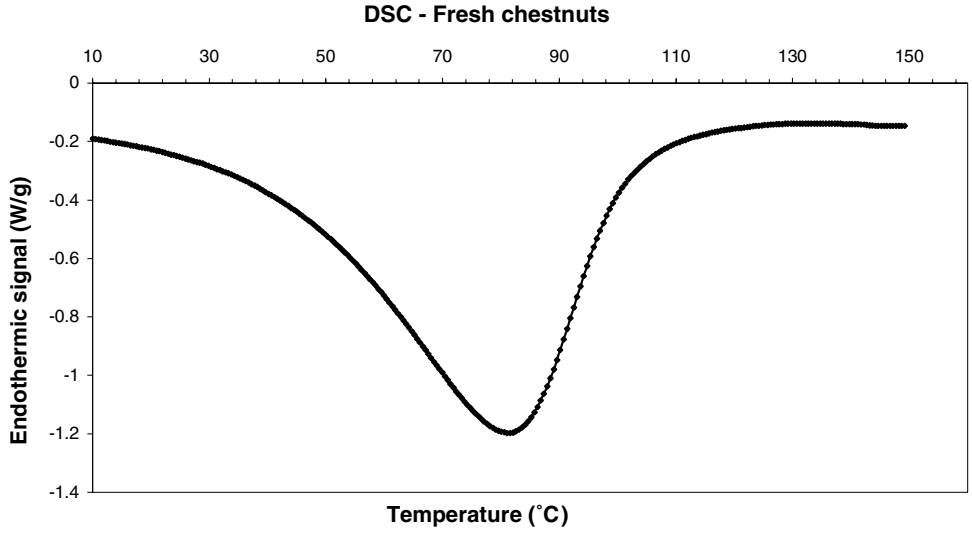

Fig. 3. DSC thermogram of native chestnut starch.

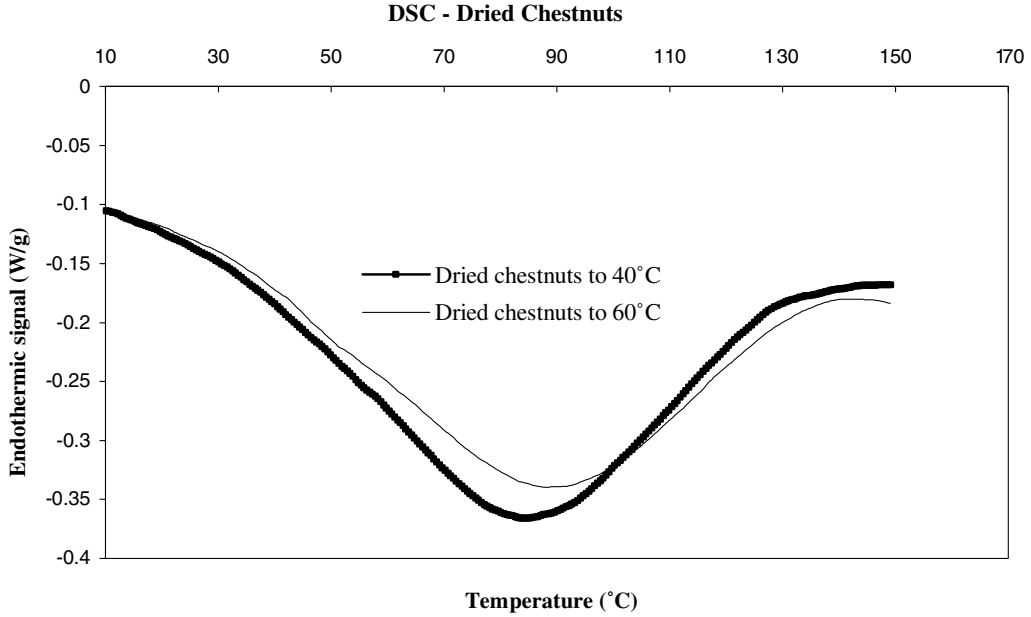

Fig. 4. DSC thermograms of dried chestnut starch.

