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Innovative technologies optimizing the production process of “*Castagne del Prete*”: Impact on microstructure and volatile compounds

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ABSTRACT

“*Castagne del Prete*” are traditional processed chestnuts from the Campania Region obtained through a treatment involving a drying, roasting and rehydration phase. The last one is a long, costly and non-standardized treatment that is therefore in need of improvement. In the present study, different technologies (heat treatment, steam and thermosonication) were evaluated to optimize the chestnuts rehydration phase. To this end, microstructure, thermal properties and volatile organic compounds (VOCs) of “*Castagne del Prete*” obtained by different rehydration methods were evaluated.

Results highlighted that thermosonication strongly influenced the starch gelatinization process, responsible for the total destructuration and denaturation of the crystalline network.

VOCs analysis showed that the samples rehydrated by thermosonication with chestnuts/water ratio 1:5 had a volatile compounds pattern very similar to the control sample obtained by classic rehydration method. In addition, the time for obtaining “*Castagne del Prete*” by using thermosonication (approx. 5 h) as rehydration treatment was significantly reduced in comparison to the classic method which took approximately 7 days. Thus, thermosonication emerged as the most promising technique among those investigated for the production of “*Castagne del Prete*”, as it saves energy and time, while guaranteeing the flavor and structural characteristics of the finished products.

1. Introduction

Chestnuts are seasonal fruits with a limited shelf-life due to their high water activity, high metabolic activity and the porous structure of the epicarp (Correia et al., 2009). So, efficient preservation techniques, such as drying, are required to promote their consumption over long periods. Among the traditional food products of the Campania Region (Italy) are “*Castagne del Prete*”, local chestnuts (*Castanea Sativa* L.) that undergo a very long treatment involving a drying phase (about 15 days), followed by a roasting phase and a rehydration phase (about 7 days).

The drying process is the oldest method of preserving these fruits (Breisch, 1996). During the drying process, the product undergoes changes in volume, either through shrinkage due to moisture loss or expansion due to gas generation or pore formation. The size of the chestnuts pores plays an important role in determining the structural properties of the product as well as its rehydration characteristics (Donsi

et al., 1996).

The dried and roasted product has a moisture content of about 10%. The thermal processes of drying and roasting determine a change in the product structure caused by the Maillard reaction directly influenced by the procedure and the process parameters, time and temperature. As reported by Lund and Ray (2017), in food products subjected to high-temperature treatments, such as roasting, the formation of a series of compounds with very different chemical composition, structure and properties is observed, mainly due to the Maillard reaction.

Rehydration usually follows the drying and roasting process and it aims to restore the properties of raw materials when exposed to water. Classic rehydration by dipping is difficult, due to the long time it requires and the uneven distribution of the absorbed water, so the use of steam can improve the process (Lewicki, 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

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drying kinetics, the volume fraction of open pores (Karathanos et al., 1996) and the structural change of starch. Indeed, the drying process can alter physico-chemical organization of the starch granules, leading to a change in the morphological characteristics and, consequently, the sensory properties of the chestnuts. 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 et al., 1994; Santagata et al., 2021; Singh et al., 2003; Turco et al., 2019), but specific studies providing information on starch modification after drying and rehydration of chestnuts are scarce (Pizzoferrato et al., 1999).

Over the years, in contrast to the extensive investigations on the properties of chestnut products, few or no technological innovations have been reported on the processing of chestnuts. Since the classical rehydration step of chestnuts is rather long and uncontrolled, due to the irregular distribution of water absorbed by the product (Attanasio et al., 2004; Lewicki, 1998), in this paper the authors focused the attention on likely faster rehydration processes.

Thus, novel hydration tests were carried out using both ultrasound technology and exposure to moist heat in autoclave. Ultrasound is an emerging sustainable technology that enhances the rate and the efficiency of several processes in the food industry. It can also be applied in combination with temperature (thermosonication) and pressure (manosonication) to produce a synergistic effect, which further enhances its efficacy (Condón-Abanto et al., 2016). Moreover, in the food industry, ultrasound technology has been the object of research and development being considered a “novel processing technology” (Chemat et al., 2011; Patist & Bates, 2008; Pingret et al., 2013; Soria & Villamiel, 2010). Ultrasound technology is known to have a positive effect bringing important advantages in terms of productivity, yield, increased quality, reduced chemical and physical hazards and safety for the environment.

However, ultrasound has been used as a pretreatment for drying, dehydration as well as rehydration operations of different dried food such as cabbage, minced meat, kiwi-fruit, shiitake mushrooms, carrot, apple (Bhargava et al., 2021). Different authors highlighted that the application of ultrasound results in a reduction in time of rehydration of dried vegetables such as white cabbage (Tao et al., 2019), green pepper (Szadzińska et al., 2017), bean (Miano et al., 2016) and strawberry (Amami et al., 2017).

For this reason, the present work aims to assess the effect of different technologies (heat treatment, steam and thermosonication) to wet dried chestnuts in order to reduce hydration time, to optimize production and standardize the end product. Particular attention was paid to the effects of the proposed processes on the physical (water content and weight variation by thermogravimetric analysis), chemical (changes in starch microstructure by Scanning Electron Microscopy, SEM) and volatile (volatile organic profile by Headspace-Solid Phase Micro Extraction-Gas Chromatography/Mass Spectrometry, HS-SPME-GC/MS) properties of the treated chestnuts.

2. Materials and methods

2.1. Sample preparation

Dried and roasted chestnuts (*Castanea sativa* Mill., cultivar “Palummina”) were provided by the Agricultural Cooperative Society of Montella (Montella (AV), Campania Region, Italy). In detail, fresh chestnuts of the same batch of harvesting were previously dried at room temperature for 16 days and roasted in ventilated rotating ovens at 140 °C for about 1 h.

The dried and roasted chestnuts were hydrated by 3 different methods in the laboratories of the Institute of Food Sciences of National Research Council (Avellino) as described below:

- Dynamic method: chestnuts were placed in a beaker containing water with two different chestnut/water ratios (1:5 and 1:10) (w/v). The beakers were placed inside an ultrasonic bath (I.S.Co. srl, Milan)

for 5 h at 55 °C, 40 KHz and 70 Watt. The chestnuts were spread on a single layer that allowed uniform transmission of the ultrasonic waves to the samples. Sample CD1 and CD2 were obtained.

- Steamed method: chestnuts were treated into autoclave (STER-ISTEAM2 Model, NEWMED, Reggio Emilia) with steam at 105 °C for 40 min (Sample CS).
- Static method: chestnuts were placed in a beaker containing water with two different chestnut/water ratios (1:5 and 1:10) (w/v). The beaker was placed in a pot for 5 h at 55 °C. (Sample CST1 and CST2).

Each treatment was carried out twice, and at the end of each treatment, the chestnuts were left to dry overnight at room temperature and stored at 4 °C.

A sample of “*Castagne del Prete*” obtained by the classic rehydration method and provided by Agricultural Cooperative Society of Montella was used as a control (C). Rehydration consisted of moisturizing the chestnuts with 1-h jets of water twice a day for 7 days.

The analysis carried out on the hydrated chestnut samples were: moisture determination, water activity, Scanning Electron Micrography (SEM), thermogravimetric analysis (TGA), and volatile compounds profile by HS-SPME-GC/MS.

2.2. Determination of moisture and water activity

The moisture content of the samples was determined by the gravimetric method using a drying oven at 101 ± 2 °C to constant weight according to Yang et al. (2015).

The water activity (a_w) of all the samples was determined at 20 °C by using the Rotronic HP23 HygroPalm model, following the manufacturer’s instructions. For the determination, about 5g of ground chestnuts were spread on the bottom of the circular dishes to expedite the attainment of vapour equilibrium. All analyses were carried out in triplicate.

2.3. Scanning Electron Microscopy (SEM)

Morphological analysis of chestnuts was performed by means of a Scanning Electron Microscope (SEM) (Quanta 200 FEG, 338 FEI, Eindhoven, The Netherlands), on cryogenically fractured cross sections. SEM observations were performed at room temperature, internal water vapour pressure of 66.66 Pa, high vacuum mode, an acceleration voltage of 20 kV and using a large field detector (LFD). Cross-sections samples were fixed onto aluminium stubs using carbon adhesive. Prior to the observation, the surfaces were coated with a homogeneous layer (18 ± 0.2 nm) of Au and Pd alloy by means of a sputtering device (MED 020, Bal-Tec AG, Tucson, AZ, USA).

2.4. Thermogravimetric analysis (TGA)

Thermogravimetric analyses were carried out with a Mettler Thermogravimetric Analyzer Mod. TG 50. Measurements were performed on samples of about 8–10 mg, placed in open ceramic crucibles and heated from room temperature to 600 °C at 20 °C/min in nitrogen atmosphere, with a nominal gas flow rate of 30 mL/min. Prior to testing, a blank curve was measured and subtracted from the single thermograms, to correct for instrumental drift (Vyazovkin et al., 2014). TGA analysis were performed in duplicate and the results were expressed as the averaged values obtained by the two measurements.

2.5. Headspace-Solid Phase Micro Extraction-Gas Chromatography/Mass Spectrometry (HS-SPME-GC/MS)

The volatile fraction of samples was analyzed by headspace sampling, using the solid-phase microextraction technique (HS-SPME) according to Reale et al. (2019) with some modifications. Briefly, about 10 g of chestnuts of each treatment were hulled and grounded to a course

granulate (about 2 mm). Then, 1 g of samples was placed into a 20 mL headspace vial and 5 μ L of 3-octanol (internal standard, 100 mg/L standard solution) was added. The vial was placed in a thermostatic block (40 °C) on a stirrer and, after equilibration, the headspace of the samples was sampled using an SPME fiber coated with DVB/CAR/PDMS (divinylbenzene/carboxen/polydimethylsiloxane, thickness 50/30 μ m) at 40 °C for 50 min. The HS-SPME was automatically performed with a multi-purpose sampler (Gerstel MPS2).

