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Almond by-products: extraction and characterization of phenolic compounds and evaluation of antioxidant activity, odor notes and rheological behavior of composite doughs with wheat flour

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Running title: Use of almond by-products in bakery

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Abstract

Blanched skins and blanching water, by-products of almond processing, were evaluated as potential ingredients of bakery products. The research included three phases: i) optimization of skin drying; ii) optimization of quali-quantitative determination of phenolic compounds, by comparing three extracting protocols; iii) assessment of the impact of by-products on the rheology of composite meals with wheat flour. The least time-consuming drying mode (at 60 °C for 30 min) retained better odor notes, higher content of phenolics (813.89 µg/g by HPLC, with the most effective extracting method) and greater antioxidant activity than sun-drying. Blanching water showed 917.46 µg/mL phenolics. Dried almond skins altered the alveograph and farinograph indices of dough at doses higher than 3 and 5 g 100/g, respectively, whereas blanching water did not cause significant changes. Therefore, almond skins could be used in products tolerating weak gluten network, such as cookies, whereas blanching water could be added to any bakery good.

Keywords: almond skins; almond blanching water; bioactive compounds; antioxidant activity; rheological properties.

1. Introduction

Almonds (*Prunus dulcis* (Mill.) D.A. Webb or *Amygdalus communis* L.) are largely used in the preparation of several traditional bakery and confectionery products including almond cookies, marzipan and almond milk (Bennet, 2016). The first processing step in the production of these delicacies is the removal of the brown skin from almonds, by means of blanching in hot water and subsequent mechanical peeling. Skins account for 6-8% of the seed (Garrido, Monagas, Gómez-Cordovés & Bartolomé, 2008) and are mainly destined to cattle feeding (Grasser, Fadel, Garnett &

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DePeters, 1995). Blanching water represents merely a waste, and the producers have to face costs for its disposal.

However, blanched skins and blanching water contain several antioxidant bioactive compounds, such as phenolic acids, flavan-3-ols, flavonols, and flavanones (Chen, Milbury, Lapsley & Blumberg, 2005; Garrido et al., 2008; Mandalari et al., 2010) and act synergistically with other dietary antioxidants to protect LDL from oxidation (Sang et al., 2002). To increase the value of almond by-products, so as to reduce economic and environmental issues, the bioactive compounds could be extracted or, alternatively, the by-products themselves could be used as food ingredients. Even when baking may result in the modification of phenolic content and composition, nevertheless the addition of phenolic-rich by-products results in an overall improvement of the health-promoting value of cereal-based end products (Laddomada, Caretto & Mita, 2015; Pasqualone et al., 2014; 2015; 2016). The direct use of by-products would encompass the costs of extraction and, in case of blanched skins, would also contribute dietary fiber, mostly insoluble (Mandalari et al., 2010).

Bakery products such as cookies and bread, are considered a suitable carrier for functional ingredients (Laddomada et al., 2015; Rahaie, Gharibzahedi, Razavi & Jafari 2014). Functional food production, however, may involve quality drawbacks in terms of possible alterations of physico-chemical and sensory features. Fiber-rich functional ingredients, in fact, could interfere with gluten formation. These awkward would be minimal in case of using concentrated bioactive extracts from almond by-products, but could be more evident in case of direct addition of blanching water and, above all, of blanched skins to wheat flour.

Therefore, after evaluating the physico-chemical properties, odor notes, antioxidant activity, and content of phenolic compounds of blanching water and blanched almond skins - the latter not dried and dried in different conditions- the aim of this work has been to evaluate the potential of these by-products as food ingredients in view of obtaining cereal-based bakery products.

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The research followed a multi-step approach and was developed in three main phases: i) optimization of the drying conditions of blanched almond skins; ii) optimization of the quali-quantitative determination of phenolic compounds of blanched skins and blanching water, by comparing three different protocols; iii) determination of the impact of dried blanched skins (powdered) and blanching water on the rheological behavior of composite meals with wheat flour.

2. Materials and Methods

2.1. Sampling of almond by-products

Blanched almond skins (detached by soaking almonds in water at 95 ± 2 °C for 4 ± 1 min, followed by mechanical peeling) and blanching water were collected at an almond processing industry (Calafiore S.r.l., Floridia, Siracusa, Italy). Two different samplings were carried out. Blanched almond skins were dried by a rotary air drier (Scirocco, Società Italiana Essiccatoi, Milano, Italy) at: i) 60 °C for 30 min; ii) 45 °C for 90 min; iii) 32 °C for 150 min. Natural sun-drying at 38-40 °C for 6-8 h was also carried out. Dried blanched almond skins were milled (Cutting Mill SM 100, Retsch, Haan, Germany) to a particle size in the range 450-500 µm. Not dried blanched almond skins were milled in the same way, after lyophilization (Analitica De Mori, Milano, Italy).

2.2. Preparation of wheat flour composite meals containing almond by-products

Refined wheat flour type 0 (*Triticum aestivum* L.) (Molini Spigadoro, Bastia Umbra, Italy), having 10.4 g/100 g protein and 13.7 g/100 g moisture, was used to prepare composite meals containing 3

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g/100 g, 5 g/100 g, 7 g/100 g and 10 g/100 g of almond skins dried at 60 °C for 30 min, opportunely milled.

2.3. Determination of physico-chemical properties of almond by-products

The moisture content of blanched almond skins was determined by drying (Eurotherm, Gibertini, Novate Milanese, Italy) at 105 °C until constant weight; a_w was determined by AquaLab Vapor Sorption Analyzer (Decagon Devices, Pullman, WA, USA) according to manufacturers' instructions. The lipid fraction was extracted by Soxhlet apparatus with diethyl ether. Color parameters *L**, *a**, *b** were determined by Chromameter CR-300 (Minolta, Osaka, Japan), under the illuminant D65. Wheat flour and composite meals were put into the granular materials attachment CR-A50 (Minolta, Osaka, Japan) of the colorimeter, whereas blanching water was put into a Petri dish placed on a white paper sheet and the color of a 1-cm thick layer of sample was determined. Brown index was calculated as 100 – *L**.

