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Air drying of chopped chestnuts at several conditions: effect on colour and chemical characteristics of chestnut flour

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Abstract

Fresh chopped chestnuts were dried using a hot air convective tray dryer at different temperatures (45, 65 and 85ºC) and loading densities (2.5 and 6.3 kg/m^2). Drying kinetics were experimentally determined and modelled by means of Page model. Model parameters correlations with temperature and loading density were found. Dried samples were milled and chemical composition (starch, amylose, fat, fibre, proteins, sugars) and colour characteristics of the flour were determined. Total and damaged starch varied significantly with drying temperature and loading density, but not amylose content. Protein and sugars content were also influenced by drying conditions whereas no meaningful differences were observed for fibre and fat content. Lightness of flour chestnut decreased and redness increased with drying temperature. At high loading density, the thermal effects on the flour colour were less promoted.

Introduction

Chestnut (*Castanea sativa* Mill.) is a gluten-free seasonal nut that is mainly consumed fresh, however, some ways of processing like drying make chestnuts available outside the traditional season. Air drying is widely used to preserve food materials (Krokida *et al*., 2003). It is based on the reduction of water activity by moisture loss up to reach physicochemical and microbiological stability. The prevailing mechanism of the water transport during chestnut drying is the water diffusion due to the existence of moisture gradients in the bulk of the material (Moreira *et al*., 2008).

Convective drying kinetics depends on several variables such as temperature, relative humidity and velocity of the drying air; size, shape and loading density of the samples. The effects of these variables on drying rates were studied for many food products (Krokida *et al*., 2003; Kaya *et al*., 2007). In the case of chestnut fruits, air drying temperature has an important role on the total drying time which was not significantly influenced by the air drying velocity showing that internal resistance for water transfer is predominant (Koyuncu *et al*., 2004). Other authors stated that velocity and relative humidity of the hot air show lesser influences on the drying kinetics than temperature (Moreira *et al*., 2005; Kaya *et al*., 2007). Although whole chestnuts drying has been widely reported (Attanasio *et al*., 2004; Koyuncu *et al*., 2004; Moreira *et al*., 2005); the air drying of chopped chestnut previous to the milling operation for making flour is scarcely studied (Correia *et al*., 2007). Namely, few studies have been found using chestnut drying temperatures above the onset temperature of chestnut starch gelatinization, which modifies significantly the rheological behaviour of chestnut flour doughs (Moreira *et al*., 2013).

In starchy products, like chestnuts, it is important to determine the modifications of the chemical properties promoted by temperature during drying process, (such as the amylose/amylopectin ratio, the total and damaged starch content and other chemical properties like fat, sugar or fibre content). Technological aptitude of the flours depends on these parameters. Namely, thermal and rheological behaviours of gluten-free doughs from these flours can be notably influenced by the processing conditions (Moreira *et al*., 2013). A previous study of flour obtained from chestnuts dried at several temperatures, below 70ºC, showed that amylose/amylopectin ratio and resistant starch content are modified during drying (Correia and Beirao-da-Costa, 2012). No tests assessing the influence of load density during drying of chestnuts were performed. Furthermore, physical properties as flour colour are decisive in the costumer acceptance of the final product.

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The main aim of this work is to study the influence of drying temperature on the drying kinetics of layers of chopped chestnut at different loading densities. Page model is applied to modelling the experimental drying kinetics. Finally, the chemical characteristics and colour properties of chestnut flours were experimentally determined and these results are related to both operational factors of drying operation.