For VOCs evaluation, an Agilent Technologies (Agilent Technologies, USA) 7890A gas-chromatograph coupled to an Agilent Technologies 5975 mass spectrometer equipped with a 30 m \times 0.25 mm ID, film thickness 0.25 μ m capillary column (HP INNOWAX, Agilent Technologies, USA) was used. The gaseous carrier was helium (flow 1.5 mL/min) and SPME injections were splitless (straight glass line, 0.75 mm I.D.) at 240 °C for 10 min during which time thermal desorption of analytes from the fibre occurred. The oven parameters were as follows: initial temperature of 50 °C held for 5 min, followed by an increase to 200 °C at a rate of 5 °C/min, then held for 18 min. Injector temperature was 240 °C. Mass spectrometer operated in scan mode over a mass range of 40–500 amu (2 s/scan) at an ionization potential of 70 eV. VOCs were firstly identified based on their mass spectra using Wiley7, Nist 05 libraries. For the next identification step, a retention index (RI) was calculated for each compound according to Van den Dool and Kratz (1963) and based on a series of alkanes. The calculated RIs were then compared with van Den Dool and Kratz RIs for high polar stationary phases using the NIST online database (<http://webbook.nist.gov/chemistry/>). The data are expressed as RAP = Relative Peak Area (area Peak Compound/Area peak Internal Standard)*100. Blank experiments were conducted in two different modalities: blank of the fiber and blank of the empty vial. These types of control were carried out every 4 analyses. All analyses were performed in duplicate.

2.6. Statistical analysis

The means and standard deviations of experimental results were calculated from two replicate trials. To determine differences between volatile compounds, one-way ANOVA and Tukey's test were used. Principal component analysis (PCA) was used to find the correlation between volatile compounds and samples treated in a different way and was carried out using Tanagra 1.4 software.

3. Results and discussion

3.1. Moisture and water activity

Table 1 shows the values of moisture content and water activity of dried and roasted chestnuts rehydrated by different hydration methods. The control samples of "Castagne del Prete", obtained by the classical method, had moisture and a_w values of 30.20% and 0.850, respectively.

The samples of chestnuts rehydrated by the different processes had moisture values ranging from 15.14% (CS) to 31.37% (CST1). From the data showed in Table 1, it can be seen that the samples rehydrated by static method, for both the 1:5 and 1:10 chestnut/water ratios, showed

higher hydration than the CD1, CD2 and CS samples.

As also reported by Górnicki et al. (2020), water absorption in chestnuts depends on soaking time and temperature. During hydration, the absorbed water migrates from the outside to the core of the product due to the presence of pores in the non-lignified epicarpal structures of the chestnuts, thus providing both moisture and softness (Sacchetti et al., 2005). Rehydration is also greatly influenced by the drying process, which modifies the morphological structure of samples by increasing their porosity (Moreira et al., 2008; Senadeera et al., 2003).

The a_w values (Table 1) were in the range of 0.860 ± 0.009 (CST1) and 0.811 ± 0.002 (CD2) with the exception of the CS sample which showed a lower a_w of 0.602 ± 0.014 . As highlighted by López-Malo and Alzamora (2015), at a_w values < 0.90–0.92, bacteria fail to grow. Yeasts and moulds, on the other hand, are more tolerant to low a_w values, but usually do not grow in foods with a_w values < 0.62 (Hocking & Pitt, 1987; Scott, 1957).

These results suggest that even "Castagne del Prete" obtained by the new rehydration methods (CD1, CD2, CST1 and CST2), like control chestnuts (C) obtained by the traditional method, may be exposed to mold if they are not stored in a suitable way (e.g. packed in modified atmosphere).

3.2. Scanning Electron Microscopy (SEM)

The morphological micrographs of the fresh chestnut samples are shown in Fig. 1a (2000x), 1b (4000x). They show the presence of perfectly globular granules, whose average sizes vary between 5 μ m and 18 μ m. The intact structure of native starch refers to a regular distribution of the amylose and amylopectin fraction packed into a highly organized three-dimensional crystalline network (van Soest & Vliegenthart, 1997).

As a result of the thermal roasting process, the chestnut starch underwent a rather evident structural change, known as destructure, responsible for the loss of the initial crystallization process at the expense of amylose and the development of a new gelatinized network resulting from the interaction of starch with water, see Fig. 1c (2000x). During this process, known as gelatinization, the globular shape collapsed and gave way to a more complex morphology, in which native starch particles were still evident, but completely covered and interconnected by linear fractions of amylose chains, swollen as a result of the interaction with water (Sandhu & Singh, 2007).

Actually, it should be underlined that all the morphological analysis of the treated chestnuts were performed on the gelatinized and retrograded samples, as at room temperature starch undergoes to a new form of crystalline process, known as retrogradation process. Anyway, in this regard, the retrograded starch freezes the conformational network, thus preserving the structural information coming from gelatinized process.

Fig. 1d shows the scanning electron micrograph of "Castagne del Prete" starch obtained by the classic rehydration method (control sample C). The morphological analysis revealed the presence of swollen particles, due to the interaction of starch with hydration water. However, the presence of globular particles, together with other open and concave ones, as shown by the arrows in Fig. 1d, highlights that the roasting

Table 1
Moisture (%) and water activity (a_w) of dried and roasted chestnuts rehydrated with different hydration method.

Sample	Weight (g)	Water (mL)	Rehydration temperature (°C)	Rehydration time (hours, min)	Moisture (%)	a_w
C (control)	–	–	–	–	30.20 ± 0.16^c	0.850 ± 0.002^c
CD1	100	500	55	8 h	27.14 ± 0.14^c	0.843 ± 0.007^c
CD2	100	1000	55	8 h	25.14 ± 0.21^b	0.811 ± 0.002^b
CS	100	Steam vapour	105	40 min	15.14 ± 0.34^a	0.602 ± 0.014^a
CST1	100	500	55	5 h	31.37 ± 0.41^f	0.860 ± 0.009^c
CST2	100	1000	55	5 h	29.23 ± 0.23^d	0.852 ± 0.004^c

C = Chestnuts rehydrated by classic method; CD1 and CD2 = chestnuts rehydrated by thermosonication with two chestnuts/water ratio (1:5 and 1:10); CS = chestnuts rehydrated by steam; CST1 and CST2 = chestnuts rehydrated by static method with two chestnuts/water ratio (1:5 and 1:10).

The data are the means of three independent measurements and values in the same column followed by different lowercase letters (a to f) differ significantly ($p \leq 0.05$).

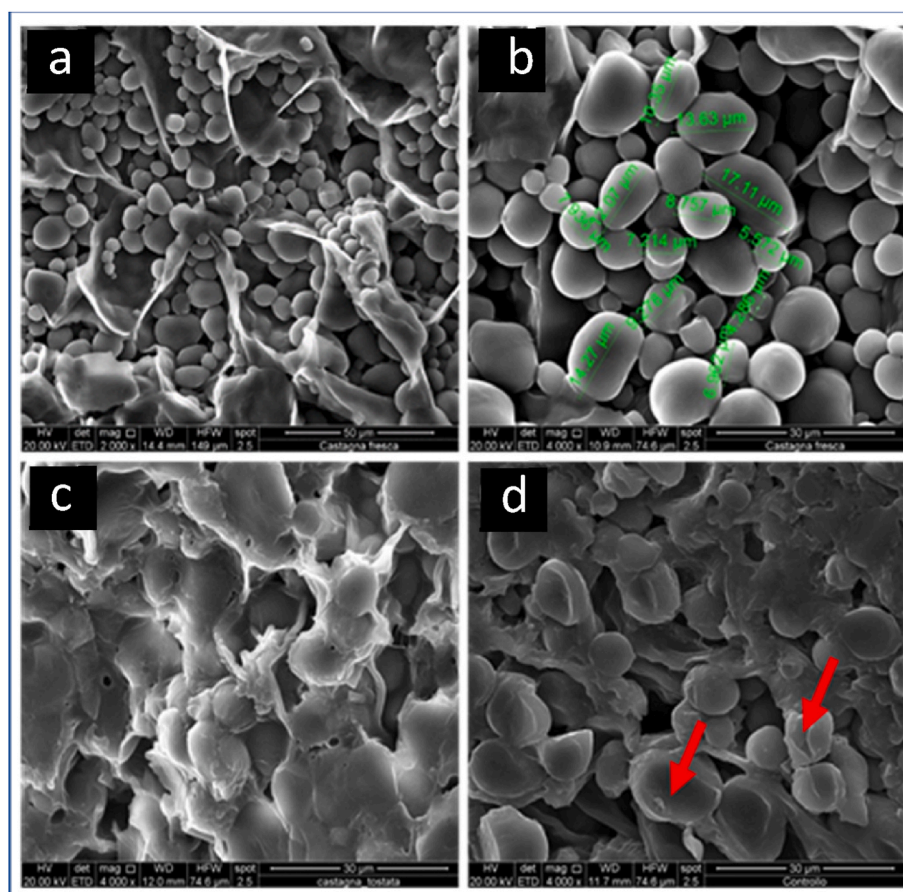


Fig. 1. Scanning Electron Microscopy images of the cross sectional starch surfaces of fresh chestnuts (a and b, 2000x and 4000x, respectively), dried and toasted chestnuts (c, 4000x) and “*Castagne del Prete*” obtained by classic rehydration method (d, 4000x).

treatment followed by rehydration caused a non-uniformly distributed destruction on the chestnut surface. Therefore, the classical rehydration treatment of chestnuts does not guarantee a homogeneous gelatinization and rehydration process; on the other hand, the presence of water within the starch structure could be responsible for the solubilisation process of the flavour and polyphenolic compounds trapped in the chestnut shell. Therefore, in this case, starch has not totally lost its original crystalline structure, partially preserving the macromolecular organization of packed chains and preventing water from interacting with the hydroxyl groups of amylose and amylopectin, which are not physically available (Vázquez et al., 2008).