2.4. Sensory determination of main odor notes of almond by-products

The intensity of the main odor notes of almond blanched skins and blanching water was determined by 8 trained panelists (four males, four females, aged between 24 and 49 years), selected for their reliability, consistency and discriminating ability as in Pasqualone et al. (2011). All the samples, identified by alphanumerical codes, were randomly served to panelists into plastic cups covered by an aluminum foil. The odor notes "leafy", "rancid" and "sour" were rated on an anchored line scale that provided a 0-9 score range, in the conditions reported in Pasqualone et al. (2011).

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2.5. Extraction of phenolic compounds from blanched almond skins

The phenolic compounds were extracted from blanched almond skins according to three different protocols (Garrido et al., 2008; Laddomada et al., 2017; Mandalari et al., 2010) (Figure 1). Two of these protocols (Garrido et al., 2008; Mandalari et al., 2010) were slightly modified by substituting methanol/HCl 0.1% (v/v) with 80:20 acetone/water (v/v), based on a preliminary comparison of the extracting efficiency of these two different solvent combinations. Internal standard solution (10 μL of 1.5 mg/mL 3,5-dichloro-4-hydroxybenzoic acid in 80:20 methanol/water v/v) was added to each sample prior to proceed with extraction. At the end of each protocol, the dried extracts were dissolved in 2 mL of 80:20 methanol/water (v/v) and used for analyses.

2.6. Extraction of phenolic compounds from almond blanching water

An aliquot (20 mL) of blanching water was centrifuged at 4,200 *g* for 5 min, then the supernatant was lyophilized. The dry residue was extracted two times with 20 mL acetone/water (80:20), under mixing and sonication for 10 min (LBS1-10 sonicator, Tecno Lab, Brescia, Italia), followed by centrifugation (10 min at 5000 *g*, 4°C). The acetone/water extracts were combined and dried by rotary evaporator. The same procedure was repeated without sonication to point out the effect of ultrasound. The dried extracts were dissolved in 2 mL of 80:20 methanol/water (v/v) and used for analyses.

2.7. HPLC analysis of phenolic compounds

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An aliquot of 50 µL of phenolic extract obtained according to the above reported procedures was filtered on 0.45 µm polytetrafluoroethylene (PTFE) filters (Teknokroma, Barcelona, Spain) and quali-quantitatively analyzed using an Agilent 1100 Series HPLC-DAD system (Agilent Technologies, Santa Clara, CA, USA) equipped with a reversed phase C18(2) Luna column (Phenomenex, Torrance, CA, USA) (5 µm, 250 × 4.6 mm) according to (Laddomada et al., 2017). Peaks were identified by comparing their retention times and UV-Vis spectra to those of authentic phenolic standards. All phenolic compounds were quantified via a ratio to the internal standard (3,5-dichloro-4-hydroxybenzoic acid) added to every sample and using calibration curves of phenolic standards.

2.8. Determination of in vitro antioxidant activity

The *in vitro* antioxidant activity of the phenolic extracts was assessed by the 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging capacity assay in the conditions reported in (Pasqualone et al., 2015), and was expressed as µmol of 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox).

2.9. Farinograph and alveograph analyses

The farinograph (Brabender instrument, Duisburg, Germany) and alveograph (Tripette et Renaud, Chopin Technologies, Villeneuve-la-Garenne, France) analyses of wheat flour and composite meals with almond by-products were carried out according to the methods AACC 54-21 (AACC International, 2000) and UNI 10453 (UNI, 1995), respectively. When almond blanching water was tested, it totally replaced the amount of liquid (water or 2.5 g/100 mL NaCl water solution for farinograph and alveograph analysis, respectively) added to control wheat flour.

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2.10. Statistical analyses

Two different samplings were carried out, and all analytical tests were carried out in triplicate. Data were analyzed using SigmaStat version 11.0 software (Systat Software Inc., Chicago, IL, USA). Chemical data are presented as mean values ± standard deviation of three independent experiments. One-way ANOVA followed by Bonferroni's post-hoc comparisons tests was performed to establish significant differences between means.

3. Results and discussion

3.1. Moisture content, odor notes, antioxidant activity and color of almond by-products

All drying treatments effectively reduced the moisture content and a_w of blanched almond skins in comparison with not dried blanched almond skins (Table 1). Among the four drying conditions considered, skins dried at 60 °C for 30 min showed the lowest moisture content and a_w, whereas those dried at 32 °C for 150 min were the moistest ($p < 0.05$). Considering that blanched and peeled almonds are oven-dried at 70 °C to allow successive milling into flour, the skins could be dried immediately after turning off the oven, so as to recover the residual heat and save energy. Sun-drying (at 38-40 °C for 6-8 h) could be equally inexpensive, but depends on weather conditions and makes difficult keeping high hygienic standards.

Moisture content and a_w data allow to expect a relatively long shelf life for all the examined dried samples. However, drying does not prevent oxidative degradations, which could be important in materials containing polyunsaturated lipids, such as almond skins. The lipid content, indeed, accounted for approximately 22 g/100 g d.m., in agreement with other researches (Mandalari et al.,

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2010). As a consequence, it was not surprising that the sensory panelists detected an intense rancid odor note (score 6.3 ± 0.2) in sun-dried blanched skins, due to both exposure to light and longer duration compared to the other drying conditions considered. The other dried samples were characterized by an intense leafy odor (score from 5.8 to 6.4), with negligible rancid odor. No statistically significant differences were observed among the three oven-based drying conditions for all the odor notes considered ($p < 0.001$). Not dried skins exhibited a well-perceivable sour note, probably coming from fermentation processes occurred during shipping to the processing company and successive storage at room temperature. This odor note was substantially lost with drying, irrespective of the thermal conditions adopted. Blanching water was characterized by moderate leafy odor and slight sour odor, with almost no rancid note. Indeed, blanching water is known to contain only traces of lipids, typically subjected to rancidity (Mandalari et al., 2010).