the most resistant starch (Zhang & Oates, 1999). This phenomenon is tied to the sample content of amylopectin, as a low gelatinisation temperature is typical of starches with less amylopectin (Zhang & Oates, 1999). Thus, in fresh chestnut, the high amount of amylopectin, as shown in Table 1, explains the gelatinisation temperature. Moreover, in fresh samples the onset temperature (T_o) was about 10.5 °C, the endset temperature (T_e) was about 130 °C, and heat of transition (ΔH) was −502.5 J/g. After drying, the (T_o) did not change in either sample, whilst the (T_e) increased to about 140 °C, without differences between the dried samples. The ΔH values were: −222.2 J/g in samples dried at 40 °C and −175.5 J/g in samples dried at 60 °C. The wide non-symmetric shape of thermograms was due to the starch transformation occurring during the drying processes, whilst the reduction of enthalpy depended on the decrease of water content caused by drying (Fig. 4).

3.6. Morphological granular characteristics of starch

The fresh chestnut starch granules appeared to be round or oval; the surfaces were smooth when viewed under SEM, with characteristic diameter dimensions in the range 3.0–15.0 μm (Fig. 5). The SEM, which gives interesting information about the three-dimensional structure of products, is particularly suitable for observing the morphological changes caused by the manufacturing processes. Samples dried at 40 °C exhibited fractures in starch granules (Fig. 6), and this behaviour was more visible in samples dried at 60 °C (Fig. 7). Anyway, in all dried samples, the granules appeared

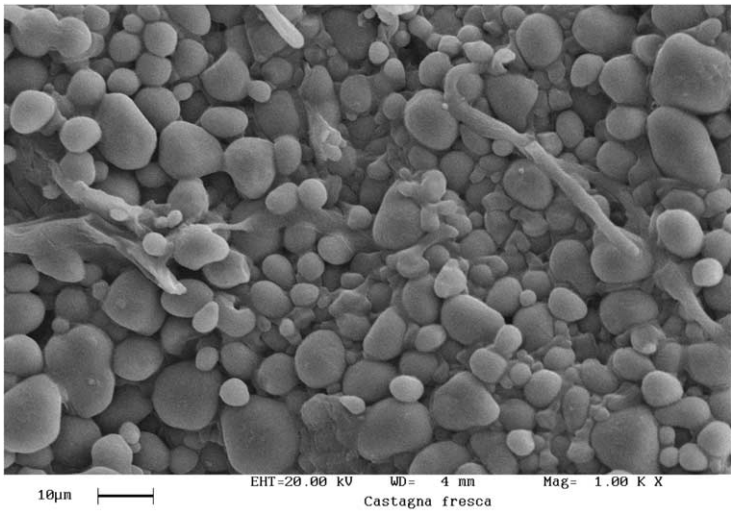

Fig. 5. SEM photomicrograph of fresh chestnut starch granules.

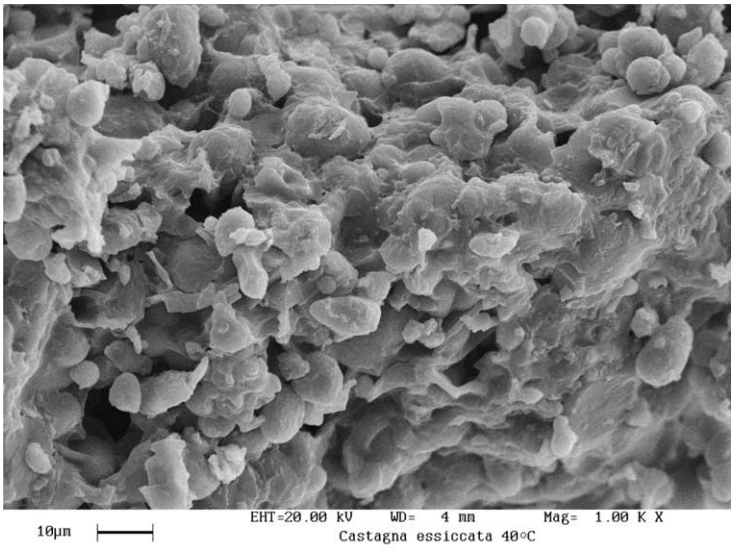

Fig. 6. SEM photomicrograph of 40 °C-dried chestnut starch granules.