Fig. 2 shows the micrographs of chestnuts rehydrated with the new methods. The morphological changing of starch surface, highlighted by the above micrographs, could be explained as follows.

The dynamic method, whose chestnuts/water ratio was 1:10 (CD2), was strongly destructuring from a morphological point of view, as can be seen from the micrographs shown in Fig. 2 a, b.

Generally, ultrasound treatment could affect the physicochemical and functional properties of starch from different botanical origins, as reported by Li et al. (2018). This method can modify the amorphous and crystalline domains of starch and thereby inducing significant modifications in thermal and functional properties of different starch-derived vegetables, such as chestnuts. Actually, the mechano-physical method used, by increasing the porosity of the starch, enhanced its surface area and, in turn, improved its chemical reactivity. In particular, the globular shape of chestnut's native starch was practically destroyed as a result of the structural collapse caused by water acting as a plasticizer, heating and ultrasound treatment, both of which induced a radical change in the polymer morphology. The linear and branched fraction of starch lost its structural coherence and crystalline pattern; the gelatinization process,

caused by the water heating during ultrasound treatment, was optimized by the physical interaction of the polar groups of amylose and amylopectin with water molecules. Nevertheless, it was possible to detect a linear, continuous and smoothed polymer surface, typical of a plasticized polymer surface (Shi & BeMiller, 2002; Sujka, 2017).

The ultrasound treatment performed on the samples with chestnuts/water ratio of 1: 5 (CD1), is shown in Fig. 2 c, d. The morphological analysis showed that the chestnuts/water ratio played a key role in the starch swelling processes. In particular, the rehydration process caused water to enter in the starch structure, resulting in the destroying of globular structure as also highlighted by other authors (Freitas et al., 2004). The physical interaction between the polar groups of water and likely amylose fraction, usually easily released from starch destructurization, favored the process of gelatinization and swelling, as evidenced by the micrographs (Fig. 2c, d). However, the lower water concentration results in the formation of structural micro-domains, in which the globular structures of the still native starch were detected, covered by gelatinous layers of unstructured starch. By comparing the micrographs of the starch structures resulting from the two dynamic methods, it is worth noting that the higher water concentration (Fig. 2a, b), although resulting in a rapid saturation of the chestnut, was able to destroy and totally modify the starch structure, whereas the lower water concentration (Fig. 2 c, d) induced a generally microporous structure, with some native starch granules perfectly covered by partially gelatinized starch. This structural difference of starch modification probably affected the moisture content of the samples, where CD1 samples had higher moisture and water activity values than CD2 samples.

Fig. 2 e, f shows the delaminated surface of steamed chestnut starch. As a consequence of steam treatment (CS), a swelling and partial destructuration of the starch was observed; the particles, well embedded

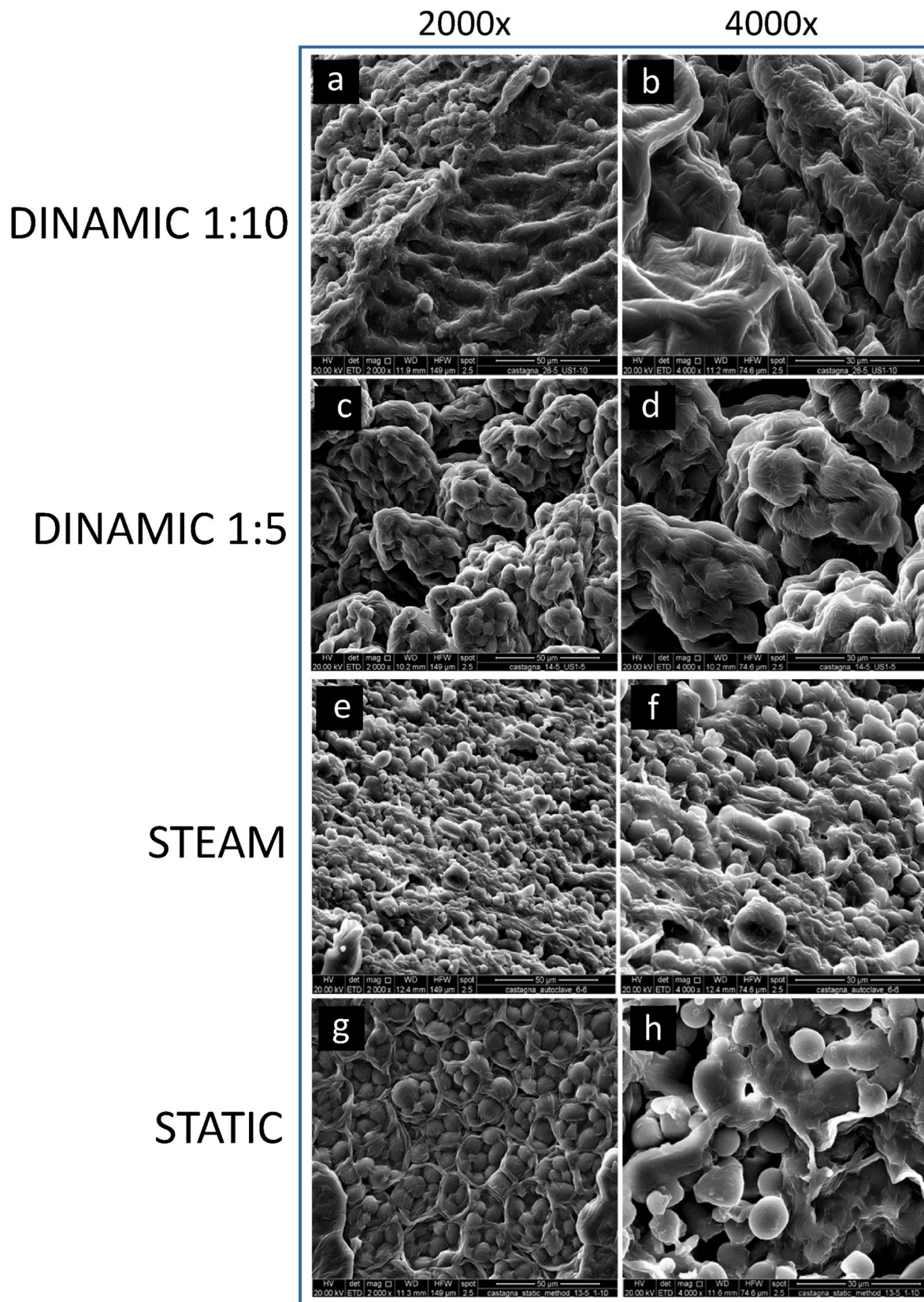


Fig. 2. Scanning Electron Micrographs 2000x (a, c, e and g) and 4000x (b, d, f and h) of the cross sectional starch surfaces of chestnuts treated by dynamic method, by steam and by static method.

in water molecules, were characterized by globular, elongated or elliptical shapes. However, after the autoclave heating process, a remarkable change in the surface characteristics was noticed. The morphological aspect was rough, irregular and partially eroded with some deep grooves between starch macromolecules and fissures on the starch granules surfaces. It was likely that the high temperature accelerated the movement of the starch molecules and created cracks and holes throughout the whole polymer structure (Gou et al., 2019).

Finally, the micrographs related to the cross-sectional surface of the starch resulting from the static method are reported in Fig. 2 g, h. For brevity, only the SEM analysis related to the 1:10 system has been reported since not significant difference was found with the static method of the 1:5 chestnuts/water ratio. The morphological analysis highlighted the presence of agglomerates of starch particles interspersed with macromolecular chains of amylose leaking from the native cell. Actually, the static method is unable to destructure the starch causing its total gelatinization, since the absence of ultrasound waves, responsible for cell rupture and water absorption. As a consequence, the starch hydration resulted in the formation of macromolecular rearrangements interspersed within polymeric clusters; in a such morphology, the water absorption, not engaged in the starch gelatinization process, could be more readily available to food spoilage bacteria (Fu et al., 2012).

From the morphological analysis performed on starch fracture surfaces of the “*Castagne del Prete*” samples, which were treated with different rehydration methods, it could be concluded that thermosonication strongly influenced the starch gelatinization process, responsible for the total destructure and denaturation of the crystalline network.

3.3. Thermogravimetric analysis (TGA)

Fig. 3 (a–b) shows the overlapped thermograms for the systems studied. In order to allow a clear understanding of the thermal profiles, only some curves have been overlapped; specifically, the thermal profiles of the following chestnut samples were reported and investigated: C, CD1, CST1, CS as well as the dried and roasted chestnuts.

In particular, the results of the weight loss profile (TGA) were reported in Fig. 3a, while the thermal degradation kinetics of the different systems were described (DTG) in Fig. 3b. From a general view of the curves, at least three main different degradation profiles were highlighted. The first was attributed to the water evolution, the second to the degradation of the cellulose residues, a natural component of chestnut fibre, and the third to the starch depolymerisation profile.