The *in vitro* antioxidant activity of not dried almond skins, determined by the DPPH radical scavenging capacity assay, accounted for 40.37 μmol Trolox/g. A significant increase was observed after drying, irrespective of the conditions adopted, probably due to the formation of Maillard-related antioxidant products (Garrido et al., 2008; Piga, Del Caro & Corda, 2003). Significant differences of antioxidant activity were assessed among the drying treatments, with the highest values in skins dried at 45 °C for 90 min and the lowest in sun-dried skins, in agreement with the sensory evaluation of rancid odor. The antioxidant activity of blanching water was similar to that reported for herbal medicinal products such as sage tea (aqueous infusions of *Salvia officinalis* L.), which displays an antioxidant activity in the range 4-18 μmol Trolox/mL (Walch et al., 2011).

All by-products were brown colored, with red tones. This color was the result of the enzymatic oxidation of phenolics to brown quinones due to air exposure (Taranto et al., 2017), as well as to the probable formation of several Maillard reaction products during drying. Significantly higher values of brown index were observed in sun-dried almond skins, which also showed the highest value of

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red index and the lowest yellow index. Not dried almond skins, on the contrary, showed the lowest brownness. Blanching water was markedly reddish-brown, indicating that colored compounds were water soluble.

3.2. Phenolic compounds of almond by-products

In order to optimize the extraction of phenolic compounds, three different methods were compared schematized in Figure 1. The method proposed by Laddomada et al. (2017) involved a basic hydrolysis, whereas the protocol of Mandalari et al. (2010) included a sonication step. Both these treatments were aimed at freeing the insoluble phenolic compounds linked to polymers of the plant cell wall. In particular, the effectiveness of basic hydrolysis in enhancing the extracting yield of phenolic compounds was previously evidenced in wheat bran (Laddomada et al., 2017; Pasqualone et al., 2017). The protocol of Garrido et al. (2008), instead, neither required these treatments nor involved the preliminary lipid removal included both in Laddomada et al. (2017) and in Garrido et al. (2008) methods.

Table 2 reports the content of phenolic compounds extracted from not dried blanched almond skins. Overall, the comparison of different extracting conditions allowed to point out their significant effect ($p < 0.05$) on the quali-quantitative pattern of phenolic compounds. These findings resulted from the complex combination of chemical and physical phenomena varying from one extracting method to another: hydrolysis of glycosides, decomposition of aglycones, partition between solvents having different polarity. In particular, not dried blanched almond skins showed total amounts of phenolic compounds ranging from 1110.12 μg/g d.m., determined according to Garrido et al. (2008), to 1773.35 μg/g d.m. extracted according to Laddomada et al. (2017). As expected, the lowest amount of phenolic compounds was measured by means of the method of Garrido et al.

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(2008). However, although less effective than the other two protocols, this method allowed to recover interesting amounts of the targeted molecules, and had the advantage of being easier to manage and faster.

The most abundant compounds in not dried blanched almond skins were flavan-3-ols, irrespective of the method of extraction adopted. Interestingly, (+)-catechin prevailed on (-)-epicatechin in the extracts obtained with the methods of Laddomada et al. (2017) and Mandalari et al. (2010), whereas (-)-epicatechin was the most represented when the method of Garrido et al., (2008) was carried out. These observations were confirmed also in dried blanched skins (Tables 3-5). It has to be considered that (-)-epicatechin is more hydrophobic than its stereoisomer (+)-catechin (Crozier, Ashihara & Tomás-Barbéran, 2001), and probably was partly lost during the lipid removal step included in the protocols of Mandalari et al. (2010) and Laddomada et al. (2017). Among phenolic acids, procatechuic acid was the most represented in the extract obtained from not dried skins by means of Mandalari et al. (2010) and Laddomada et al. (2017) methods, whereas vanillic acid, less polar (Noubigh, Abderrabba & Provost, 2009), was the most abundant with Garrido et al. (2008) protocol. Among flavonol aglycons, isorhamnetin was significantly more abundant in the extracts obtained by the method of Garrido et al. (2008). Again, these findings agree with the water/oil partition coefficients of flavonols, because aglycons are more lipophilic than conjugates (Rothwell, Day & Morgan, 2005). The presence of a conjugate moiety of any form, indeed, aids hydrophilicity and solubility.

Tables 3-5 show the concentration of phenolic compounds of blanched almond skins dried at different conditions, determined according to Garrido et al. (2008), Mandalari et al. (2010) and Laddomada et al. (2017), respectively. The total amounts of phenolic compounds extracted from dried skins were always lower than those extracted from not dried skins, indicating that oxidative

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and other degradative phenomena occurred during drying. In particular, the most evident decrease affected flavan-3-ols, except for (+)-catechin in Mandalari et al. (2010) extracts.

Significant differences were observed also between drying conditions. The extracts obtained according to Mandalari et al. (2010) (Table 4) magnified these differences: the content of phenolic compounds ranged from 317.06 μg/g d.m. to 857.11 μg/g d.m. Oven drying, irrespective of the temperature/time combination chosen, was always more effective than sun-drying in preserving phenolic compounds. Moreover, perusal of data in Tables 3-5 points out that drying at 45 °C for 90 min or at 60 °C for 30 min allowed to retain the highest levels of phenolic compounds, without significant differences among these two treatments. Drying at 60 °C for 30 min is faster than the other thermal treatments, therefore could be easily transferred at industrial level, where speed and productive capacity are essential factors. Besides, this thermal treatment led to dried skins having lower final moisture content (Table 2).

Table 6 reports the content of phenolic compounds of almond blanching water, which showed (+)-catechin as the most abundant compound. Overall, the phenolic profile of blanching water was similar to that of skins, with the exception of the aglycones, that were almost absent. Isorhamnetin, in particular, strongly decreased, probably due to its low polarity. Therefore, blanching water tended to solubilize and retain compounds having high polarity. Moreover, to point out the effect of ultrasound, the extraction of phenolics from blanching water was carried out with and without sonication. The latter condition gave worst results, in fact (+)-catechin was quantified in half amounts than with sonication. The ultrasound treatment probably promoted the liberation of the catechin monomers from tannins (Ferraretto & Celotti, 2016).