Materials and Methods

Chestnut flours production: drying and milling conditions

Fresh chestnuts (*Castanea sativa* Mill.) were purchased in a local market, selected with a uniform size and ripeness and stored in a cold chamber at 4±1ºC until use. Chestnuts initial moisture content was determined after samples drying up to constant weight in a vacuum oven (<15 kPa) at 70ºC (Heraeus Vacutherm 5250 VT6025, Germany) (AOAC, 1995). The average initial moisture content was 45±2%, expressed on a wet basis (w.b.). Chestnuts were hand-peeled, cut in small pieces (3-4 mm edge cubes) and dried in a hot air convective dryer (Angelantoni, Challenge 250, Italy). Chopped samples were placed on a plastic mesh, allowing a transversal drying air flow, testing two loading densities: low loading density (LLD) of 2.63±0.26 kg/m^2 and a high loading density (HLD) of 6.13±0.10 kg/m^2. Relative humidity of the drying air, 30%, and air velocity, 2 m/s, were maintained constant in all experiments. Three temperatures of air drying were studied: 45, 65 and 85ºC. Drying kinetics were determined by weigh at specific drying times until sample moisture content achieved to 13-16% dry basis, d.b. Dried samples were milled in a centrifugal mill (ZM 200, Retsch GMBH, Germany). The average particle size (from 36 up to 47 µm) of the obtained flours was determined after sieving employing standard sieves of 250, 125, 80, 63 and 40µm (Cisa Cedaceria Industrial, Spain). These experiments were at least performed in triplicate. All assayed systems were labelled according to the following nomenclature XLD YY where X corresponds to high (H) or low (L) loading density (LD) and YY shows the drying temperature employed.

Mathematical modelling of drying kinetics

The experimental drying curves were fitted by means of the Page model (Page, 1949):

$$MR = \exp\left(-kt^n\right) \quad (1)$$

where t is the drying time (h), k is the drying rate constant (h^{-n}) and n is a model parameter. The moisture ratio (MR) of chestnut particles was calculated by:

$$MR = \frac{(M - M_e)}{(M_0 - M_e)} \quad (2)$$

where M is the moisture content (d.b.) at any time, M_0 is the initial moisture content (d.b.) and M_e is the equilibrium moisture content of the sample (d.b.), whose values were obtained from (Vázquez *et al*., 2001).

Chemical characterization of chestnut flours

Starch

Chestnut starch was isolated from chestnut fruits following a protocol previously described (Chenlo *et al*., 2011). Total starch (TS, % g starch/g dry flour) was determined using an enzymatic procedure employing the "total starch assay kit" (Megazyme International Ireland, Wicklow, Ireland). This method was approved by the American Association of Cereal Chemists (AACC, 2000). Damaged starch (DS, % g damaged starch/ g flour) was determined by using the enzymatic kit "Starch damage assay procedure" (Starch damage assay procedure, Megazyme International Ireland Limited) (ICC, 1996). The amylose content was determined according to the procedure previously established (McGrance, 1998). The tests were made at least in triplicate.

Fat, fibre, protein and sugar

Total fat amount of flours was determined following ISO standards (ISO, 1982). Total, soluble and insoluble dietary fibre content of flours was evaluated by means of a standard enzymatic-gravimetric method according to AOAC (AOAC, 1996). Flours protein content was established by the Kjeldahl method (AOAC, 1995). Sugar content was determined by HPLC according to the AACC standard method (AACC, 1994). The assays were performed at least in triplicate.

Colour measurements

Colour characterization of chestnut flours was performed using a colorimeter Minolta CR-400 (Minolta, Japan). The apparatus was previously calibrated with standard white tile. The three colour parameters (L*, a* and b*) were determined, where L* defines the lightness and a* (degree of redness or greenness) and b* (degree of yelowness or blueness) are the chromaticity responsible parameters (CIE, 1976). The total colour difference (ΔE*) was calculated in comparison to commercial chestnut

flour data (Torres *et al*., 2013), according to the following equation (Hunter and Harold, 1987):

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

Colour differences can be classified as different ($1.5<\Delta E^*<3$) or very different ($\Delta E^*>3$) (Drlange, 1994). Measurements were performed at least ten times for each flour.