Considering the first stage of degradation, linked to water loss, it is

significant to highlight that for all samples, the temperature range involved in the phenomenon is between 25 °C and 160 °C. This wide temperature range covers the different types of physical bonds between starch and water. As reported by different authors (Santagata et al., 2018; del Rosario Salazar-Sánchez et al., 2022), it is known that there are three types of bound water, adsorbed or free water, which is easily released from the polymer matrix as it mostly covers the surfaces of macromolecular chains; adsorbed or freeze-bound water, which is associated with the polar residues of the polymer by means of hydrogen bonds; and non-freeze-bound water, which is responsible for highly temperature-resistant polymer-water crystallization complexes. Nevertheless, the samples analyzed behave differently, showing dissimilar thermal curves. This was particularly evident in the DTG curves (Fig. 3b). Being mainly polar, polysaccharides show a strong physical affinity for water; therefore, they can be easily hydrated, resulting in macromolecules with rather disordered networks. As is well known, the hydration properties of polysaccharides depend on the primary and supramolecular structure (Attanasio et al., 2004). Therefore, changes in peak area and/or temperature related to water loss are expected to reflect physical and molecular changes caused by the different strength and mode of interaction between water molecules and the starch macromolecular network. A close examination of Fig. 3a, b reveals that the different starch-water interaction, previously detailed and discussed in the morphological analysis, is reflected in a diverse water-holding capacity under thermal stress. Specifically, the roasted/dried sample, as expected, included about 6% bound water already existing within the starch structure, as no rehydration process occurred in this sample. This is coherent with a polymer network in which polymer-polymer associations are stronger than polymer-water interactions, resulting in lower water retention capacity. The samples C, CS and CST1 absorbed about 12% water, as evidenced by TGA thermograms, released from room temperature up to about 150 °C by individual degradation profiles (see DTG curves) also if CST1 sample showed a slight split in double kinetics related to the different water-binding pattern, as clarified below. Interestingly, water was slowly released from the macromolecular structure, as evidenced by the TGA thermal profile, thus confirming that it was strongly bound to starch granules, as previously detailed in the SEM analysis. Finally, the most interesting result was provided by the CD1 samples. Indeed, from the TGA curve, it was possible to detect the highest water retention ability, about 30%, characterized by a double release kinetics. Actually, the destructuring of the native starch ordered structure, due to chemical modification caused by gelatinization process following thermosonication, may significantly contribute to a

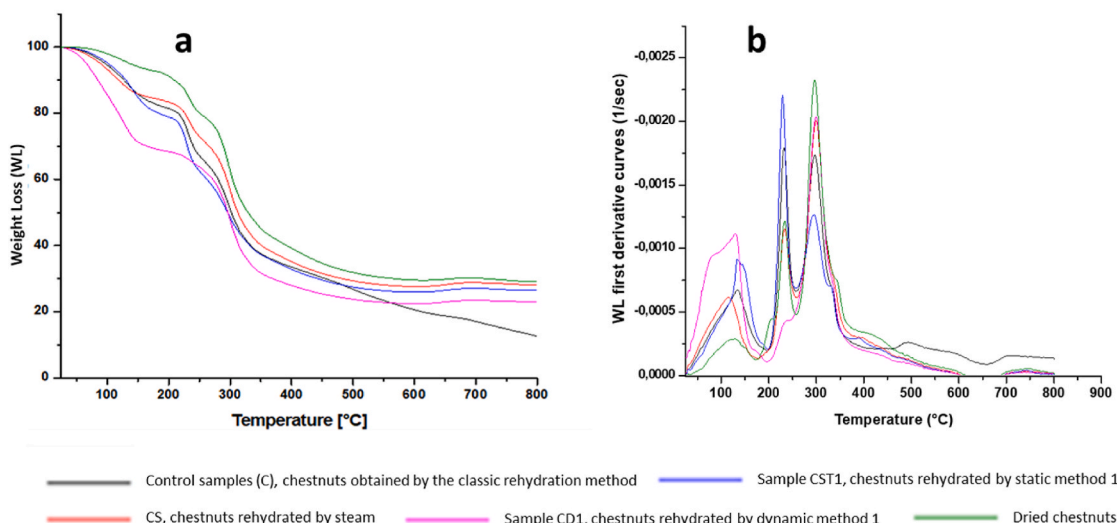


Fig. 3. Weight loss profile, TGA, (a) and thermal degradation kinetics, DTG, (b) of different samples of chestnuts.

substantial increase in both adsorbed and absorbed water content, with the maximum rate of water evolution occurring at 80 °C and 130 °C, respectively.

With regard to the depolymerisation patterns of cellulose-based fibres and starch, two well-defined thermal profiles have been clearly identified. It could be hypothesized that the first degradation peaks occurring at around 230 °C was mainly attributed to hemicellulose degradation, whereas the second main peak at around 300 °C was attributed to starch depolymerisation. Actually, although this hypothesis is widely reported in literature (Sablani et al., 2007), a careful analysis of the thermogram trends suggested that there was a cooperation of depolymerisation kinetics of both polysaccharides (hemicellulose, cellulose and starch), as the first degradation stage occurring between 200 °C and 260 °C was attributed to the hemicellulose-rich phase of the chestnuts, while the degradation phase occurring between 260 °C and 350 °C was attributed to the starch-cellulose-rich phase; the last degradation around 450 °C could be associated with some evolution of the residual lignin mass. The previous hypothesis could be supported by the slight peak of degradation of the hemicellulose occurring in CD1 sample, as evidenced by DTG thermograms. Actually, it is widely reported that mechano-physical methods, like thermosonication, could be responsible for the breakdown of polysaccharide cells and the leaching of hemicellulose/cellulose from the starch matrix (Tan et al., 2021). It is no coincidence that this result was only observed for the chestnut sample hydrated according to dynamic method. The same sample evidenced a very intense peak related to starch-rich phase degradation at about 300 °C, slightly higher than the other samples. Usually, for polysaccharides, the weight loss occurring in the 250 °C–350 °C range can be attributed to the removal of branches from the main chain and their degradation into volatiles, since the backbone of the chain is pyrolyzed at a much higher temperature. It is no coincidence that for all the samples, up to 800 °C, about 30% residual chain can be detected, which could correspond to inorganic matter and acid-soluble lignin (Karaseva et al., 2019; Wang et al., 2012).

3.4. Volatile components

The volatile compounds of “*Castagne del Prete*” obtained by different rehydration methods were identified through HS-SPME-GC/MS technique. A total of 126 volatile compounds were identified (Table 2), belonging to twelve compound classes: ketones (20), alcohols (17), pyrazines (16), phenols (15), benzene derivatives (15), furans and furanones (15), aldehydes (12), acids (5), esters (3), terpenoids (4), sulphides (2) and pyrroles (2). Depending on the rehydration method used for “*Castagne del Prete*” production, the samples showed a different profile of volatile compounds. In detail, in Fig. 4, are reported the percentages of each class of compounds in relation to the total amount of VOCs detected in each sample. The control sample (C) was mainly characterized by phenols (38%), benzene derivatives (13%), acids (12%), alcohols (11%), ketones (9%), furans and furanones (8%), aldehydes (5%) and terpenoids (4%). A different profile of volatile compounds was found for chestnuts rehydrated by steam (CS). The compounds detected in the highest percentage were pyrazines (79%) followed by ketones (5%), furans and furanones (5%) and aldehydes (3%). Alcohols, terpenoids, acids, benzene derivatives and pyrroles were found in traces. The samples obtained by dynamic method (CD1-CD2), regardless of the chestnut/water ratio, had a similar volatile compound profile to that of “*Castagne del Prete*” obtained by the classic rehydration method (C). The mean values for each class of compounds detected were: acids (34%), phenols (25.5%), benzene derivatives (12%), alcohols (10.5%), ketones (7%), aldehydes (4.5%), furans and furanones (3.7%), terpenoids (2%) and esters (0.6%). Samples rehydrated by static method (CST1-CST2) also showed a similar volatile compound profile regardless of the chestnut/water ratio used. In particular, the compounds detected in higher percentage were acids (43%), aldehydes (15.5%), phenols (12.5%), furans and furanones (11.5%) and ketones

(8%). The other compounds were found in low percentages.

Pyrazines are considered markers of heat treatment and are formed during heating via Maillard sugar-amine reactions and Strecker reaction (Alasalvar et al., 2003). Among the pyrazines, 3-ethyl-2,5-dimethylpyrazine, 2,5-dimethyl pyrazine, 2-ethyl-5-methylpyrazine and 2,6-dimethylpyrazine were found in higher percentages. These compounds are generally characterized by a nutty, roasted, cocoa, mold-like and coffee aroma (Di Renzo et al., 2018) and have been found to predominate in other dried fruits (Wang et al., 2018; Pu et al., 2018). Traces of pyrazines were also found in the samples obtained by static method (CST1-CST2). Acids were found in considerable percentages in both static and dynamic samples, where the main carboxylic acid present was acetic acid while isovaleric and propanoic acids were present in low percentage. Samples CS had low percentage of acids, mainly propanoic and isovaleric acids, while the control sample had low amount of acetic acid. These results are in agreement with Song et al. (2020) that highlighted that dried jujubes, regardless of the drying methods, had higher amount of acids (mainly acetic, propanoic, butanoic and hexanoic acids) respect with fresh red jujubes.

Alcohols were detected in all the samples. The main alcohols detected were isoamyl alcohol, 2-butoxy ethanol, 2,3-butanediol and phenyl ethyl alcohol, characterized for fruity and floral odor notes. Also other authors (Bonneau et al., 2016) found high amount of alcohols in dried fruits assuming that their presence could be due to acid hydrolysis of the corresponding glycosides favored by the temperature applied during drying treatments.

Phenolic compounds were detected with similar and higher values in C, CD1 and CD2 samples (see Table 2) while they were in low amount in CST1 and CST2 samples and in traces in CS sample. The phenolic compounds found in higher amount were phenol, 2-methoxyphenol, 2-methoxy-4-methylphenol and 4-methylphenol. These compounds were also found with lower values in samples CS, CST1 and CST2. As also evidenced by other authors (Aponte et al., 2014; Cirilini et al., 2011), these compounds could be generated by the thermal degradation of lignin that occurs during drying treatment in kilns, where chestnut fruits are placed on a rack located on a flame.

Aldehydes were detected in high amount in CS samples and in both samples obtained by static method (CST1 and CST2). The main representatives were 2-methylbutanal, 3-methylbutanal, benzaldehyde, and hexanal and nonanal. Methyl-branched chain aldehydes are most likely degradation products of the corresponding amino acids upon thermal treatment.

All aldehydes, possessing a linear chain, exhibit fatty, herbaceous and green notes and could result from the degradative oxidation of unsaturated fatty acids, especially oleic and linoleic acids, in which chestnuts are rich (Borges et al., 2007). Aromatic aldehydes, such as benzaldehyde, were present in all the samples, probably as a result of the degradation of aromatic amino acids (Aponte et al., 2013). Benzaldehyde was also found as a major component in Italian chestnut honey (Krist et al., 2004) and Bonvehi and Coll (2003) also reported its contribution to the characteristic aroma profile of unifloral chestnut honey.