3.3. Rheological properties of composite meals and dough containing almond by-products

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Based on moisture content, antioxidant activity and phenolic quali-quantitative profile of blanched almond skins dried in different ways, only those dried at 60 °C for 30 min were considered for preparing composite meals with refined wheat flour. At this purpose, they were opportunely milled and added to flour at increasing levels from 3 g/100 g to 10 g/100 g. Besides, also blanching water was used to prepare dough to be tested for rheological properties.

Table 7 reports the results of farinograph and alveograph analyses of the dough obtained from composite meals with blanched almond skins or blanching water, compared with control dough without supplementations. The mixing properties of dough, determined by farinograph, show that dough stability (i.e. the time needed to reach the dough consistency of 500 Brabender Units) did not change significantly with addition of dried almond skin powder up to 5 g/100 g, followed by marked increases at higher levels. Accordingly, the softening index (i.e. the loss of dough consistency after 12 min), significantly decreased only from 7 g/100 g onwards. On the contrary, dough development time (i.e., the time needed from the first addition of water to reach the maximum consistency) did not show statistically significant differences among samples ($p < 0.05$). Water absorption capacity (i.e., the percentage of water required to yield a dough consistency of 500 Brabender Units) of the composite meals was in the range from 59.2 g/100 g to 61.2 g/100 g. Due to their fiber content, the almond skins influenced water absorption of dough, making it significantly ($p < 0.05$) and progressively increase, compared to control, as the amount added increased. If the effect of almond skins on the main farinograph indices was evident, on the contrary the addition of blanching water did not cause significant changes ($p < 0.05$).

The alveograph indices were both negatively affected by the addition of skin powder. The deformation energy (i.e. the dough “strength”) significantly decreased at doses as low as 3 g/100 g ($p < 0.05$). The tenacity/extensibility ratio (P/L) varied accordingly, with significant increases

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starting from 3 g/100 g of skins added. Again, the effect of blanching water was negligible also on alveograph indices.

These findings were mainly due to the hygroscopic effect of fiber, which is known to be abundant in almond skins (Mandalari et al., 2010). Furthermore, fiber interferes with the formation of a complete and strong gluten network. However, in certain bakery products, such as cookies, additional ingredients such as fats and/or emulsifiers are usually included. During mixing, lipids act as a lubricant, competing with water to coat the surface of flour, and preventing a complete formation of the gluten network (Manohar and Rao, 1999). In cookies, therefore, the values observed for farinograph and especially for alveograph indices, even at the highest level of supplementation, would be well tolerated since the friability typically required in these products takes advantage from weak gluten network and the addition of fats and/or emulsifiers could mitigate the excessively high P/L value (Addo and Pomeranz, 1992; Agyare, Addo, Xiong & Akoh, 2005). Blanching water, instead, did not affect the rheological properties of dough and could therefore be added to any kind of bakery end product.

4. Conclusions

This study has ascertained the great potential of almond processing by-products as food ingredients, with the aim of improving the health value of cereal-based bakery products.

Blanched skins show the advantage of being, after proper drying, relatively stable to oxidation and easy to store and manage. Blanching water, instead, could undergo microbiological spoilage and should be necessarily used fresh, soon after production, so that an ideal layout would see an almond-processing industry joined, on place, with a second plant exploiting the liquid by-product.

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The results showed that blanching water did not affect the rheological properties of dough and could therefore be added to any kind of bakery end product. On the contrary, blanched almond skins could be added to cookies, which formulation usually includes other ingredients, able to mitigate the negative effect of skins on dough rheology.

The use of almond by-products for food specialties with functional properties could represent an important approach for a better qualification of this crop, in view of fulfilling consumer expectations for healthy niche products.

Acknowledgments

The research has been carried out with the financial support of "Cluster Tecnologici Regionali" Regione Puglia FSC 2007–2013, Progetto Bioteca QCBRAJ6. The authors acknowledge Leone D'Amico for skillful assistance in HPLC analyses. Support from the Wheat Initiative to work within the Expert Working Group on Improving Wheat Quality for Processing and Health and build international collaborations is highly appreciated.

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Figure caption

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Figure 1. Flow chart of the main steps of the protocols of Laddomada et al. (2017), Mandalari et al. (2010) modified, and Garrido et al. (2008) modified, used to extract phenolic compounds from blanched almond skins.

