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Physico-chemical properties and sensory profile of durum wheat Dittaino PDO (Protected Designation of Origin) bread and quality of re-milled semolina used for its production

Virgilio Giannone [a], Mariagrazia Giarnetti [b], Alfio Spina [c], Aldo Todaro [a], Biagio Pecorino [d], Carmine Summo [b], Francesco Caponio [b], Vito Michele Paradiso [b], Antonella Pasqualone [b,*]

[a] Department of Agricultural and Forest Sciences, University di Palermo - Viale delle Scienze Ed.4, 90128 Palermo, Italy

[b] Department of Soil, Plant and Food Sciences, University of Bari Aldo Moro, Via Amendola, 165/A, 70126 Bari, Italy

[c] Consiglio per la ricerca in agricoltura e l'analisi dell'economia agraria (CREA), Centro di Ricerca per la Cerealicoltura e le Colture Industriali, Corso Savoia 190, 95024 Acireale, Italy

[d] Department of Agriculture, Food and Environment (Di3A), University of Catania, Via Santa Sofia 100, 95123 Catania, Italy

*Corresponding author. E-mail address: antonella.pasqualone@uniba.it (A. Pasqualone)

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Abstract

To help future quality checks, we characterized the physico-chemical and sensory properties of Dittaino bread, a sourdough-based durum wheat bread recently awarded with Protected Designation of Origin mark, along with the quality features of re-milled semolina used for its production. Semolina was checked for Falling Number (533-644 s), protein content (12.0-12.3 g/100 g d.m.), gluten content (9.2-10.5 g/100 g d.m.), yellow index (18.0-21.0), water absorption (59.3-62.3 g/100 g), farinograph dough stability (171-327 s), softening index (46-66 B.U.), alveograph W (193×10^{-4}-223×10^{-4} J) and P/L (2.2-2.7). Accordingly, bread crumb was yellow, moderately hard (16.4-27.1 N) and chewy (88.2-109.2 N×mm), with low specific volume (2.28-3.03 mL/g). Bread aroma profile showed ethanol and acetic acid, followed by hexanol, 3-methyl-1-butanol, 2-phenylethanol, 3-methylbutanal, hexanal, benzaldehyde, and furfural. The sensory features were dominated by a thick brown crust, with marked toasted odor, coupled to yellow and consistent crumb, with coarse grain and well-perceivable sour taste and odor.

Keywords: durum wheat bread, re-milled semolina, quality control, sensory profile, texture, volatile compounds

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1. Introduction

In many areas of the world, while taking into account the developments of new production methods and materials, farmers and food producers have tried to keep traditions alive, in terms of local artisanal processing methods. The cultural and gastronomic heritages are important factors contributing to the diversity of agricultural food productions and, besides the social aspects, a certain economic impact has been established. In fact, during the last decades, consumers have shown an increasing appreciation of traditional and typical foods, thus inducing the European Union to regulate this subject.

According to the EU Regulation no. 1151/2012, "traditional" is the claim used for foods that historically – i.e. for a period of at least 30 years, that allows transmission between generations – are part of the cultural heritage of people living in a specific geographical area (European Parliament and European Council, 2012). "Typical" is the attribute of food whose quality features strictly depend on the geographical area of production, due to the combined effect of soil and water physico-chemical characteristics, climate, microflora, and local processing techniques (D'Amico, 2004). In particular, the "protected designation of origin" (PDO) identifies a product originated and totally produced in a specific geographical area, whereas to obtain the "protected geographical indication" (PGI) mark, less stringent than PDO, is sufficient that at least one of the production steps take place in the defined geographical area (European Parliament and European Council, 2012).

At European level, few breads have been awarded by PDO recognition: the Italian breads "Pane di Altamura" (Altamura bread), "Pagnotta del Dittaino" (Dittaino bread), and "Pane Toscano" (Tuscany bread), and the Swedish bread "Upplandskubb" (European Commission, 2016a), registered by the European Regulations nos. 1291/2003, 516/2009, 303/2016, and 843/2014, respectively (European Commission, 2003; 2009; 2014a; 2016b). Among them, Altamura PDO bread and Dittaino PDO bread, although being produced using different cultivars and in different areas, are both obtained from durum wheat re-milled semolina, according to a bread-making tradition consolidated in Southern Italy (Pasqualone, 2012). Altamura PDO bread has been extensively studied (Bianchi, Careri, Chiavaro, Musci, & Vittadini, 2008; Brescia et al., 2007; Chiavaro, Vittadini, Musci, Bianchi, & Curti, 2008; Raffo et al., 2003; Pasqualone, Summo, Bilancia, & Caponio, 2007; Pasqualone, Alba, Mangini, Blanco, & Montemurro, 2010). On the contrary, no research has been aimed until now to the quality characterization of Dittaino PDO bread, apart the inclusion of its sourdough in an array of samples for a survey on microbiotas used for traditional/typical Italian breads (Minervini et al., 2012).