Ketones were also detected in the samples with similar percentage, except in the steamed sample, where the values were higher. In the CS sample, acetone was detected with the highest percentage, followed by acetol, acetoxyacetone, 2-octanone and 3-methyl-1,2-cyclopentanedione. The control sample and those obtained by dynamic method also showed the presence of diacetyl, 2-heptanone and acetoin. Some of these compounds have also been found in roasted almonds (Franklin et al., 2018).

Esters were found in very low amount, mainly in CD1 and CD2 samples. Esters are known to contribute to the fruity notes of fruits (Pino & Queris, 2011).

Among the furan derivatives, the most representative volatile compounds were γ -butyrolactone, 2-pentylfuran and 2-acetylfuran which were not present in CD1 and CD2 samples. Moreover, the CS sample

Table 2
Profiles of volatile organic compounds detected in “Castagne del Prete” obtained with different rehydration methods.

RI	Compounds	SAMPLE						Odor notes ^a
		C	CS	CST1	CST2	CD1	CD 2	
Alcohols								
960	Ethanol	nd	nd	0,58 ± 0,01 ^a	nd	nd	nd	alcoholic
975	isopropanol	nd	1,27 ± 0,02 ^a	nd	nd	nd	nd	alcoholic, musty, woody
1181	1-Penten-3-ol	0,22 ± 0,01 ^a	nd	0,11 ± 0,00 ^b	nd	nd	nd	tropical fruity
1182	isobutanol	0,22 ± 0,01 ^a	nd	nd	nd	nd	nd	etherial
1237	isoamyl alcohol	2,01 ± 0,13 ^a	3,59 ± 0,05 ^b	0,83 ± 0,03 ^c	0,94 ± 0,00 ^d	5,66 ± 0,03 ^e	5,50 ± 0,03 ^f	alcoholic, fruity, banana
1256	1-Pentanol	0,38 ± 0,00 ^a	nd	0,27 ± 0,02 ^b	0,18 ± 0,01 ^c	nd	nd	fermented, bready, yeasty
1315	2-Penten-1-ol (Z)	0,20 ± 0,00 ^a	nd	nd	nd	nd	nd	green
1332	2-Heptanol	nd	nd	0,54 ± 0,02 ^a	nd	nd	nd	fresh, lemon, grass
1103	3-Pentanol	nd	2,28 ± 0,11 ^a	nd	nd	nd	nd	sweet, herbal, nutty
1360	1-hexanol	0,52 ± 0,02 ^a	0,65 ± 0,01 ^b	nd	0,51 ± 0,02 ^a	0,55 ± 0,00 ^c	0,48 ± 0,00 ^d	flowery, woody, sweet
1397	2-butoxy ethanol	2,17 ± 0,10 ^a	nd	0,82 ± 0,01 ^b	0,35 ± 0,01 ^c	0,97 ± 0,02 ^d	0,97 ± 0,02 ^d	mild ether
1498	2-ethylhexanol	0,57 ± 0,02 ^a	nd	nd	nd	0,45 ± 0,01 ^b	0,46 ± 0,01 ^b	green, vegetable
1552	2,3-Butanediol	0,53 ± 0,00 ^a	1,85 ± 0,03 ^b	nd	nd	3,55 ± 0,01 ^c	3,21 ± 0,01 ^d	creamy
1639	2-Nonanol	0,38 ± 0,00 ^a	nd	nd	nd	nd	nd	waxy, creamy, citrus
1853	2,5-Hexanediol	nd	nd	0,37 ± 0,00 ^a	nd	0,74 ± 0,02 ^b	0,75 ± 0,02 ^b	–
1836	Benzenemethanol	nd	0,25 ± 0,00 ^a	0,19 ± 0,00 ^b	nd	0,16 ± 0,01 ^c	0,19 ± 0,01 ^b	pleasant aromatic
1865	phenyl ethyl alcohol	1,01 ± 0,00 ^a	1,33 ± 0,04 ^b	0,92 ± 0,01 ^c	0,45 ± 0,02 ^d	nd	nd	flowery
	Tot	8,20 ± 0,03^a	11,22 ± 0,03^b	4,63 ± 0,11^c	2,42 ± 0,02^d	12,08 ± 0,14^e	11,54 ± 0,12^f	
Aldehydes								
920	2-methylbutanal	0,36 ± 0,01 ^a	4,57 ± 0,01 ^b	1,60 ± 0,08 ^c	2,46 ± 0,01 ^d	0,64 ± 0,01 ^e	0,69 ± 0,01 ^f	almond, malty
922	3-methylbutanal	0,88 ± 0,00 ^a	1,72 ± 0,17 ^b	2,91 ± 0,02 ^c	4,22 ± 0,01 ^d	1,50 ± 0,06 ^e	1,60 ± 0,05 ^e	malty, roasty cucumber-like
1090	Hexanal	1,22 ± 0,03 ^a	1,05 ± 0,03 ^b	1,33 ± 0,02 ^c	0,73 ± 0,00 ^d	nd	nd	green, grassy, tallow
1191	Heptanal	nd	0,85 ± 0,00 ^a	1,25 ± 0,04 ^b	0,59 ± 0,00 ^c	nd	nd	fresh, green, herbal
1240	2-Hexenal, (E)	nd	0,26 ± 0,00 ^a	nd	nd	nd	nd	–
1340	2-Heptenal, (E)	nd	nd	0,27 ± 0,02 ^a	nd	nd	nd	fatty, nutty, roasted
1392	Nonanal	nd	1,66 ± 0,11 ^a	1,26 ± 0,04 ^b	1,13 ± 0,01 ^b	nd	nd	waxy, rose, fresh
1502	Decanal	nd	0,72 ± 0,00 ^a	nd	nd	nd	nd	weet, orange, floral
1519	Benzaldehyde	0,55 ± 0,00 ^a	1,74 ± 0,03 ^b	1,54 ± 0,11 ^c	2,76 ± 0,04 ^d	1,42 ± 0,04 ^c	1,54 ± 0,05 ^c	floral, green
1557	5-methylfurfural	nd	nd	0,81 ± 0,03 ^a	0,40 ± 0,00 ^b	nd	nd	almond, sweet, bitter
1910	Tetradecanal	nd	nd	0,50 ± 0,02 ^a	nd	nd	nd	fatty, incense, dry citrus
1644	benzene acetaldehyde	0,74 ± 0,00 ^a	1,75 ± 0,20 ^b	0,71 ± 0,02 ^c	nd	1,33 ± 0,08 ^d	1,43 ± 0,09 ^d	Honey, floral rose
	tot	3,75 ± 0,02^a	14,32 ± 0,26^b	12,18 ± 0,41^c	12,30 ± 0,05^c	4,88 ± 0,19^d	5,25 ± 0,20^d	
Ketones								
778	acetone	0,54 ± 0,01 ^a	11,57 ± 0,22 ^b	1,57 ± 0,09 ^c	1,88 ± 0,01 ^d	0,93 ± 0,05 ^e	1,06 ± 0,06 ^f	sweetish aromatic
900	2-butanone	0,30 ± 0,01 ^a	0,36 ± 0,02 ^b	0,13 ± 0,00 ^c	0,32 ± 0,01 ^a	0,20 ± 0,02 ^d	0,31 ± 0,03 ^a	sharp, sweet
998	diacetyl	1,43 ± 0,01 ^a	0,91 ± 0,02 ^b	0,81 ± 0,00 ^c	0,74 ± 0,00 ^d	0,99 ± 0,04 ^e	1,23 ± 0,04 ^f	strong butter, creamy, caramel
1075	2,3-pentanedione	nd	0,51 ± 0,01 ^a	0,22 ± 0,01 ^b	0,30 ± 0,00 ^c	nd	nd	buttery, nutty, toasted caramellic
1188	2-heptanone	1,60 ± 0,09 ^a	1,23 ± 0,01 ^b	nd	0,67 ± 0,02 ^a	1,10 ± 0,05 ^c	1,27 ± 0,05 ^b	fruity, spicy, woody
1259	3-octanone	nd	nd	nd	0,09 ± 0,00 ^a	nd	nd	fresh herbal, lavender, mushroom

(continued on next page)

Table 2 (continued)