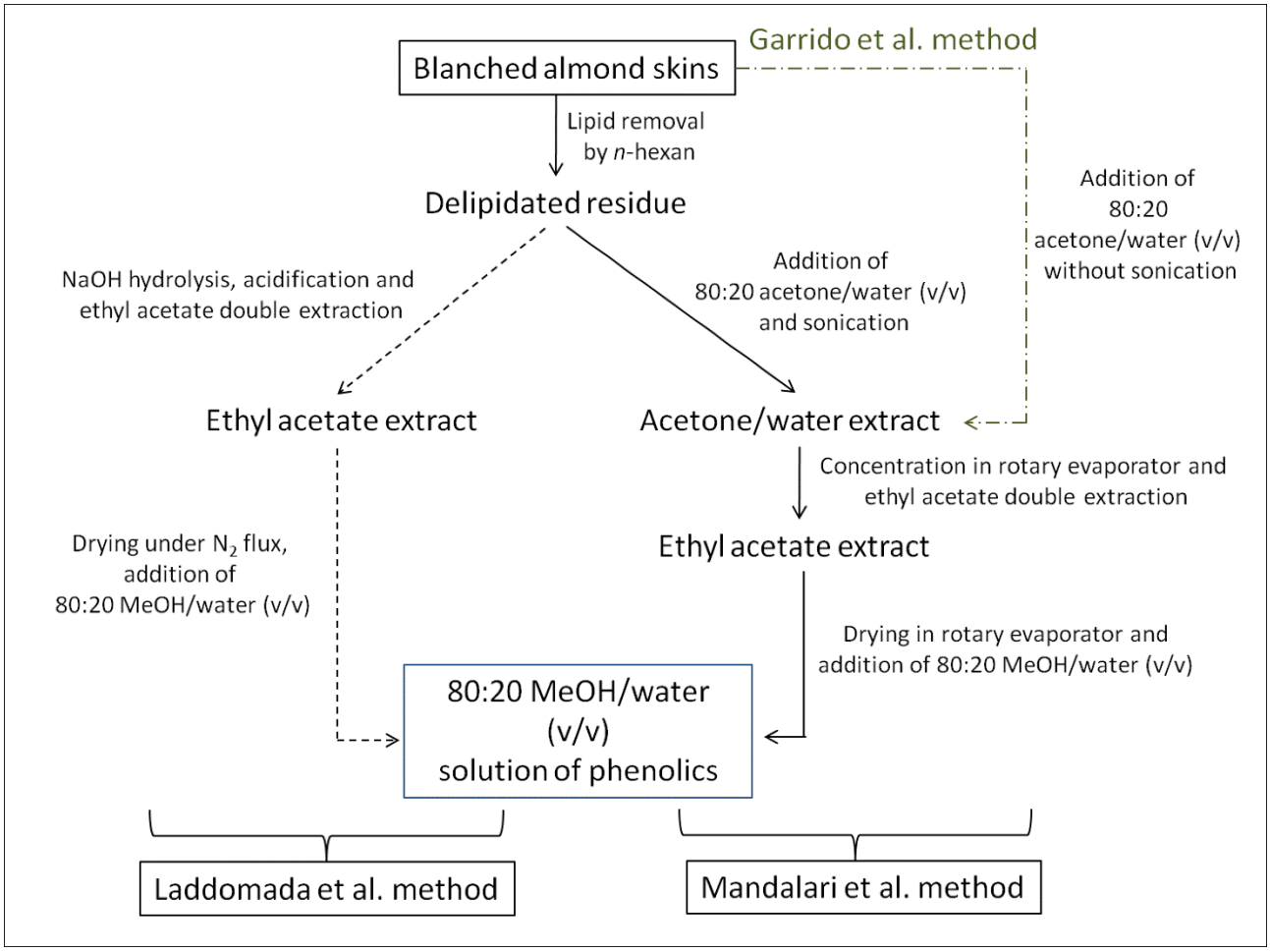

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Table 1
Main physico-chemical properties and odor notes of blanched almond skins (not dried and dried in different conditions) and blanching water.

	Blanched almond skins					Blanching water
		Dried				
	Not dried	60 °C for 30 min	45 °C for 90 min	32 °C for 150 min	38-40 °C for 6-8 h	
a_w	0.98 ± 0.01 [a]	0.41 ± 0.01 [e]	0.44 ± 0.01 [d]	0.54 ± 0.01 [b]	0.48 ± 0.01 [c]	n.d.
Moisture content (g/100 g)	58.49 ± 0.47 [a]	7.86 ± 0.06 [e]	8.55 ± 0.02 [c]	9.59 ± 0.06 [b]	8.09 ± 0.04 [d]	99.26 ± 1.13
Lipid content (g/100 g d.m.)	21.90 ± 0.20	22.00 ± 0.20	22.10 ± 0.40	21.60 ± 0.30	21.90 ± 0.30	n.d.
Antioxidant activity (µmol Trolox/g)	40.37 ± 0.17 [e]	81.56 ± 0.46 [b]	90.44 ± 0.95 [a]	72.25 ± 0.66 [c]	59.23 ± 1.50 [d]	17.43 ± 0.07 *
Color						
Brown index (100 – *L**)	43.71 ± 0.09 [e]	52.26 ± 0.34 [b]	50.70 ± 0.06 [c]	49.36 ± 0.03 [d]	58.66 ± 0.11 [a]	63.78 ± 0.21
Red index (*a**)	10.02 ± 0.09 [d]	11.20 ± 0.42 [abc]	11.36 ± 0.03 [b]	10.87 ± 0.02 [c]	11.71 ± 0.11 [a]	14.23 ± 0.87
Yellow index (*b**)	27.94 ± 0.09 [a]	25.56 ± 0.04 [b]	25.58 ± 0.10 [b]	25.14 ± 0.06 [c]	18.22 ± 0.33 [d]	20.13 ± 0.26
Odor notes						
Leafy odor	4.5 ± 1.1 [b]	6.1 ± 0.5 [a]	5.8 ± 0.2 [ab]	6.4 ± 0.2 [a]	1.7 ± 0.5 [c]	3.3 ± 0.5
Rancid odor	0.1 ± 0.1 [b]	0.2 ± 0.1 [b]	0.3 ± 0.1 [b]	0.4 ± 0.2 [b]	6.3 ± 0.2 [a]	0.1 ± 0.2
Sour odor	4.9 ± 1.0 [a]	0.2 ± 0.1 [b]	0.1 ± 0.1 [b]	0.1 ± 0.1 [b]	0.1 ± 0.0 [b]	0.5 ± 0.3

[a-e] Values followed by the same letter, within each row, are not significantly different at $p < 0.05$ except for the odor notes, where $p < 0.001$ (blanching water was not included in statistical comparison being a totally different by-product); n.d. = not determined; * expressed as µmol Trolox/mL.

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Table 2
Content of identified phenolic compounds (μg/g d.m.) of freshly blanched almond skins (not dried), determined according to three different protocols of extraction.