Statistical analysis

The goodness of fitting of experimental data to the different models applied was evaluated from the corresponding coefficients of determination (R^2) and standard deviations. Reduced chi-square (χ^2) and root mean square error (RMSE) calculations were also employed. These statistical values were calculated as follows:

$$\chi^2 = \frac{\sum_{i=1}^{N} (MR_{exp,i} - MR_{pre,i})^2}{N - z} \quad (4)$$

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N} (MR_{exp,i} - MR_{pre,i})^2\right]^{1/2} \quad (5)$$

where MR_{exp} is the experimental dimensionless moisture ratio, MR_{pre} is the predicted dimensionless moisture ratio, N is the number of experimental data points, and z is the number of parameters in the model. Furthermore, significance analysis of data was carried out by means of one-factor analysis of variance (ANOVA). Fisher's least significant test was used to determine significant differences. In the case of positive significance, p values ≤ 0.05, post hoc analyses using the Duncan comparison test were performed to establish statistical differences between the calculated means at each experimental condition tested (SPSS 18.0 statistical package).

Results and Discussion

Drying kinetics

Drying kinetics, MR vs time, of chopped chestnut particles at different experimental conditions are shown in Figure 1. Final MR of samples was 0.13±0.02 because this moisture content was previously determined as adequate for milling operation to produce the chestnut flour. As expected, the drying rate increased with temperature, thus the drying time decreased. Specifically, drying time was shortened from 100 to 75 min and from 180 to 150 min with increasing temperature from 45 to 85ºC at LLD and HLD, respectively. It is also noticeable that the decrease of the drying time is higher from 45 to 65ºC than from 65 to 85ºC for both loading densities. These results show the loading density as a determining variable in the necessary time to dehydrate the product. In chestnuts dried at LLD, water transfer from within the interior to the exterior of the particles is controlled mainly by the resistance in the bulk of the particle. At HLD, most of the chestnut particles are not in direct contact with the air (only the outer layers may be considered as such) but instead the prevalent contact is between particles. Thus, the governing internal resistance in LLD conditions is accompained simultaneously by the resistance associated to water transport through the particle bed. This phenomenon is also favoured by the fact that the interstitial air of the bed has higher humidity content than the external drying air (constant relative humidity and temperature). Additionally, the interstitial air temperature is also lower than external air temperature and close during first period of drying to the wet temperature of air dying. Overall, these simultaneous factors lead to a decrease in the drying rate. From a practical standpoint, simple empirical models, which take into account the existence of the different property transfer mechanisms described above, can be used in the modelling of the particle bed drying. In this case, the well-known Page model (Eq 1) was applied. The model parameters obtained are shown in Table 1. The fitting parameters revealed different trends with temperature and loading density. Thus, parameter k increased notably with increasing temperature and regardless of loading density, although values were substantially lower in the trials at HLD at constant temperature. Meanwhile, the exponent n varied in a narrow range (1.23-1.34), which underlines that this parameter did not depend on the operating conditions but rather on the physical and geometric characteristics of the product. The average value of the exponent n was determined for all the assayed conditions and the parameter k was recalculated (Table 1). The parameter k was correlated following an Arrhenious relation with temperature:

Table 1. Values of the Page model, Eq (1), parameters (k, n) and parameter k with constant n

	Eq. (1)			n constant		
System	k (h^{-n})	n	R^2	k (h^{-n})	n	R^2
HLD 45	0.37±0.12	1.28±0.01	0.999	0.37±0.09	1.28	0.999
LLD 45	1.30±0.15	1.23±0.13	0.998	1.33±0.10		0.998
HLD 65	0.45±0.11	1.28±0.02	0.997	0.45±0.11		0.998
LLD 65	1.46±0.10	1.29±0.03	0.999	1.45±0.12		0.999
HLD 85	0.58±0.07	1.28±0.01	0.999	0.58±0.07		0.999
LLD 85	1.83±0.13	1.34±0.06	0.999	1.69±0.06		0.999

$$\ln k = A + \frac{B}{T} \quad (6)$$

where T (K) is the absolute drying temperature. According to this equation, the fitting parameters A and B were calculated for both loading densities. Hence, Page model for our experiments is given by:

$$MR = \exp(-\exp(2.37 + \frac{-668.2}{T})t^{1.28}) \quad (7)$$

$$MR = \exp(-\exp(2.98 + \frac{-1268.6}{T})t^{1.28}) \quad (8)$$

for LLD and HLD respectively. Figure 1 shows the drying kinetics fitted by means of Eqs (7) and (8). A satisfactory fitting of the experimental data can be observed. The values of χ^2 and RMSE were 6.7 10^{-4} and 2.6 10^{-2} for LLD and 8.8 10^{-5} and 9.2 10^{-2} for HLD, respectively.

Convective air drying of potato cubes showed values of the parameter n similar to those obtained for chestnut particles (Singh and Pandey, 2012). In the case of drying of rice, at similar conditions of the air drying velocity and temperature than the employed in this study, the parameter n values showed lower values in comparison to values obtained in this work (Hacıhafizoglu *et al*., 2008).

Chemical characterization

Starch

Table 2 shows starch properties of chestnut flour dried at different experimental conditions up to the same MR and milled at the same size. It should be emphasized that total starch, as it was determined, corresponds to the amount of non-gelatinizated starch in the samples. The total starch amount of chestnuts dried at LLD dropped significantly with increasing drying temperature (61.5-31.6% w/w d.b.) which indicated that at high temperatures and at LLD condition, the chestnut starch gelatinization was higher. These results were consistent with those obtained using whole chestnut fruits (Attanasio *et al*., 2004) and also with chopped nuts (Correia *et al*., 2009). Furthermore, results agreed with those achieved for the air drying kinetics, due to the gelatinization of starch granules improve the drying rate (Majzoobi *et al*., 2011). This behaviour was not observed for samples dried at HLD condition. An explanation of this result is that although the average moisture content of chestnut particles remains high (variable that favours starch gelatinization by means of the decrease of gelatinization temperature) during longer period of time in comparison to those dried at LLD, a HLD also avoids the heating of the bulk of the particles above starch gelatinization temperature. Consequently, the total starch at HLD conditions remained almost constant with air temperature.

Table 2. Starch content of the different assayed systems (% w/w. d.b.)*

System	Total starch	Damage starch	Amylose
HLD 45	49.2±2.3^{a,b}	4.4±0.3^{a}	19.4±0.3^{c}
LLD 45	61.6±10.1^{b}	7.9±0.1^{a,b}	16.8±0.2^{a,b}
HLD 65	44.0±9.5^{a,b}	6.1±1.8^{a}	19.9±1.3^{c}
LLD 65	51.5±6.6^{a,b}	4.4±0.1^{a}	19.1±0.3^{c}
HLD 85	45.5±5.2^{a,b}	7.6±0.1^{a}	16.6±0.2^{a}
LLD 85	31.6±3.1^{a}	11.3±1.6^{b}	18.6±0.2^{b,c}

*Data are presented as means ± standard deviation. Data value with different superscript letters in columns are significantly different. $p \leq 0.05$.

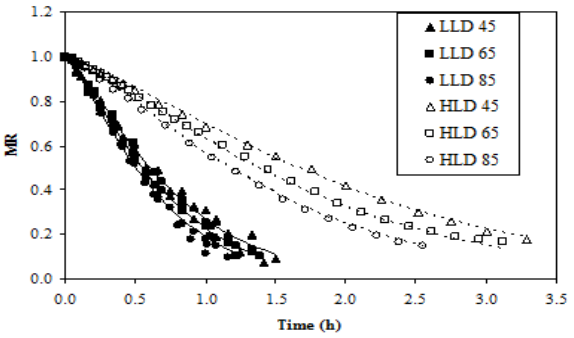

Figure 1. Experimental drying kinetics for chestnuts dried at several temperatures (45, 65 and 85ºC) and loading densities (LLD= 2.5 kg/m^2 and HLD= 6.3 kg/m^2). Lines correspond to Eq (7) (—) and Eq (8) (---)

Damaged starch, which is the starch fraction mechanically (peeling, cut, crushing and milling) and/or thermally (drying and milling) damaged during the flour production process, varied between 4.1 and 11.3% for the assayed systems, Table 2. This range can be considered low and relatively narrow (Tester, 1997). In spite of the effect of drying temperature is not significant, it can be observed the trend of more amounts of damaged starch in the flour dried at high temperature. Mechanical operations can increase the damaged starch proportion at different grades, due to the different mechanical properties of dried products, and hinder the analysis of the thermal effect.