Starting from durum wheat cultivation, all processing steps of Dittaino PDO bread take place within the area closely surrounding the Sicilian town of Enna (Italy), along the Dittaino river. Bread production follows a very simple and genuine recipe exclusively based on re-milled semolina, water, sourdough, and sea salt, without the addition of sugar, malt or malt extract, fats, anti-staling ingredients or any other additive. More specifically, durum wheat cultivars Simeto, Duilio, Arcangelo, Mongibello, Ciccio, Colosseo, Bronte, Iride, and Sant'Agata, grown in the Dittaino area, have to be used, alone or in combination, for at least 70% of the total semolina. The fermentation of dough is based on the dynamic equilibrium between yeasts and lactic bacteria of traditional sourdoughs (Type I) (De Vuyst & Neysens, 2005), with *Lactobacillus sanfranciscensis* (*Lactobacillus brevis* ssp. *lindneri*), *Candida milleri* and *Saccharomyces exiguus* as principal microbial species (Minervini et al., 2012; European Commission, 2014b). Dittaino PDO bread is finally baked at 230 °C for 60 min, traditionally as a round loaf of hearth bread weighing between 500 g and 1.100 g, characterized by a well-developed dark brown, highly consistent crust, and by pale yellow, uniformly porous crumb.

The official technical sheet of Dittaino PDO bread reports the main physico-chemical

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characteristics of starting durum wheat, semolina and bread (European Commission, 2014b). In particular, re-milled semolina must have protein content ≥ 10.5% (d.m.), ashes = 0.70-0.90% (d.m.) and Falling number = 480-800 s. Bread has to show 3-4 mm thick crust and moisture content ≤ 38%. However, a more detailed quality characterization of the end-product and its raw material could improve technical awareness by producers, overcoming empirical knowledge, and could even enhance quality and consumer appreciation. In this framework, the aim of this research was to characterize the physico-chemical properties and sensory profile of Dittaino PDO bread, along with the quality features of re-milled semolina used for its production.

2. Materials and methods

2.1. Sample collection

Samples of durum wheat Dittaino PDO bread, along with the starting re-milled semolina certified for PDO bread production, were collected in five samplings (coded A-E) that were carried out, within the period of two months, in local bakeries of the Dittaino area (Enna, Sicily, Italy). At each sampling, three bread loaves and two re-milled semolina samples were collected. Breads were produced according to the official procedure of Dittaino PDO bread (European Commission, 2014b), that requires the use of natural sourdough (Type I) (De Vuyst & Neysens, 2005) derived from a daily renewed starter. The renewal procedure involves mixing sourdough starter, re-milled semolina, and water at 1:4:2 ratio. The final dough contained durum wheat re-milled semolina (100 kg), water (62.5 L), renewed sourdough (18 kg), and NaCl (2 kg). After 10-12 min mixing by means of diving arm mixers, the dough was rested in bulk for 45 min, then was scaled into portions weighting about 1100 g (to take into account the weight loss due to water evaporation during baking). The portions were then shaped as round loaves and proofed for 1.5-2 h at 25-30 °C. Baking was carried out at 230 °C for 60 min in gas fueled ovens.

2.2. Physico-chemical analyses of re-milled semolina

Protein content was determined by means of Infratec 1241 Grain Analyzer 148 (Foss Tecator, Höganäs, Sweden), based on Near Infrared Transmittance. A calibration curve (range 8.3%-15.3%) was previously set up on the results of Kjeldahl nitrogen method and validated according to ISO 151 12099:2010 method (ISO, 2010) on a large set of samples.

Ash and moisture content were determined according to the AACC 44–19 and AACC 08–01 methods (AACC, 2000), respectively.

Dry gluten was determined by using a Glutomatic System consisting of Glutomatic 2200, Centrifuge 2015, Glutork 2020 (Perten Instruments AB, Huddinge, Sweden), according to the UNI 10690 method (UNI, 1979).

The α-amylase activity was determined by using the Falling Number 1500 apparatus (Perten Instruments AB, Huddinge, Sweden), following the ISO 3093:2009 method (ISO, 2009).

The color parameters in the color space L^*, a^*, b^* were determined by Chromameter CR-300 (Minolta, Osaka, Japan), under the illuminant D65. Brown index was calculated as $100 - L^*$.

The farinograph indices were determined according to the AACC 54-21 method (AACC, 2000) by a farinograph (Brabender instrument, Duisburg, Germany), equipped with the software Farinograph®161. Water absorption needed to achieve the dough consistency of 500 ± 20 Brabender Units (B.U.) (A), dough development time (B), dough stability (CD), and consistency drop off after 12 min (E12) were measured.

Alveograph trials were performed according to the AACC method 54-30A using an alveoconsistograph, equipped with the software Alveolink NG (Tripette et Renaud, 163 Villeneuve-la-Garenne, France).

All the analyses were carried out in triplicate.

2.3. Physico-chemical analyses of bread

Moisture content of bread crumb was determined by oven drying at 105 °C until constant weight. Water activity (a_w) was determined by Hygropalm 40 AW (Rotronic Instruments Ltd, Crawley, UK) according to manufacturers' instructions. For these determinations, three bread slices (11±1 mm thickness) were used, and one square crumb sample (40 mm × 40 mm) was taken from the center of each slice.