	Protocol of extraction		
	Garrido et al. (2008)	Mandalari et al. (2010)	Laddomada et al. (2017)
Phenolic acids			
Protocatechuic acid	3.20 ± 0.38 ^{c}	47.18 ± 4.08 ^{b}	92.35 ± 2.88 ^{a}
p-Hydroxybenzoic acid	4.33 ± 0.43 ^{b}	3.63 ± 0.53 ^{b}	32.80 ± 0.08 ^{a}
p-Coumaric acid	n.d.	n.d.	n.d.
Chlorogenic acid	n.d.	n.d.	n.d.
Vanillic acid	10.85 ± 0.80	n.d.	19.88 ± 0.10 ^{a}
Flavan-3-ols			
(+)-Catechin	126.10 ± 5.68 ^{c}	1085.15 ± 27.88 ^{b}	1392.59 ± 24.58 ^{a}
(-)-Epicatechin	724.18 ± 24.40 ^{a}	20.63 ± 1.95 ^{c}	200.65 ± 8.03 ^{b}
Flavonol glycosides and aglycones			
Quercetin-3-*O*-glucopyranoside	n.d.	n.d.	n.d.
Kaempferol-3-*O*-rutinoside	69.13 ± 1.63 ^{a}	31.58 ± 3.58 ^{b}	n.d.
Kaempferol-3-*O*-glucoside	n.d.	4.40 ± 0.75	n.d.
Isorhamnetin-3-*O*-rutinoside	110.50 ± 5.63 ^{a}	48.28 ± 2.45 ^{b}	n.d.
Isorhamnetin-3-*O*-glucoside	n.d.	n.d.	n.d.
Quercetin dehydrate	n.d.	n.d.	n.d.
Kaempferol	9.06 ± 3.21	n.d.	14.88 ± 1.35
Isorhamnetin	38.56 ± 2.93 ^{a}	25.33 ± 1.78 ^{b}	9.75 ± 0.28 ^{c}
Flavanone glycosides and aglycones			
Naringenin-7-*O*-glucoside	14.23 ± 1.28 ^{ab}	17.45 ± 0.80 ^{a}	10.48 ± 1.95 ^{b}
Eriodictyol	n.d.	n.d.	n.d.
Naringenin	n.d.	2.98 ± 0.23	n.d.
Total	1110.12 ± 51.24 ^{c}	1286.58 ± 79.12 ^{b}	1773.35 ± 82.36 ^{a}

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[a-b] Values followed by the same letter, within each row, are not significantly different ($p < 0.05$); n.d. = not detected.

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Table 3

Content of identified phenolic compounds (μg/g d.m.) of blanched almond skins dried in different thermal conditions. Drying treatments at 60 °C for 30 min, 45 °C for 90 min, and 32 °C for 150 min were carried out by a rotary air drier. Drying at 38-40 °C for 6-8 h was made in the sun. The extraction of phenolics was carried out according to Garrido et al. (2008) modified.

	Drying treatment			
	60 °C for 30 min	45 °C for 90 min	32 °C for 150 min	38-40 °C for 6-8 h
Phenolic acids				
Protocatechuic acid	2.89 ± 0.31 ^{c}	4.01 ± 0.32 ^{b}	4.44 ± 0.81 ^{b}	9.63 ± 2.11 ^{a}
p-Hydroxybenzoic acid	3.92 ± 0.98 ^{a}	3.53 ± 0.77 ^{a}	3.81 ± 0.71 ^{a}	1.47 ± 0.30 ^{b}
p-Coumaric acid	n.d.	n.d.	n.d.	n.d.
Chlorogenic acid	n.d.	n.d.	n.d.	n.d.
Vanillic acid	3.04 ± 0.03 ^{b}	3.09 ± 0.40 ^{b}	6.40 ± 0.28 ^{a}	1.47 ± 0.06 ^{c}
Flavan-3-ols				
(+)-Catechin	29.96 ± 1.01 ^{a}	18.26 ± 0.98 ^{c}	15.61 ± 0.56 ^{d}	21.15 ± 1.07 ^{b}
(-)-Epicatechin	233.95 ± 10.97 ^{b}	294.50 ± 11.25 ^{a}	279.77 ± 13.19 ^{a}	160.76 ± 9.07 ^{c}
Flavonol glycosides and aglycones				
Quercetin-3-*O*-glucopyranoside	n.d.	n.d.	n.d.	n.d.
Kaempferol-3-*O*-rutinoside	10.31 ± 0.14 ^{d}	11.48 ± 0.21 ^{c}	12.50 ± 0.17 ^{b}	13.47 ± 0.52 ^{a}
Kaempferol-3-*O*-glucoside	19.62 ± 0.78 ^{a}	17.81 ± 0.65 ^{a}	5.11 ± 0.34 ^{b}	0.67 ± 0.10 ^{c}
Isorhamnetin-3-*O*-rutinoside	26.53 ± 1.17 ^{b}	20.39 ± 1.58 ^{c}	30.74 ± 1.21 ^{a}	24.02 ± 1.09 ^{b}
Isorhamnetin-3-*O*-glucoside	n.d.	n.d.	n.d.	n.d.
Quercetin dehydrate	0.98 ± 0.07 ^{b}	0.82 ± 0.03 ^{c}	0.69 ± 0.02 ^{d}	1.70 ± 0.07 ^{a}
Kaempferol	0.86 ± 0.03 ^{b}	0.83 ± 0.02 ^{bc}	0.78 ± 0.02 ^{c}	1.52 ± 0.05 ^{a}
Isorhamnetin	7.77 ± 0.19 ^{a}	6.86 ± 0.13 ^{bc}	6.66 ± 0.16 ^{c}	7.09 ± 0.11 ^{b}
Flavanone glycosides and aglycones				
Naringenin-7-*O*-glucoside	12.87 ± 0.21 ^{c}	14.14 ± 0.19 ^{b}	20.81 ± 0.31 ^{a}	10.64 ± 0.14 ^{d}
Eriodictyol	n.d.	n.d.	n.d.	n.d.
Naringenin	n.d.	n.d.	n.d.	n.d.
Total	352.70 ± 18.92 ^{a}	395.72 ± 23.46 ^{a}	387.32 ± 21.24 ^{a}	253.59 ± 11.26 ^{b}

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Table 4

Content of identified phenolic compounds (μg/g d.m.) of blanched almond skins dried in different thermal conditions. Drying treatments at 60 °C for 30 min, 45 °C for 90 min, and 32 °C for 150 min were carried out by a rotary air drier. Drying at 38-40 °C for 6-8 h was made in the sun. The extraction of phenolics was carried out according to Mandalari et al. (2010).