RI	Compounds	SAMPLE						Odor notes ^a
		C	CS	CST1	CST2	CD1	CD 2	
1273	acetoin	0,85 ± 0,00 ^a	0,95 ± 0,04 ^b	0,94 ± 0,04 ^b	nd	1,90 ± 0,12 ^c	2,08 ± 0,20 ^d	butterscotch, butter, yogurt
1272	2-octanone	nd	1,18 ± 0,01 ^a	0,36 ± 0,02 ^b	0,76 ± 0,02 ^c	nd	nd	soapy, fruity
1289	acetol	nd	3,13 ± 0,03 ^a	nd	1,02 ± 0,01 ^b	nd	nd	pungent, sweet caramellic
1348	6-methyl-5-hepten-2-one	0,54 ± 0,00 ^a	nd	0,76 ± 0,01 ^b	0,32 ± 0,01 ^c	1,04 ± 0,03 ^d	1,17 ± 0,04 ^e	citrus, green, musty
1368	2-methyl-2-Cyclopenten-1-one	0,45 ± 0,03 ^a	nd	nd	nd	nd	nd	–
1388	2-Nonanone	0,16 ± 0,01 ^a	nd	nd	nd	nd	nd	fruity
1479	acetoxycetone	nd	2,20 ± 0,15 ^a	nd	nd	nd	nd	fruity, buttery, dairy
1501	2,3-dimethyl-2-cyclopenten-1-one	nd	0,26 ± 0,00 ^a	nd	nd	nd	nd	–
1505	2,3,4-trimethyl-2-Cyclopenten-1-one	nd	nd	0,65 ± 0,02 ^a	nd	nd	nd	–
1513	3-methyl-2-Cyclopenten-1-one	0,43 ± 0,01 ^a	nd	1,13 ± 0,02 ^b	0,19 ± 0,01 ^c	0,47 ± 0,01 ^d	0,63 ± 0,02 ^e	sweet, fruity, fatty
1640	acetophenone	0,48 ± 0,01 ^a	nd	nd	nd	nd	nd	sweet, mimosa, almond
1781	3-methyl-1,2-Cyclopentanedione	nd	1,31 ± 0,02 ^a	nd	nd	0,12 ± 0,00 ^b	0,17 ± 0,00 ^c	sweet caramellic, coffee, woody
1891	3-ethyl-2-hydroxy-cyclopenten-1-one	nd	0,23 ± 0,02 ^a	0,15 ± 0,01 ^b	nd	0,13 ± 0,01 ^b	0,10 ± 0,01 ^c	sweet, brown caramellic, whiskey
1976	1-indanone	nd	nd	nd	nd	0,22 ± 0,01 ^a	0,34 ± 0,01 ^b	–
	Tot	6,79 ± 0,01^a	23,84 ± 0,48^b	6,72 ± 0,02^c	6,29 ± 0,08^d	7,09 ± 0,09^e	8,34 ± 0,08^f	
	Acids							
1459	acetic acid	7,87 ± 0,01 ^a	0,57 ± 0,00 ^b	30,65 ± 1,78 ^c	30,54 ± 1,40 ^c	35,68 ± 0,26 ^d	34,75 ± 0,35 ^d	sour, acid, pungent
1523	propanoic acid	nd	2,74 ± 0,04 ^a	0,54 ± 0,03 ^b	1,18 ± 0,03 ^c	0,54 ± 0,03 ^b	0,53 ± 0,04 ^b	pungent, cheesy, vinegar
1545	isobutanoic acid	0,29 ± 0,01 ^a	0,85 ± 0,00 ^b	nd	0,55 ± 0,00 ^c	nd	nd	sweaty, butter, fatty, rancid
1655	isovaleric acid	0,38 ± 0,02 ^a	4,48 ± 0,25 ^b	1,69 ± 0,14 ^c	2,45 ± 0,09 ^d	1,63 ± 0,04 ^e	1,73 ± 0,04 ^f	sweaty
1811	Hexanoic acid	nd	0,27 ± 0,01 ^a	nd	nd	nd	nd	sweaty, cheesy, fatty
	tot	8,54 ± 0,02^a	8,92 ± 0,20^b	32,87 ± 1,95^c	34,72 ± 1,34^c	37,84 ± 0,19^d	37,00 ± 0,42^e	
	Pyrazines							
1262	methylpyrazine	nd	8,72 ± 0,21 ^a	nd	nd	nd	nd	popcorn, nutty
1331	2,5-dimethyl pyrazine	nd	98,16 ± 0,93 ^a	nd	0,31 ± 0,01 ^b	nd	nd	cocoa, roasted, nuts roast
1337	2,6-dimethylpyrazine	nd	16,92 ± 0,47 ^a	nd	nd	nd	nd	chocolate
1344	ethylpyrazine	nd	3,63 ± 0,04 ^a	nd	nd	nd	nd	popcorn, roasted
1352	2,3-dimethylpyrazine	nd	2,17 ± 0,12 ^a	nd	nd	nd	nd	nutty
1450	3-ethyl-2,5-dimethylpyrazine	nd	195,10 ± 9,72 ^a	0,62 ± 0,02 ^b	0,33 ± 0,01 ^c	nd	nd	nutty
1377	2-ethyl-6-methylpyrazine	nd	10,00 ± 0,35 ^a	nd	nd	nd	nd	roasted potato
1385	2-ethyl-5-methylpyrazine	nd	22,84 ± 1,17 ^a	nd	nd	nd	nd	coffee, nutty
1342	2,6-diethylpyrazine	nd	1,30 ± 0,02 ^a	nd	nd	nd	nd	nutty, hazelnut
1463	2-ethyl-3,5-dimethylpyrazine	nd	6,25 ± 0,27 ^a	nd	nd	nd	nd	burnt, almond roasted, nutty
1489	2-ethenyl-6-methylpyrazine	nd	2,86 ± 0,05 ^a	nd	nd	nd	nd	hazelnut, nutty
1493	3,5-diethyl-2-methylpyrazine	nd	8,79 ± 0,10 ^a	nd	nd	nd	nd	nutty, meaty, vegetable
1490	2-Isobutyl-3-methylpyrazine	nd	0,33 ± 0,01 ^a	nd	nd	nd	nd	herbal, green, sugar
1666	2,5-dimethyl-3-(3-methylbutyl)pyrazine	nd	0,57 ± 0,00 ^a	nd	nd	nd	nd	fruity
1679	1-(5-Methyl-2-pyrazinyl)-1-ethanone	nd	0,69 ± 0,01 ^a	0,29 ± 0,01 ^b	nd	nd	nd	popcorn, nutty
1688	1-(6-Methyl-2-pyrazinyl)-1-ethanone	nd	0,93 ± 0,04 ^a	nd	nd	nd	nd	roasted coffee, cocoa, popcorn
	tot	nd	379,24 ± 5,54^a	0,91 ± 0,03^b	0,64 ± 0,01^c	nd	nd	
	Phenols							
1789	2-Methoxy-5-methylphenol	nd	nd	nd	nd	0,21 ± 0,00 ^a	0,21 ± 0,00 ^a	–
1823	2-methoxyphenol	8,04 ± 0,03 ^a	0,91 ± 0,03 ^b	3,02 ± 0,07 ^c	2,46 ± 0,07 ^d	8,01 ± 0,19 ^a	8,94 ± 0,29 ^c	phenolic, smoky, spicy, vanilla
1840	4-methoxy-3-methylphenol	nd	nd	nd	nd	nd	nd	–

(continued on next page)

Table 2 (continued)

RI	Compounds	SAMPLE						Odor notes ^a
		C	CS	CST1	CST2	CD1	CD 2	
				0,26 ± 0,01 ^a		0,17 ± 0,00 ^b	0,21 ± 0,00 ^c	
1918	2,6-dimethylphenol	nd	nd	nd	nd	0,97 ± 0,05 ^a	1,05 ± 0,06 ^a	sweet, coffee
1936	2-methoxy-4-methylphenol	2,77 ± 0,02 ^a	0,29 ± 0,02 ^b	2,02 ± 0,19 ^c	1,16 ± 0,03 ^d	3,61 ± 0,04 ^e	3,78 ± 0,05 ^f	spicy, vanilla, woody, smoky
1965	phenol	11,00 ± 0,03 ^a	0,45 ± 0,02 ^b	3,62 ± 0,02 ^c	2,91 ± 0,09 ^d	7,06 ± 0,12 ^e	7,21 ± 0,13 ^e	sweet, tarry
2000	4-(1-methylethyl)phenol	nd	nd	nd	nd	0,47 ± 0,03 ^a	0,44 ± 0,03 ^a	woody
2041	p-ethyl guaiacol	0,44 ± 0,01 ^a	nd	0,46 ± 0,00 ^b	0,32 ± 0,01 ^c	0,83 ± 0,04 ^d	0,88 ± 0,04 ^d	spicy, smoky, bacon
2056	2-ethylphenol	0,26 ± 0,01 ^a	nd	1,17 ± 0,02 ^b	nd	0,19 ± 0,01 ^c	0,20 ± 0,01 ^c	phenolic
2155	2,3-dimethylphenol	0,44 ± 0,01 ^a	0,08 ± 0,00 ^b	0,22 ± 0,01 ^c	0,09 ± 0,00 ^d	0,46 ± 0,00 ^e	0,51 ± 0,00 ^f	phenolic, chemical, musty
2225	3,4-dimethylphenol	1,56 ± 0,18 ^a	nd	nd	nd	nd	nd	smoky, sweet, earthy burnt
2046	4-methylphenol	2,64 ± 0,15 ^a	nd	0,87 ± 0,03 ^b	0,94 ± 0,01 ^c	2,24 ± 0,09 ^d	2,33 ± 0,08 ^d	phenolic, narcissus, mimosa
2071	3-methylphenol	nd	nd	nd	0,52 ± 0,00 ^a	1,52 ± 0,09 ^b	1,54 ± 0,08 ^b	medicinal, woody, leathery
2073	2,5-dimethylphenol	0,22 ± 0,00 ^a	nd	nd	nd	0,29 ± 0,00 ^b	0,35 ± 0,00 ^c	sweet, phenolic, smoky
2185	3-ethylphenol	0,26 ± 0,01 ^a	nd	0,28 ± 0,02 ^a	0,16 ± 0,02 ^b	0,57 ± 0,00 ^c	0,64 ± 0,00 ^d	herbal
	tot	27,61 ± 0,29^a	1,73 ± 0,07^b	11,93 ± 0,22^c	8,57 ± 0,09^d	26,60 ± 0,69^e	28,26 ± 0,79^a	
	Esters							
1070	ethyl-2-methylbutanoate	nd	0,57 ± 0,04 ^a	nd	nd	nd	nd	sharp, green apple, fruity
1250	ethylhexanoate	0,12 ± 0,00 ^a	nd	nd	nd	0,17 ± 0,00 ^b	0,19 ± 0,00 ^c	sweet, fruity, pineapple
1437	ethyloctanoate	nd	nd	nd	nd	0,43 ± 0,00 ^a	0,51 ± 0,00 ^b	fruity, winey, apricot
	tot	0,12 ± 0,00^a	0,57 ± 0,04^b	nd	nd	0,60 ± 0,00^b	0,70 ± 0,00^c	
	Terpenoids							
1099	beta-pinene	nd	10,12 ± 0,10 ^a	nd	nd	0,38 ± 0,01 ^b	0,44 ± 0,01 ^c	resinous pine, hay green
1197	limonene	2,20 ± 0,08 ^a	0,26 ± 0,00 ^b	0,22 ± 0,01 ^c	0,23 ± 0,01 ^c	0,58 ± 0,01 ^d	0,49 ± 0,00 ^e	citrus, orange
1266	p-cymene	0,35 ± 0,00 ^a	nd	nd	nd	0,50 ± 0,00 ^b	0,58 ± 0,00 ^c	fresh citrus, woody, spicy
1200	1,8-Cineole	0,18 ± 0,01 ^a	nd	0,31 ± 0,00 ^b	0,04 ± 0,00 ^c	0,74 ± 0,02 ^d	0,88 ± 0,03 ^e	eucalyptus, herbal, camphor
	tot	2,74 ± 0,07^a	10,38 ± 0,10^b	0,52 ± 0,01^c	0,28 ± 0,01^d	2,20 ± 0,03^e	2,39 ± 0,04^f	
	Sulfides							
1085	dimethyl disulfide	nd	1,04 ± 0,03 ^a	nd	nd	nd	nd	sulfurous, cabbage, onion
1372	Dimethyl trisulfide	nd	0,27 ± 0,01 ^a	nd	nd	nd	nd	Sulfurous, cooked onion
	tot	nd	1,31 ± 0,05^a	nd	nd	nd	nd	
	Furans and furanones							
1247	2-pentylfuran	0,51 ± 0,02 ^a	1,86 ± 0,02 ^b	1,68 ± 0,12 ^c	3,09 ± 0,01 ^d	0,84 ± 0,04 ^e	1,07 ± 0,05 ^f	fruity, green, earthy
1483	furfural	nd	nd	1,19 ± 0,03 ^a	nd	nd	nd	woody, almond, baked bread
1743	2(5H)-furanone	nd	nd	0,14 ± 0,00 ^a	nd	nd	nd	buttery
1621	ethyl-2-furoate	nd	nd	nd	nd	1,02 ± 0,05 ^a	1,07 ± 0,06 ^a	balsamic, fruity, floral
1510	2-acetylfuran	1,87 ± 0,01 ^a	2,20 ± 0,16 ^b	2,61 ± 0,15 ^c	1,70 ± 0,09 ^d	nd	nd	sweet, almond, caramellic,
1563	2-methyl benzofuran	nd	nd	0,71 ± 0,02 ^a	0,63 ± 0,02 ^b	nd	nd	burnt, phenolic
1587	2-Acetyl-5-methylfuran	nd	nd	0,36 ± 0,01 ^a	0,25 ± 0,00 ^b	nd	nd	musty, nutty, hay
1264	dihydro-2-methyl-3(2H)-furanone	nd	nd	0,83 ± 0,03 ^a	1,08 ± 0,01 ^b	nd	nd	bready, buttery, nutty
1625	γ-butyrolactone	2,31 ± 0,08 ^a	8,93 ± 0,29 ^b	1,01 ± 0,02 ^c	0,72 ± 0,01 ^d	1,94 ± 0,16 ^e	2,18 ± 0,20 ^a	creamy, oily, caramellic
1636	2,5-dihydro-3,5-dimethyl-2-furanone	nd	nd	0,74 ± 0,03 ^a	nd	nd	nd	–
1647	2-furanmethanol	nd	8,22 ± 0,24 ^a	0,32 ± 0,00 ^b	0,87 ± 0,01 ^c	nd	nd	bready