The Texture Profile Analysis (TPA) of bread was carried out by means of an Universal Testing machine (model 3344, Instron, Norwood, MA, USA), equipped with 5.0 cm diameter cylindrical probe, 2000 N load cell, and Bluehill® 2 software (Instron, Norwood, MA, USA), in the conditions reported in Giannone et al. (2016).

Specific volume was determined by rapeseed displacement, as in AACC method 10-10 (AACC, 2000).

Color parameters of crumb and crust in the color space L^*, a^*, b^* were determined by Chromameter CR-300 (Minolta, Osaka, Japan), under the illuminant D65. Brown index was calculated as 100 – L^*.

Total carotenoid pigments were determined according to AACC approved method 14–50.01 (AACC, 2000) with slight modifications: bread crumb was lyophilized and ground in a mortar, then 1 g of each sample was extracted with 5 mL of water-saturated *n*-butyl alcohol on an orbital shaker for 3 h at 260 rpm. Samples were centrifuged for 7 min at 2400 × *g*, and the absorbance of water-saturated *n*-butyl alcohol extracts was measured at 435.8 nm by a Cary 60 UV-Vis spectrophotometer (Agilent Technologies Inc., Santa Clara, CA, USA). Total carotenoid content was expressed as β-carotene, and calculations were made based on the extinction coefficient of 1.6632 for a solution of 1 mg β-carotene in 100 mL water-saturated *n*-butyl alcohol.

All the analyses were carried out in triplicate.

2.4. Sensory analysis of bread

Quantitative Descriptive Analysis (QDA) of bread samples was performed by a sensory panel consisting of 8 members in the conditions described in a previous work (Pasqualone et al., 2007). The list of sensory terms included descriptors of appearance (crust color, crust thickness, crumb color, crumb grain), visual-tactile and chewing characteristics (crumb cohesiveness, crumb consistency), odor (semolina, sour, toast), and taste (sweet, salty, sour, bitter). The descriptors were rated on an anchored line scale that provided a 0-9 score range (0 = minimum; 9 = maximum intensity). The definition of each descriptor and the scale anchors are reported in Table 1.

2.5. Determination of volatile compounds of bread

Volatile compounds of bread samples were determined by solid phase micro-extraction (SPME) coupled to gas-chromatography/mass spectrometry (GC/MS). Bread crust and crumb were cut into pieces of 2-3 mm and mixed in the crumb to crust ratio of 3:1, preliminarily determined in entire bread samples. Amounts of crumb and crust mixture of 400 ± 0.05 mg were then weighed in a 20-mL vial, and added of 4 mL of a 20% aqueous solution of NaCl (w/v). Vials were sealed by butyl rubber septa and aluminum crimp caps. Before volatile extraction, the sample was homogenized for 2 min using a laboratory vortex shaker. The extraction of volatile compounds was carried out by exposing a 75 μm carboxen/polydimethylsiloxane (CAR/PDMS) SPME fiber (Supelco, Bellefonte, PA, USA) in the headspace of the sample at 50 °C for 40 min. The fiber was then desorbed for 2 min in the injection port of the gas-chromatograph, operating in split-less mode. An Agilent 6850

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gas-chromatograph equipped with an Agilent 5975 mass-spectrometer (Agilent Technologies Inc., Santa Clara, CA, USA) was used. The volatile compounds were separated on a HP-Innowax (Agilent Technologies Inc., Santa Clara, CA, USA) polar capillary column (60 m length × 0.25 mm i.d. × 0.25 μm film thickness), under the following conditions: injector temperature, 300 °C; flow of 2.0 mL/min. The oven temperature was held for 5 min at 35 °C, then increased by 5 °C/min to 50 °C and held in isothermal conditions for 5 min, then raised to 230 °C at 5.5 °C/min, and finally held constant at 230 °C for 5 min. The mass detector was set at the following conditions: interface temperature 230 °C; source temperature 230 °C; ionization energy 70 eV; scan range 33-260 amu. Peak identification was performed by computer matching with the reference mass spectra of National Institute of Standards and Technology (NIST) and Wiley libraries. Semi-quantitative data (peak areas expressed as total ion counts - TIC) were considered. The analysis was carried out in triplicate.

2.6. Statistical analyses

The statistical analyses were performed by using the CoStat Anova Statistic Software version 6.311 (Cohort, Monterey, CA, USA) for Windows. Data were submitted to analysis of variance (ANOVA) using Duncan's multiple range test.

3. Results and discussion

3.1. Re-milled semolina characteristics

Several parameters are able to assess the bread-making quality of re-milled semolina. Fermentative aptitude (Falling Number), protein and gluten content, gluten strength (alveograph W), extensibility/tenacity balance (alveograph P/L ratio), water absorption capacity, mixing and kneading behavior (farinograph stability, dough development time, and softening index), are all useful parameters to predict the possibility to obtain voluminous breads with good yield. Therefore, re-milled semolina used in the production of Dittaino PDO bread was checked for the above mentioned parameters, as well as for color indices (Table 2).

The observed values of fermentative aptitude, expressed by Falling Number, although with significant differences among samplings, were always within the range 480-800 s required by the official sheet of Dittaino PDO bread (European Commission, 2014b). This range corresponds to a very limited starting amylase activity, more suitable for pasta than for bread-making. However, such limited values are needed because the drop of pH consequent to sourdough use increases the activity of enzymes, such as proteases and amylases (Arendt, Ryan, & Dal Bello, 2007). The lowest fermentative aptitude was found in sample set E.