	Drying treatment			
	60 °C for 30 min	45 °C for 90 min	32 °C for 150 min	38-40 °C for 6-8 h
Phenolic acids				
Protocatechuic acid	4.14 ± 0.89 [a]	3.84 ± 0.78 [a]	5.14 ± 0.92 [a]	3.09 ± 0.04 [b]
p-Hydroxybenzoic acid	14.43 ± 0.22 [b]	10.43 ± 0.25 [c]	15.31 ± 0.12 [a]	14.03 ± 0.43 [b]
p-Coumaric acid	n.d.	n.d.	n.d.	n.d.
Chlorogenic acid	n.d.	n.d.	n.d.	n.d.
Vanillic acid	n.d.	n.d.	n.d.	n.d.
Flavan-3-ols				
(+)-Catechin	572.23 ± 12.21 [b]	621.23 ± 13.67 [a]	410.72 ± 10.25 [c]	110.83 ± 12.36 [d]
(-)-Epicatechin	51.62 ± 2.74 [a]	46.29 ± 2.25 [ab]	45.52 ± 2.17 [b]	29.36 ± 1.51 [c]
Flavonol glycosides and aglycones				
Quercetin-3-*O*-glucopyranoside	n.d.	n.d.	n.d.	n.d.
Kaempferol-3-*O*-rutinoside	48.31 ± 2.65	47.88 ± 2.35	49.21 ± 3.01	49.76 ± 4.41
Kaempferol-3-*O*-glucoside	3.31 ± 0.11 [c]	4.31 ± 0.10 [a]	3.55 ± 0.03 [b]	n.d.
Isorhamnetin-3-*O*-rutinoside	93.19 ± 2.09 [b]	94.45 ± 3.11 [b]	105.32 ± 2.98 [a]	92.54 ± 2.75 [b]
Isorhamnetin-3-*O*-glucoside	n.d.	n.d.	n.d.	n.d.
Quercetin dehydrate	n.d.	n.d.	n.d.	n.d.
Kaempferol	0.31 ± 1.98 [b]	0.88 ± 1.65 [a]	0.30 ± 0.01 [a]	0.30 ± 0.02 [a]
Isorhamnetin	12.23 ± 0.76 [a]	12.25 ± 1.17 [a]	12.92 ± 0.45 [a]	9.59 ± 0.37 [b]
Flavanone glycosides and aglycones				
Naringenin-7-*O*-glucoside	12.36 ± 0.23 [c]	13.54 ± 0.34 [b]	16.08 ± 0.45 [a]	2.62 ± 0.03 [d]
Eriodictyol	n.d.	n.d.	n.d.	n.d.
Naringenin	1.76 ± 0.08 [c]	2.01 ± 0.04 [b]	2.03 ± 0.03 [b]	2.94 ± 0.07 [a]
Total	813.89 ± 31.56 [a]	857.11 ± 38.91 [a]	666.10 ± 18.37 [b]	317.06 ± 12.34 [c]

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[a-c] Values followed by the same letter, within each row, are not significantly different ($p < 0.05$); n.d. = not detected.

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Table 5

Content of identified phenolic compounds (μg/g d.m.) of blanched almond skins dried in different thermal conditions. Drying treatments at 60 °C for 30 min, 45 °C for 90 min, and 32 °C for 150 min were carried out by a rotary air drier. Drying at 38-40 °C for 6-8 h was made in the sun. The extraction of phenolics was carried out according to Laddomada et al. (2017).

	Drying treatment			
	60 °C for 30 min	45 °C for 90 min	32 °C for 150 min	38-40 °C for 6-8 h
Phenolic acids				
Protocatechuic acid	162.33 ± 8.29 ^{b}	163.28 ± 11.65 ^{ab}	150.24 ± 11.24 ^{b}	200.11 ± 15.15 ^{a}
p-Hydroxybenzoic acid	65.98 ± 4.32 ^{a}	61.07 ± 3.86 ^{ab}	55.84 ± 3.68 ^{b}	57.15 ± 4.09 ^{ab}
p-Coumaric acid	7.27 ± 0.87 ^{ab}	8.10 ± 0.71 ^{a}	5.80 ± 0.66 ^{b}	7.78 ± 0.89 ^{a}
Chlorogenic acid	n.d.	n.d.	n.d.	n.d.
Vanillic acid	27.85 ± 2.04 ^{a}	30.67 ± 2.25 ^{a}	26.76 ± 2.41 ^{a}	14.97 ± 2.01 ^{b}
Flavan-3-ols				
(+)-Catechin	116.97 ± 9.97 ^{c}	178.10 ± 11.94 ^{a}	127.65 ± 7.97 ^{bc}	136.12 ± 7.89 ^{b}
(-)-Epicatechin	84.22 ± 3.60 ^{b}	120.64 ± 4.02 ^{a}	117.61 ± 4.54 ^{a}	50.53 ± 9.21 ^{c}
Flavonol glycosides and aglycones				
Quercetin-3-*O*-glucopyranoside	n.d.	n.d.	n.d.	n.d.
Kaempferol-3-*O*-rutinoside	23.88 ± 2.83 ^{a}	10.10 ± 1.12 ^{b}	7.87 ± 0.99 ^{b}	8.67 ± 1.12 ^{b}
Kaempferol-3-*O*-glucoside	30.18 ± 1.51 ^{a}	24.01 ± 1.40 ^{b}	17.51 ± 0.97 ^{c}	33.32 ± 1.15 ^{a}
Isorhamnetin-3-*O*-rutinoside	67.08 ± 5.32 ^{a}	45.24 ± 2.62 ^{bc}	47.60 ± 2.87 ^{b}	30.91 ± 1.98 ^{c}
Isorhamnetin-3-*O*-glucoside	n.d.	n.d.	n.d.	n.d.
Quercetin dehydrate	2.79 ± 0.21 ^{a}	1.57 ± 0.15 ^{b}	1.90 ± 0.80 ^{a}	1.59 ± 0.13 ^{b}
Kaempferol	7.53 ± 0.57 ^{a}	5.95 ± 0.65 ^{b}	6.11 ± 0.87 ^{ab}	7.23 ± 0.73 ^{ab}
Isorhamnetin	36.00 ± 3.10 ^{a}	23.11 ± 2.19 ^{b}	24.25 ± 1.22 ^{b}	26.23 ± 1.07 ^{b}
Flavanone glycosides and aglycones				
Naringenin-7-*O*-glucoside	15.71 ± 1.13 ^{a}	7.56 ± 1.22 ^{b}	5.93 ± 1.14 ^{b}	2.79 ± 0.21 ^{c}
Eriodictyol	n.d.	n.d.	n.d.	n.d.
Naringenin	n.d.	n.d.	n.d.	n.d.
Total	647.79 ± 19.02 ^{a}	679.40 ± 20.97 ^{a}	595.07 ± 14.46 ^{b}	577.40 ± 23.12 ^{c}