Experimental amylose content obtained for the assayed flours varied between 16.6 and 19.9%. These values were lower than those reported for other gluten-free flours such as corn (24.4%) (Sabaratnam *et al.*, 2013) or rice (22.8%) (Vilaplana *et al*., 2012) flours. Furthermore, they are far from the values determined for fresh chestnuts (33.1 and 32.4%) from other *Castanea sativa* varieties (Correia and Beirao-da-Costa, 2012). The later authors also found that amylose content increases with the drying temperature (from 50 to 70ºC), due to the increase in activity of enzymes such as α-amylase, β-amylase,

Table 3. Chemical composition of the different assayed systems (% w/w. d.b.)*

System	Total fat	Total dietary fibre	Soluble fibre	Insoluble fibre	Total protein	Total sugar
HLD 45	1.43±0.06^{a}	4.53±0.11^{a}	1.39±0.06^{c}	3.14±0.10^{a}	6.71±0.10^{c}	16.4±0.5^{a}
LLD 45	1.44±0.05^{a}	4.52±0.17^{a}	1.36±0.04^{c}	3.15±0.21^{a}	6.74±0.05^{c}	17.2±0.3^{a}
HLD 65	1.51±0.07^{a}	4.57±0.04^{a}	0.93±0.03^{b}	3.64±0.04^{b}	6.42±0.04^{b}	19.0±0.3^{b}
LLD 65	1.50±0.07^{a}	4.59±0.08^{a}	0.86±0.03^{b}	3.73±0.06^{b,c}	6.41±0.06^{b}	19.8±0.3^{b}
HLD 85	1.54±0.07^{a}	4.61±0.08^{a}	0.56±0.02^{a}	4.05±0.10^{c}	6.21±0.04^{a,b}	21.4±0.4^{c}
LLD 85	1.63±0.05^{a}	4.63±0.07^{a}	0.53±0.02^{a}	4.10±0.08^{c}	6.16±0.06^{a}	22.4±0.4^{d}

*Data are presented as means ± standard deviation. Data value with different superscript letters in columns are significantly different. $p \leq 0.05$.

glucoamylase and pullulanase, during the chestnut drying process. The peak of activity for these enzymes occurs at temperatures between 55 and 60ºC (Matherwson, 1998). Our results showed maximum values of amylose content at 65ºC for LLD and HLD conditions indicating the existence of high enzymatic activity in the intermediate temperature assayed.

Fat, fibre, protein and sugar

Total fat, total dietary fibre, soluble and insoluble fibre, protein and sugars content of chestnut flours are shown in Table 3. Fat (1.43-1.63% w/w d.b.), total fibre (4.53-4.63% w/w d.b.) and insoluble fibre (3.14-4.10% w/w d.b.) content were lower than those observed by other authors (Correia *et al*., 2009), while soluble fiber (0.53-1.39% w/w d.b.) and protein (6.16-6.71% w/w d.b.) content were in the same range (Gonçalves *et al*., 2010; Torres *et al*., 2013). Total sugar content (16.4-22-4% w/w d.b.) was consistent with the results previously found for several chestnut varieties (Míguez Bernárdez *et al*., 2004). Both total fat and fibre content were not significantly modifed by the chestnuts drying conditions, Table 3. In contrast, the amount of soluble fibre decreased significantly with increasing the drying temperature due to the thermal degradation of its components (Hincapié *et al*., 2010). Drying at different temperatures also affected the protein content in the flours. The amount of protein decreased significantly with increasing the drying temperature. This result can be explained by the thermal weakening of proteins (Scharnagl *et al*., 2005). Total sugar content increased with increasing the drying temperature in a narrow range. This trend can be partially explained by the fact that both amylose and amylopectin can be hydrolyzed by amyolitic enzymes, which produces an increment in the amount of depleted sugars as glucose (Nomura *et al*., 1995). Nevertheless, the relationship among starch and sugar content is not clear and difficult to establish because several factors influence the enzymatic process (presence of damaged starch, crystalline structure, amylose/amylopectin ratio, etc) and other non-enzymatic processes can be simultaneously developed (Nomura *et al*., 1995). At the same drying temperature, the loading density employed during the drying process did not affect significantly the chemical composition of the samples, with the exception of the sugar content of the flour from chestnuts dried at 85ºC, where this amount rose with decreasing the loading density.