Protein content was always above 10.5 g/100 g, as required by the official sheet (European Commission, 2014b). Similarly, ash content accomplished the basic legal requirements being in the range 0.70-0.90 g/100 g. Protein content has a positive effect on bread loaf volume (Goesaert et al., 2005). The observed protein levels were similar to those reported in a previous survey on the quality characteristics of durum wheat re-milled semolina from Southern Italy (11.0-12.9 g/100 g d.m.), the area where the majority of Italian durum wheat milling capability is concentrated (Pasqualone, Caponio, & Simeone, 2004). Gluten content was comprised between 9.2 g/100 g and 10.5 g/100 g, with slightly lower values than those observed in previous surveys (Pasqualone et al., 2004). Significant differences of gluten content were detected among the collected semolina samples, related to significant differences observed also in protein levels.

Well-balanced visco-elastic properties and strong gluten are essential to allow optimal bread development. In soft wheat, the alveograph P/L ratio should range from 0.4 to 0.8. A tenacious gluten, instead, is expected in durum wheat re-milled semolina, and alveograph P/L values higher than 1.0 are tolerated. In particular, values up to 2.5 were observed in commercial re-milled semolina in previous investigations (Pasqualone et al., 2004). In re-milled semolina destined to the production of Dittaino PDO bread were ascertained P/L values from 2.2 to 2.7, indicating that all the samples gave a highly tenacious dough, expected to increase in volume very slightly during leavening. P/L, in fact, is known to be negatively correlated with bread specific volume (Pasqualone et al., 2004). On the other hand, the alveograph index W was comprised between 193×10^{-4} J and 223×10^{-4} J, indicating the presence of sufficiently strong gluten, able to bear the prolonged leavening times required by sourdough-based bread-making.

Mixing behavior was evaluated by farinograph. Dough development time, i.e. time needed to reach the maximum torque, was in the range 93-114 s, evidencing the absence of obstacles in gluten formation. Prolonged dough stability to mixing, and limited drop of consistency (softening index), both essential for sourdough propagation, were observed in particular in sample set A, which showed better values than those reported for starting re-milled semolina used in the production of another sourdough-based traditional bread, namely Altamura PDO bread (Raffo et al., 2003). The capacity to absorb water, determined by farinograph, depends on the content of protein and of damaged starch (Raffo et al., 2003), as well as on gluten strength, and is known to be positively related to bread yield. Water absorption was high in all the samples, but with significant differences among them and with the highest value in sample set A.

Another quality trait of durum wheat re-milled semolina is the amber-yellow color, partly transferred to bread and due to carotenoid pigments. Yellowish crumb is highly appreciated by the consumers of durum wheat breads, therefore yellow index of starting re-milled semolina should be high. Rather low values of yellow index were observed in sample sets C, D and E, whereas a remarkably high value was found in sample set A. In any case, the negligible contribution of red index, coupled to values of brown index considerably lower than in whole-meal semolina

(Pasqualone et al., 2015), allowed to perceive a brilliant and luminous color in all the samples.

Overall, the quality characteristics of semolina samples were variable, but remained within the ranges required by the official sheet of Dittaino PDO bread (European Commission, 2014b). However it has to be pointed out that some parameters, such as the alveograph and farinograph indices, as well as the yellow index, although being strongly related to bread quality, are not included in the official list of pre-requisites for the production of this kind of bread. Their future inclusion would be very useful to enhance quality and keep it more constant. In this perspective, the "A" semolina could be assumed as a superior quality reference, having the highest values of yellow index, protein and gluten content, and optimal values of alveograph and farinograph indices.

3.2. Dittaino PDO bread characteristics

Crumb moisture and a_w (Table 3) were within the typical range for 1-kg hearth bread loaves (Pasqualone et al., 2007; Raffo et al., 2003; Licciardello et al., 2017), but with significantly higher values in samples A and B than in the others. According to the official procedure of Dittaino PDO bread (European Commission, 2014b) the amount of water added to semolina has to be 62.5 L, therefore the difference in crumb moisture and a_w was attributable to different water absorption capacity of semolina samples (with the highest value in sample set A) and to slight variations in the thermal effects of baking. It would be useful to specify in the official procedure that water should be added on the basis of farinograph-determined absorption, instead of indicating a fixed water amount.

Crumb color was nearly yellow and reflected semolina color, with sample set A showing the highest yellow index. Yellow color is typical, with varying intensity, of all durum wheat breads, such as "Pane di Altamura" (Pasqualone et al., 2007; Brescia et al., 2007), "Pane di Laterza", and "Pane di Matera" (Brescia et al., 2007). Carotenoid pigments were detected in levels between 2.30 and 3.65

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mg/kg, in the range observed in other durum wheat breads and definitely higher than in common wheat breads (Pasqualone et al., 2004). Crust color was dark brown, with some red reflexes, due to the prolonged baking process needed to allow heat reaching the inner part of the big-sized loaves. The sample sets A, B and C had a darker crust than the others.