Preprint of the article: Pasqualone, A., Laddomada, B., Spina, A., Todaro, A., Guzmàn, C., Summo, C., Mita, G. and Giannone, V., 2018. Almond by-products: Extraction and characterization of phenolic compounds and evaluation of their potential use in composite dough with wheat flour. *LWT*, *89*, pp.299-306. https://doi.org/10.1016/j.lwt.2017.10.066

[a-c] Values followed by the same letter, within each row, are not significantly different ($p < 0.05$); n.d. = not detected.

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Table 6

Content of identified phenolic compounds (μg/mL) of almond blanching water (dry residue = 7.4 mg/mL) extracted according to Mandalari et al. (2010) with slight modifications. The extraction of phenolics was carried out with and without sonication.

	With sonication	Without sonication
Phenolic acids		
Protocatechuic acid	5.68 ± 0.81	5.89 ± 0.78
p-Hydroxybenzoic acid	2.94 ± 0.23 [b]	4.91 ± 0.54 [a]
p-Coumaric acid	n.d.	n.d.
Chlorogenic acid	n.d.	n.d.
Vanillic acid	n.d.	n.d.
Flavan-3-ols		
(+)-Catechin	847.24 ± 11.21 [a]	408.31 ± 7.34 [b]
(-)-Epicatechin	30.90 ± 1.27 [b]	63.92 ± 2.01 [a]
Flavonol glycosides and aglycones		
Quercetin-3-*O*-glucopyranoside	n.d.	n.d.
Kaempferol-3-*O*-rutinoside	11.51 ± 1.25 [a]	8.07 ± 1.31 [b]
Kaempferol-3-*O*-glucoside	n.d.	1.46 ± 0.05
Isorhamnetin-3-*O*-rutinoside	18.56 ± 1.98 [a]	13.35 ± 1.45 [b]
Isorhamnetin-3-*O*-glucoside	n.d.	3.64 ± 0.09
Quercetin dehydrate	n.d.	n.d.
Kaempferol	n.d.	n.d.
Isorhamnetin	0.04 ± 0.01 [b]	0.27 ± 0.03 [a]
Flavanone glycosides and aglycones		
Naringenin-7-*O*-glucoside	0.49 ± 0.03 [a]	0.31 ± 0.02 [b]
Eriodictyol	n.d.	n.d.
Naringenin	n.d.	0.12 ± 0.01
Total	917.46 ± 41.12 [a]	510.25 ± 23.46 [b]

[a-b] Values followed by the same letter, within each row, are not significantly different ($p < 0.05$); n.d. = not detected.

Table 7

Results of farinograph and alveograph analyses carried out on composite meals containing refined wheat flour and increasing amounts of blanched almond skins (dried at 60 °C for 30 min and milled) or blanching water.

Parameter	Almond skins (g 100 g^{-1} wheat flour)					Blanching water*
	0 (CTRL)	3	5	7	10	
Farinograph data						
Water absorption (g/100 g)	57.5 ± 0.4 [d]	59.2 ± 0.4 [c]	60.2 ± 0.5 [bc]	61.2 ± 0.1 [a]	61.2 ± 1.5 [ab]	56.4 ± 0.5 [d]

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Dough development time (s)	93 ± 4	93 ± 4	87 ± 12	87 ± 4	90 ± 5	94 ± 4
Dough stability (s)	144 ± 8 ^{c}	126 ± 9 ^{c}	138 ± 8 ^{c}	264 ± 16 ^{b}	630 ± 51 ^{a}	140 ± 7 ^{c}
Softening index (B.U.)	60 ± 5 ^{a}	54 ± 5 ^{ab}	45 ± 9 ^{ab}	43 ± 6 ^{b}	27 ± 2 ^{c}	58 ± 4 ^{a}
Alveograph data						
Deformation energy × 10^{-4} (J)	175.7 ± 2.9 ^{a}	170.0 ± 1.7 ^{b}	163.0 ± 5.2 ^{b}	133.0 ± 1.7 ^{c}	117.3 ± 8.1 ^{d}	173.4 ± 3.1 ^{a}
Tenacity/Extensibility ratio	0.8 ± 0.1 ^{e}	1.6 ± 0.3 ^{d}	2.2 ± 0.2 ^{c}	4.8 ± 0.4 ^{b}	6.0 ± 0.2 ^{a}	0.8 ± 0.2 ^{e}

* The amount of blanching water accounted for 56.4 mL per 100 g wheat flour for farinograph and 53.2 mL for alveograph, according to the corresponding analytical protocols; $^{a\text{-}e}$ Values followed by the same letter, within each row, are not significantly different ($p < 0.05$). B.U. = Brabender Units.