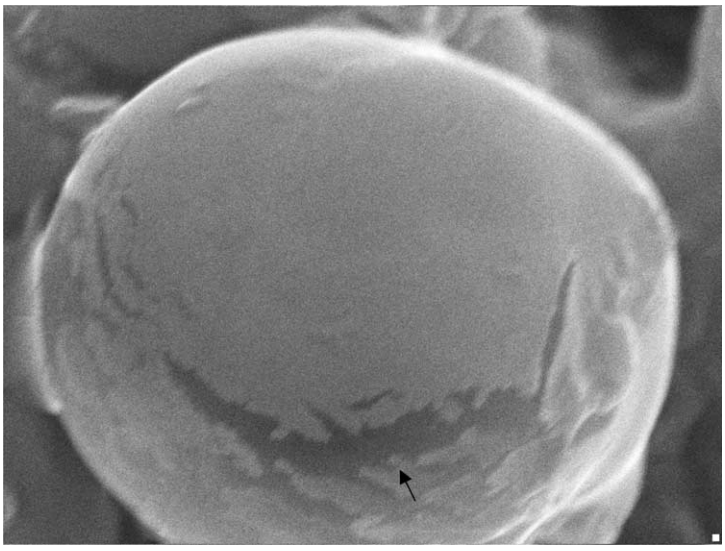

Fig. 7. SEM photomicrograph of dried chestnut starch granules at a greater enlargement.

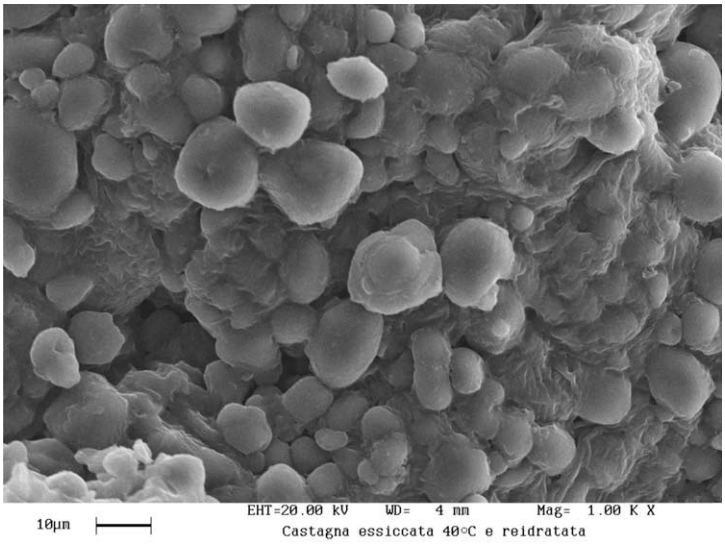

Fig. 8. SEM photomicrograph of rehydrated chestnut starch granules.

more shapeless and the surfaces were quite rough; thus, the SEM pictures clearly showed a surface bursting, responsible for increased free volume. The rehydrated samples, obtained using steam, showed a disruption of molecular order within starch granules, involving the chain rearrangement (Fig. 8).

4. Conclusions

This research has shown that different chestnut drying temperatures produced changes in the physical structure of the nuts, as well as modification of starch granules, whose original diameter dimensions ranged from 3.0 to 15.0 μm. The morphological characteristics of fresh starch granules, originally ovoidal or spherical shaped, after drying and rehydration appeared more shapeless. The SEM showed a surface bursting, quite rough with some fractures, more visible in samples dried at 60 °C. These changes in physical structure after drying can be associated with the increase in porosity and the DSC behaviour. The open pore volume increased in 40 °C-dried samples with respect to the fresh ones, and this was more accentuated at 60 °C. The thermograms of dried samples showed wider and non-symmetric shape, due to starch modification occurring during the processes.

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