The specific volume of Dittaino bread was rather low (2.28-3.03 mL/g), as expected, due to the combined effect of sourdough (Martínez-Anaya, Pitarch, Bayarri, & Benedito de Barber, 1990) and tenacious gluten. Bread sample set E showed the lowest value of specific volume, probably due to further negative effect of low fermentative aptitude of semolina. Textural data, obtained by means of double cycle compressions at 40% depth, evidenced moderately high values of hardness and chewiness, with significant differences among samplings. In particular, hardness ranged from 16.4 N to 27.1 N, and chewiness from 88.2 N × mm to 109.2 N × mm. The differences were in accordance with specific volume: bread sample sets A and C, that were the softest and less chewy, also had the highest specific volume. The observed values of resilience and springiness indicated a good ability of all bread samples to regain the original position after compression, again with significantly better values in sample sets A and C. Resilience and springiness are known to decrease with storage time, with a tendency of bread to become crumblier and to lose its cohesive structure.

The volatile compounds of Dittaino PDO bread included alcohols, aldehydes, ketones, carboxylic acids, furan compounds, pyrazines, and sulfur compounds (Table 4). In fact, bread aroma results from the complex combination of many volatile compounds derived from semolina and originated or modified during leavening and baking steps.

Ethanol and acetic acid, derived from fermentation reactions, were by far the most abundant volatiles. The latter, in particular, together with ethyl acetate, was typical of sourdough-based leavening (Rehman, Paterson, & Piggott, 2006). Among alcohols, also hexanol, 3-methyl-1-butanol (isoamyl alcohol) and 2-phenylethanol were quantitatively relevant. Ruiz, Quilez, Mestres, &

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Guasch (2003) observed the same alcohols in baguette and ciabatta crumb. Isoamyl alcohol was found in important amounts by Chang, Seitz, & Chambers IV (1995) in white pan and whole wheat breads. Short-chain alcohols and fatty acids derive from sugar fermentation, whereas higher molecular weight alcohols arise from aminoacid metabolism (Rehman et al., 2006). In particular, 2-phenylethanol derives from phenylalanine.

Carbonyl compounds such as 3-methylbutanal, hexanal, benzaldehyde and furfural, important components of bread volatiles (Chang et al., 1995; Ruiz et al., 2003), were found in high amounts in Dittaino PDO bread. Hexanal (as well as nonanal) takes its origin in lipid oxidation (Frankel, 1983). 3-Methylbutanal is a Strecker's aldehyde arising from leucine, and is responsible for a malty note (Pozo-Bayón, Guichard, & Cayot, 2006). Benzaldehyde can derive from metabolic or thermal degradation of phenylalanine (Pripis-Nicolau, De Revel, Bertrand, & Maujean, 2000).

Pyrazines and furan compounds raised from thermal reactions such as Maillard reaction and caramelization (Martínez-Anaya, 1996). These reactions are more intense at loaf surface, generating the crust and its typical odor notes. In particular, were detected: methylpyrazine (associated to popcorn odor), 2-ethyl-3-methylpyrazine (toasted, nutty, crust-like), 2,6-dimethylpyrazine (nutty), ethylpyrazine (musty, nutty), furfural (brown), and 2-furanmethanol (burnt) (Chang et al., 1995). Overall, pyrazines were more abundant in sample sets A, B and C than in D and E, indicating a more intense thermal effect during baking, that agreed with colorimetric data of crust. Pyrazines, however, were less abundant than furan and furan-derivatives. In fact, at pH lower than 7 (such as in sourdough breads) the formation of furan compounds is favorite over pyrazines (Jousse, Jongen, Agterof, Russell, & Braat, 2002). Moreover, also furan compounds were more abundant in sample sets A-C than in samples D and E.

Among sulfur compounds only dimethyl disulfide was detected, in very low amounts. It derives from methionine via the decomposition of its Strecker aldehyde, namely methional (Ho, Zheng, &

Li, 2015).

The sensory profile of Dittaino PDO bread, presented in Table 5, largely agreed with instrumental data. Sensory properties are a key factor in food marketing. For some food products, such as extra virgin olive oil, sensory descriptors are even included in the list of legal parameters to be checked for quality categorization. Although bread sensory properties are not ruled by current laws, their evaluation is very useful to discriminate among bread types and quality levels. Durum wheat breads, in fact, have peculiar sensory features, different from those of common wheat breads (Pasqualone, 2012).

Crust was always scored as brown and very thick, with a moderately crispy consistency, as expected in 1-kg bread loaves submitted to prolonged baking. This result accomplished the requirements of the official technical sheet of Dittaino PDO bread (European Commission, 2014b). Sample sets A, B and C showed significantly darker and thicker crust than D and E, as already indicated by colorimetric data. Also crumb color, perceived as yellow (score range 5.0-6.7), agreed with the colorimeter determination and carotenoid content of bread crumb.

Crumb was highly consistent (score range 4.6-5.7) and cohesive (score range 4.6-5.9), in agreement with instrumental evaluations of hardness and resilience, respectively. Crumb grain was rather coarse (score range 4.7-5.8) due to the process of leavening based on sourdough, which causes a slower and more gradual production of CO_2 allowing small alveoli to merge into larger ones (Crowley, Schober, Clarke, & Arendt, 2002).