(continued on next page)

Table 2 (continued)

RI	Compounds	SAMPLE						Odor notes ^a
		C	CS	CST1	CST2	CD1	CD 2	
1730	5-methyl-2-furanmethanol	nd	1,55 ± 0,06 ^a	nd	nd	nd	nd	–
1697	3-methyl-2(5H)-furanone	0,42 ± 0,01 ^a	nd	nd	nd	nd	nd	–
1500	2H-Pyran-2-one	0,35 ± 0,01 ^a	nd	nd	nd	nd	nd	–
2034	dihydro-3-hydroxy-4,4-dimethyl-2(3H)-furanone	nd	1,02 ± 0,01 ^a	nd	nd	nd	nd	cotton, candy
	tot	5,46 ± 0,05^a	23,79 ± 0,61^b	9,60 ± 0,40^c	8,33 ± 0,05^d	3,80 ± 0,26^e	4,31 ± 0,25^e	
	Benzene derivatives							
1116	ethyl benzene	0,42 ± 0,01 ^a	1,37 ± 0,02 ^b	0,11 ± 0,00 ^c	0,12 ± 0,00 ^d	0,27 ± 0,00 ^e	0,32 ± 0,00 ^f	–
1174	o-xylene	1,16 ± 0,01 ^a	0,49 ± 0,02 ^b	0,66 ± 0,03 ^c	nd	0,30 ± 0,01 ^d	0,38 ± 0,01 ^e	geranium
1242	1-ethyl-3-methylbenzene	0,36 ± 0,01 ^a	nd	nd	nd	nd	nd	–
1244	1-ethyl-2-methylbenzene	0,47 ± 0,02 ^a	nd	nd	nd	nd	nd	–
1260	styrene	1,23 ± 0,02 ^a	0,44 ± 0,02 ^b	0,19 ± 0,00 ^c	0,39 ± 0,00 ^d	1,60 ± 0,04 ^e	1,73 ± 0,05 ^f	sweet, balsam, floral
1335	1,2,3-trimethyl benzene	0,79 ± 0,01 ^a	nd	0,21 ± 0,00 ^b	0,22 ± 0,01 ^b	0,34 ± 0,00 ^c	0,41 ± 0,00 ^d	–
1355	anisole	nd	nd	nd	nd	0,74 ± 0,02 ^a	0,73 ± 0,00 ^a	phenolic, gasoline, ethereal
1400	o-methyl anisole	0,55 ± 0,00 ^a	nd	nd	nd	1,24 ± 0,06 ^b	1,31 ± 0,05 ^b	sweet, nutty, floral, walnut
1425	2-methoxy-1,3-dimethyl-benzene	nd	nd	nd	nd	0,29 ± 0,00 ^a	0,24 ± 0,00 ^b	–
1439	p-methyl anisole	nd	nd	nd	nd	1,37 ± 0,06 ^a	1,18 ± 0,01 ^b	naphthyl, narcissus, nutty
1441	m-methyl anisole	nd	nd	nd	nd	1,19 ± 0,04 ^a	1,05 ± 0,03 ^b	–
1533	3,5-dimethyl anisole	nd	0,36 ± 0,00 ^a	nd	nd	0,87 ± 0,03 ^b	0,98 ± 0,04 ^c	–
1735	1,2-dimethoxybenzene	4,64 ± 0,15 ^a	nd	0,33 ± 0,01 ^b	nd	2,25 ± 0,07 ^b	2,33 ± 0,08 ^b	sweet, creamy, vanilla
1739	naphthalene	nd	0,48 ± 0,03 ^a	0,86 ± 0,03 ^b	0,67 ± 0,03 ^c	0,85 ± 0,06 ^b	0,93 ± 0,07 ^b	pungent dry tarry
1806	1,2-dimethoxy-4-methyl-benzene	nd	nd	0,16 ± 0,01 ^a	0,15 ± 0,01 ^a	1,24 ± 0,04 ^b	1,28 ± 0,05 ^b	–
	tot	9,64 ± 0,19^a	3,14 ± 0,08^b	2,52 ± 0,06^c	1,55 ± 0,05^d	12,54 ± 0,33^e	12,83 ± 0,38^e	
	Pyrroles							
1941	2-acetyl pyrrole	nd	3,36 ± 0,19 ^a	0,27 ± 0,02 ^b	0,72 ± 0,00 ^c	nd	nd	roasted, biscuits
2031	1H-Pyrrole-2-carboxaldehyde	nd	0,08 ± 0,00 ^a	nd	nd	nd	nd	musty beefy coffee
	tot	nd	3,44 ± 0,19^a	0,27 ± 0,02^b	0,72 ± 0,04^c	nd	nd	

RI = retention index. RIs were calculated with van Den Dool and Kratz formula. Calculated RIs were compared using online NIST database (<http://webbook.nist.gov/chemistry/>) for high polar column for InnoWAX or similar stationary phases.

All the compounds were identified by the matching RI and MS.

Results are expressed as RAP = Relative Peak Area (area Peak Compound/Area peak Internal Standard)*100.

Each value is expressed as mean ± SD of two replicates. Lowercase letters (a, b, c, d, e, f) in the same row indicate significant differences (p < 0.05) in volatile compounds among chestnuts obtained by the different hydration method.

nd = not detected.

^a based on online databases (www.flavornet.org, and www.thegoodscentscompany.com).

showed the presence of 2-furanmethanol and 5-methyl-2-furanmethanol. The latter two compounds are known as products of Maillard or caramelization reactions and are also derived from the decomposition of polysaccharides (Belitz et al., 2004). Furthermore, 2-pentylfuran could have been generated from linoleic acid under drying (Mandin et al., 1999). Generally, this class of compounds contributes to flavours such as caramel, bread, sweet/fruity and nutty.

Benzene derivatives were found mainly in samples CD1, CD2 and C. The compounds mainly detected were 1,2-dimethoxybenzene, o-xylene and styrene. The samples obtained by the dynamic method had also appreciable amount of o-methyl anisole, p-methyl anisole, m-methyl anisole and 1,2-dimethoxy-4-methyl-benzene. Benzene derivatives were found as the main volatile compounds in fully roasted *P. terebinthus*

fruits (Gogus et al., 2011).

Sulfides (mainly dimethyl disulphide) and pyrroles (mainly 2-acetyl pyrrole) were only detected in CS sample. The 2-acetyl pyrrole, which imparts a roasted/biscuits odor was also found in previous studies on roasting coffee beans (Moon & Shibamoto, 2009).