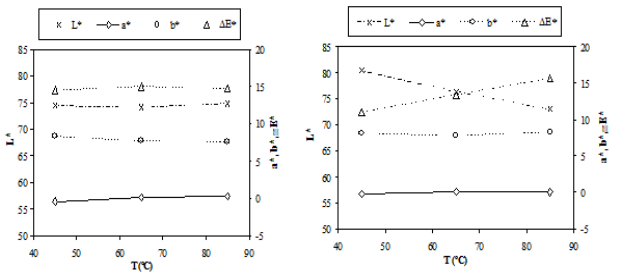

Figure 2. Trends of colour parameters with drying temperature for chestnut flour: (a) high loading density (HLD) and (b) low loading density (LLD)

Colour measurements

Figures 2a and 2b show the experimental colour paramenters of tested chestnut flours submitted to drying at HLD and LLD conditions, respectively. Flours exhibited yellow as the predominant colour. In comparison to commercial chestnut flour, all samples shared less lightness, yellowness and redness (Torres *et al*., 2013). ΔE^* values (Eq 3) showed that every flour could be considered as very different when compared with commercial chestnut flour due mainly to its less yellow colour with higher whiteness. This fact can be considered as positive for consumer acceptance (Lamsal and Faubion, 2009). The colour parameters of chestnut flours showed significant differences with the drying conditions. Parameter L^* ranged from 74.11 to 80.42, a^* from -0.41 to 0.30 and b^* from 7.16 to 8.35. In comparisson with other gluten-free flours, the studied chestnut flours presented higher L^* and a^* and lower b^* values than potato flour (Singh *et al*., 2003); lower L^* and b^* and higher a^* values than corn flour (Sandhu *et al*., 2007); higher a^* and b^* and lower L^* values than rice flour and values in the same order for the three parameters than those obtained for

buckwheat flour (Torbica *et al*., 2012).

Drying conditions modified significantly the colour parameters of the studied flours. The highest ΔE^* was observed at the highest drying temperature and at LLD. L^* values dropped with increasing of the drying temperature at LLD. Flours achieved higher redness (higher a^* values) with increasing temperature from 45 to 85ºC. This can be explained by the fact that non-enzymatic redness reactions (Maillard reactions) can take place during the drying process, and they can be favored by thermal treatments (Moreira *et al*., 2005) and also by the high sugar content (Table 3) (Gómez *et al*., 2008). ΔE^* varied as a consecuence of these changes on the colour parameters. Finally, the values of b^* did not show significant differences with temperature.

Conclusions

Drying conditions of chopped chestnut modify different physicochemical properties of the chestnut flours. Drying temperature and loading density modify total and damaged starch amounts, but not amylose content. High drying temperature decreases lightness and increases the flour redness, while high loading density minimizes the thermal impact on flour colour. Experimental drying kinetics are modelled using Page model. Model parameters depend on air drying temperature and correlations are successfully proposed. The measured characteristics indicate that the studied chestnut flours show acceptable technological aptitudes in comparison to other gluten-free commercial flours.

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