Among the taste attributes, the most markedly perceived was sour taste, scored from 2.2 to 3.9 and largely overcoming sweet, salty and bitter taste. Sour taste was due to the sourdough-based leavening procedure, that also influenced bread odor. Sour taste and sour odor (the latter scored from 2.7 to 5.3) were perceived with stronger intensity in sample sets D and E, according to the determination of volatile compounds (acetic acid, in particular). Also toasted odor was markedly perceived (score range 3.1-4.7), with significantly higher values in sample sets A, B and C, reflecting the level of volatiles of thermal origin (furans and pyrazines).

4. Conclusions

The obtained results allowed to characterize in detail the quality level of Dittaino PDO bread and its starting semolina. In particular, these data also allowed to define the width of the quality variations, related to raw material variability and to the intrinsic nature of an artisanal productive process.

It is a matter of fact that a certain niche market for artisanal agri-food products, obtained according to traditional recipes and processing technologies, with features of high quality and genuineness, has been established. However, an effort in keeping quality as much constant as possible has to be made, even in an artisanal process. With this aim, some well-established commercial quality indices of re-milled semolina, such as farinograph and alveograph parameters and color indices, could be helpful in setting up a voluntary quality standard for the producers of Dittaino PDO bread, without the need of modifying the basic official technical sheet approved at European level. These suggestions could enhance quality and further increase the appreciation of end-users.

Finally, the results of the sensory evaluation of Dittaino PDO bread were in agreement with the instrumental physico-chemical analyses and allowed to point out the distinctive characteristics of this kind of bread. In particular, although a certain variability among samples was observed, the sensory profile was dominated by a thick brown crust with a marked toasted odor note, coupled to yellow and consistent crumb with coarse grain and with well-perceivable sour taste and sour odor notes. These sensory features could be highlighted and communicated to consumers for further increasing product knowledge and appreciation.

References

1. AACC International (2000). *Approved methods of analysis* (10th Ed.). St Paul, MN: American Association of Cereal Chemists.
2. AOAC (2000). *Official methods of analysis of the Association of Official Analytical Chemists* (17th Ed.). Gaithersburg, MD: Association of Official Analytical Chemists.
3. Arendt, E. K., Ryan, L. A., & Dal Bello, F. (2007). Impact of sourdough on the texture of bread. *Food Microbiology*, *24*, 165–174.
4. Bianchi, F., Careri, M., Chiavaro, E., Musci, M., & Vittadini, E. (2008). Gas chromatographic–mass spectrometric characterisation of the Italian protected designation of origin “Altamura” bread volatile profile. *Food Chemistry*, *110*, 787–793.
5. Brescia, M. A., Sacco, D., Sgaramella, A., Pasqualone, A., Simeone, R., Peri, G., & Sacco, A. (2007). Characterisation of different typical Italian breads by means of traditional, spectroscopic and image analyses. *Food Chemistry*, *104*, 429–438.
6. Chang, C. Y., Seitz, L. M., & Chambers IV, E. (1995). Volatile flavor components of breads made from hard red winter wheat and hard white winter wheat. *Cereal Chemistry*, *72*, 237-242.
7. Chiavaro, E., Vittadini, E., Musci, M., Bianchi, F., & Curti, E. (2008). Shelf-life stability of artisanally and industrially produced durum wheat sourdough bread (“Altamura bread”). *LWT-Food Science and Technology*, *41*, 58–70.
8. Crowley, P., Schober, T. J., Clarke, C. I., & Arendt, E. K. (2002). The effect of storage time on textural and crumb grain characteristics of sourdough wheat bread. *European Food Research and Technology*, *214*, 489–96.
9. D’Amico, A. (2004). The enhancement of the typical products value: from commodity to experience. The case of Esperya.com. *British Food Journal*, *106*, 793–805.

Preprint of the article: Giannone, V., Giarnetti, M., Spina, A., Todaro, A., Pecorino, B., Summo, C., Caponio, F., Paradiso, V.M. and Pasqualone, A., 2018. Physico-chemical properties and sensory profile of durum wheat Dittaino PDO (Protected Designation of Origin) bread and quality of re-milled semolina used for its production. *Food Chemistry*, 241, 242-249. https://doi.org/10.1016/j.foodchem.2017.08.096

10. De Vuyst, L., & Neysens, P. (2005). The sourdough microflora: biodiversity and metabolic interactions. *Trends in Food Science and Technology*, *16*, 43–56.

11. European Commission. (2003). EC Council Regulation no. 1291/2003 of 18 July, 2003 supplementing the Annex to Regulation (EC) No 2400/96 on the entry of certain names in the 'Register of protected designation of origin and protected geographical indications' provided for in Council Regulation (EEC) no. 2081/92 on the protection of geographical indications and designations of origin for agricultural products and foodstuffs (Pane di Altamura). *Official Journal of the European Union*, *L181*, 12–19.

12. European Commission. (2009). EC Council Regulation no. 516/2009 of 17 June, 2009 entering a name in the register of protected designations of origin and protected geographical indications [Pagnotta del Dittaino (DOP)]. *Official Journal of the European Union*, *L155*, 7–8.