Terpenoids were mainly found in CS sample and β-pinene was the most representative one. Samples C, CD1 and CD2 were characterized by lower percentages of terpenoids and also showed the presence of limonene, p-cymene, 1,8-cineole. Also, the samples CST1 and CST2 showed traces of terpenoids. Limonene and β-pinene were also found by other authors in *Pistacia terebinthus* (Gogus et al., 2011).

The analysis of the chemical classes of compounds detected in the different “*Castagne del Prete*” samples showed that samples obtained by

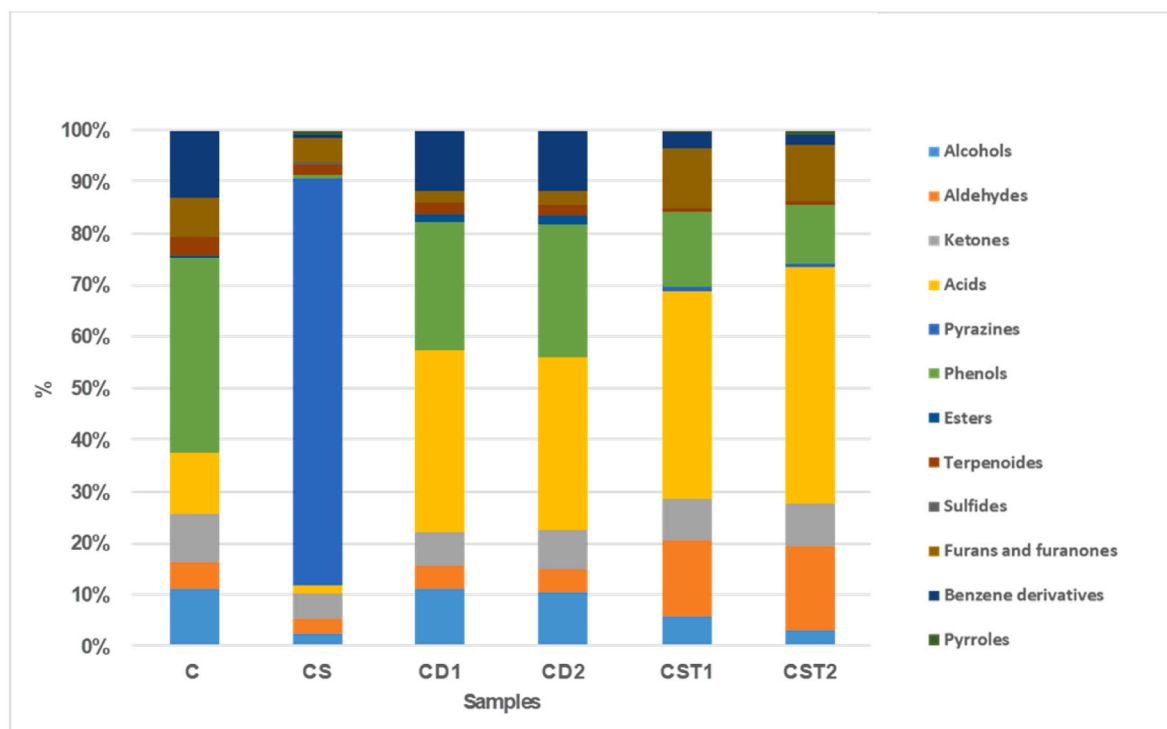


Fig. 4. Classes of volatile organic compounds (VOCs) detected in different samples of “Castagne del Prete” by HS-SPME-GC/MS analysis. Data are reported as percentage of each class of compounds in relation to the total amount of VOCs detected.

various rehydration methods were different, as confirmed by the Principal Component Analysis (PCA) (Fig. 5). Based on the main class of compounds detected, the samples rehydrated by dynamic method (CD1-CD2) were close to the control sample (C) and clearly separated from the

samples obtained by static (CST1 and CST2) and steam (CS) methods. The first two principal components (PC) explained 91.19% of the total variance in the data. In particular, PC1 explained 62.94% of the data variance, while PC2 explained 28.25%. The control, CD1 and CD2

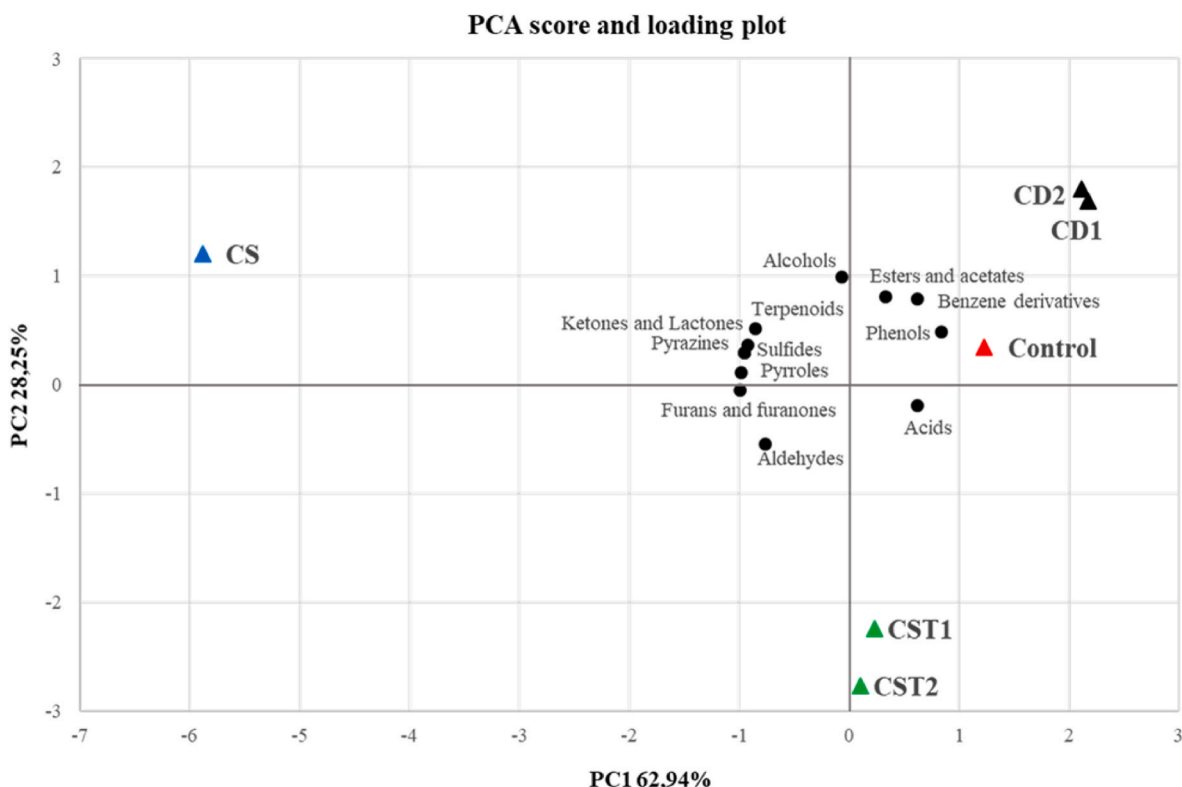


Fig. 5. Principal Component Analysis (PCA) of classes of volatile organic compounds (VOCs) in “Castagne del Prete” obtained with different rehydration methods.

samples were different from the other “Castagne del prete” samples. In fact, as determined by the two PCs (factors), Control, CD1 and CD2 were entirely located at the top of the right section of the graph, whereas CST1 and CST2 were located at the bottom of the right section of the graph. The CS sample, instead, differed from the other chestnut samples and was located alone at the top of the left section of the graph. These outcomes highlighted that “Castagne del Prete” flavour profile is the result of a complex mixture of VOCs. The different rehydration methods influence the final volatile profile of chestnuts which components derived, as also described by other authors (Li et al., 2016; Morini & Maga, 1995), from the degradation of saccharides, protein and lipids, caramelization under drying conditions of saccharides, and Maillard reaction between reducing sugar and amino acids. Furthermore, as morphological analysis shows, such rehydration treatments lead to diverse intra- and intermolecular interaction between amylose, amylopectin and water which could also influence the different release of volatile compounds.

4. Conclusions

This is the first study on the use of thermosonication to rehydrate the ‘Castagne del Prete’, a niche product of the Campania Region. The investigation demonstrated that the treatment developed (thermosonication of chestnuts with chestnut/water ratio 1:5 (w/v) for 5 h at 55 °C, 40 KHz and 70 Watt), allowed to reduce the hydration time, optimizing production and standardising the final product. Specifically, the use of thermosonication helped speed up the hydration processes for the preparation of “Castagne del Prete”, saving energy and time while guaranteeing the flavour and structural characteristics of the finished products. The HS-SPME-GC/MS technique made it possible to evaluate the effect of the different rehydration treatments on the volatile component of the “Castagne del Prete”. Furthermore, Scanning Electron Microscopy and Thermogravimetric analysis proved to be very useful techniques for evaluating the effect of different hydration treatments on chestnuts features, in particular on the structural change of starch and on the distribution of the amylose and amylopectin fractions. These analyses highlighted that the modification and the distribution of pores in the product’s microstructure strongly influence the rate of water absorption by the dried/dehydrated product. These aspects, as well as sensory evaluation, deserve further investigations in future studies to identify the best rehydration condition.

CRedit authorship contribution statement

Santagata Gabriella: Investigation, Formal analysis, Writing – original draft. **Di Renzo Tiziana:** Conceptualization, Investigation, Formal analysis, Writing – original draft, Writing – review & editing, Resources. **Mallardo Salvatore:** Formal analysis. **Reale Anna:** Formal analysis, Writing – original draft. **Cascone Giovanni:** Formal analysis. **Boscaino Floriana:** Formal analysis. **Volpe Maria Grazia:** Conceptualization, Writing – review & editing, Supervision, Resources, Funding acquisition, All authors provided critical revisions and approved the final version of the manuscript.

Declaration of Competing Interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

Data availability

The data that has been used is confidential.

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