13. European Commission. (2014a). Commission Implementing Regulation (EU) no. 843/2014 of 23 July 2014 entering a name in the register of protected designations of origin and protected geographical indications - Upplandskubb (PDO). *Official Journal of the European Union*, *L232*, 1–1.

14. European Commission (2014b). Commission Implementing Regulation (EU) no. 613/2014 of 3 June 2014 approving a minor amendment to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Pagnotta del Dittaino (PDO)]. *Official Journal of the European Union*, *L168*, 68–72.

15. European Commission. (2016a). Agriculture and Rural Development, Agriculture and food DOOR Portal. Website http://ec.europa.eu/agriculture/quality/door/list.html Accessed on May, 16th, 2016.

Preprint of the article: Giannone, V., Giarnetti, M., Spina, A., Todaro, A., Pecorino, B., Summo, C., Caponio, F., Paradiso, V.M. and Pasqualone, A., 2018. Physico-chemical properties and sensory profile of durum wheat Dittaino PDO (Protected Designation of Origin) bread and quality of re-milled semolina used for its production. *Food Chemistry*, 241, 242-249. https://doi.org/10.1016/j.foodchem.2017.08.096

16. European Commission. (2016b). Commission Implementing Regulation (EU) no. 2016/303 of 1 March 2016 entering a name in the register of protected designations of origin and protected geographical indications - Pane Toscano (PDO). *Official Journal of the European Union*, *L58*, 24–27.
17. European Parliament and European Council. (2012). Regulation (EU) no. 1151/2012 of the European Parliament and of the Council of 21 November 2012 on quality schemes for agricultural products and foodstuffs. *Official Journal of the European Union*, *L343*, 1–29.
18. Frankel, E. N. (1983). Volatile lipid oxidation products. *Progress in Lipid Research*, *22*, 1-33.
19. Jousse, F., Jongen, T., Agterof, W., Russell, S., & Braat, P. (2002). Simplified kinetic scheme of flavor formation by the Maillard reaction. *Journal of Food Science*, *67*, 2534-2542.
20. Giannone, V., Lauro, M. R., Spina, A., Pasqualone, A., Auditore, L., Puglisi, I., & Puglisi, G., (2016). A novel α-amylase-lipase formulation as anti-staling agent in durum wheat bread. *LWT-Food Science and Technology*, *65*, 381–389.
21. Goesaert, H., Brijs, K., Veraverbeke, W. S., Courtin, C. M., Gebruers, K. & Delcour, J. A. (2005). Wheat flour constituents: how they impact bread quality, and how to impact their functionality. *Trends in Food Science and Technology*, *16*, 12–30.
22. Ho, C. T., Zheng, X., & Li, S. (2015). Tea aroma formation. *Food Science and Human Wellness*, *4*, 9-27.
23. ISO (2009). *ISO 3093:2009. Wheat, rye and their flours, durum wheat and durum wheat semolina. Determination of the falling number according to Hagberg-Perten.* Geneva, Switzerland: ISO.

Preprint of the article: Giannone, V., Giarnetti, M., Spina, A., Todaro, A., Pecorino, B., Summo, C., Caponio, F., Paradiso, V.M. and Pasqualone, A., 2018. Physico-chemical properties and sensory profile of durum wheat Dittaino PDO (Protected Designation of Origin) bread and quality of re-milled semolina used for its production. *Food Chemistry*, 241, 242-249.
https://doi.org/10.1016/j.foodchem.2017.08.096

24. ISO (2010). *ISO 12099:2010. Animal feeding stuffs, cereals and milled cereal products. Guidelines for the application of near infrared spectrometry.* Geneva, Switzerland: ISO.
25. Licciardello, F., Giannone, V., Del Nobile, M. A., Muratore, G., Summo, C., Giarnetti, M., Caponio, F., Paradiso, V.M., & Pasqualone, A. (2017). Shelf life assessment of industrial durum wheat bread as a function of packaging system. *Food Chemistry*, *224*, 181-190.
26. Martínez-Anaya, M. A. (1996). Enzymes and bread flavor. *Journal of Agricultural and Food Chemistry*, *44*, 2469-2480.
27. Martínez-Anaya, M. A., Pitarch, B., Bayarri, P., & Benedito de Barber, C. (1990). Microflora of the sourdoughs of wheat flour bread x interactions between yeasts and lactic acid bacteria in wheat doughs and their effects on bread quality. *Cereal Chemistry*, *67*, 85-91.
28. Minervini, F., Di Cagno, R., Lattanzi, A., De Angelis, M., Antonielli, L., Cardinali, G., Cappelle, S., & Gobbetti, M. (2012). Lactic acid bacterium and yeast microbiotas of 19 sourdoughs used for traditional/typical Italian breads: interactions between ingredients and microbial species diversity. *Applied and Environmental Microbiology*, *78*, 1251–1264.
29. Pasqualone, A. (2012). Italian durum wheat breads. In M. T. Pedrosa Silva Clerici (Ed.), *Bread consumption and health* (pp. 57–79). Hauppage, NY: Nova Science Publisher Inc.
30. Pasqualone, A., Alba, V., Mangini, G., Blanco, A., & Montemurro, C. (2010). Durum wheat cultivar traceability in PDO Altamura bread by analysis of DNA microsatellites. *European Food Research and Technology*, *230*, 723–729.
31. Pasqualone, A., Caponio, F., & Simeone, R. (2004). Quality evaluation of re-milled durum wheat semolinas used for bread-making in Southern Italy. *European Food Research and Technology*, *219*, 630–634.

32. Pasqualone, A., Piarulli, L., Mangini, G., Gadaleta, A., Blanco, A., & Simeone, R. (2015). Quality characteristics of parental lines of wheat mapping populations. *Agricultural and Food Science*, *24*, 118-127.
33. Pasqualone, A., Summo, C., Bilancia, M. T., & Caponio, F. (2007). Variation of the sensory profile of durum wheat Altamura PDO (Protected Designation of Origin) bread during staling. *Journal of Food Science*, *72*, S191–S196.
34. Pozo-Bayón, M.A., Guichard, E., & Cayot, N. (2006). Flavor control in baked cereal products. *Food Reviews International*, *22*, 335-379.
35. Pripis-Nicolau, L., De Revel, G., Bertrand, A., & Maujean, A. (2000). Formation of flavor components by the reaction of amino acid and carbonyl compounds in mild conditions. *Journal of Agricultural and Food Chemistry*, *48*, 3761-3766.
36. Raffo, A., Pasqualone, A., Sinesio, F., Paoletti, F., Quaglia, G., & Simeone, R. (2003). Influence of durum wheat cultivar on the sensory profile and staling rate of Altamura bread. *European Food Research and Technology*, *218*, 49–55.
37. Rehman, S., Paterson, A., & Piggott, J. R. (2006). Flavour in sourdough breads: a review. *Trends in Food Science & Technology*, *17*, 557-566.
38. Ruiz, J. A., Quilez, J., Mestres, M., & Guasch, J. (2003). Solid-phase microextraction method for headspace analysis of volatile compounds in bread crumb. *Cereal Chemistry*, *80*, 255-259.
39. UNI (1979). *UNI method no. 10690. Durum wheat and semolina. Determination of gluten quality. Gluten index method.* Milan, Italy: UNI.
40. UNI (1995). *UNI method no. 10453. Durum wheat and semolina. Determination of rheological properties using an alveograph.* Milan, Italy: UNI.

Preprint of the article: Giannone, V., Giarnetti, M., Spina, A., Todaro, A., Pecorino, B., Summo, C., Caponio, F., Paradiso, V.M. and Pasqualone, A., 2018. Physico-chemical properties and sensory profile of durum wheat Dittaino PDO (Protected Designation of Origin) bread and quality of re-milled semolina used for its production. *Food Chemistry*, 241, 242-249. https://doi.org/10.1016/j.foodchem.2017.08.096

Table 1

Descriptive terms used for sensory profiling Dittaino PDO bread samples.

Descriptor	Definition	Scale anchors min (0)	max (9)
Visual appearance			
Crust color	Color tone and intensity of crust	Same color of crumb	Dark brown
Crust thickness	Crust depth	Very thin (1 mm)	Very thick (>5 mm)
Crumb color	Color tone and intensity of crumb	Whitish	Light yellow
Crumb grain	Cell structure of crumb grain	Thin and very homogeneous (1-2 mm pores)	Coarse and poorly homogeneous (the biggest pore > 30 mm)
Visual-tactile and chewing characteristics			
Crumb consistency	Consistency of crumb, evaluated by fingers and during chewing	Soft	Tough
Crumb cohesiveness	The way the crumb reacts when broken by fingers	Poorly cohesive, it crumbles	Very cohesive, it sticks
Odor attributes			
Semolina (evaluated in crumb)	Intensity of typical semolina odor	None	Strong
Sour (evaluated in crumb)	Intensity of the aromatics associated with sourdough fermentation	None	Strong
Toasted (evaluated in crust)	Intensity of the aromatics associated with toasted bread	None	Strong
Taste attributes			
Salty	Primary sensation produced by sodium chloride	None	Strong
Sweet	Primary sensation produced by sugars	None	Strong
Bitter	Primary sensation produced by caffeine	None	Strong
Sour	Primary sensation produced by acid substances	None	Strong

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Table 2

Chemical and rheological characteristics of durum wheat re-milled semolina used in the production of Dittaino PDO bread. Two re-milled semolina samples were collected at each sampling.

Parameter	**Sampling**				
	A	**B**	**C**	**D**	**E**
Falling Number (s)	586 ± 4^{B}	533 ± 3^{D}	562 ± 3^{C}	556± 5 ^{C}	644 ± 6^{A}
Protein (g/100 g dry basis)	12.3 ± 0.1^{A}	12.2 ± 0.1^{AB}	12.2 ± 0.1^{AB}	12.0 ± 0.1 ^{B}	12.1 ± 0.1^{AB}
Dry gluten (g/100 g dry basis)	10.5 ± 0.1^{A}	10.0 ± 0.1^{B}	10.0 ± 0.1^{B}	9.2 ± 0.1 ^{C}	9.9± 0.1^{B}
Ash (g/100 g dry basis)	0.88 ± 0.01^{AB}	0.87 ± 0.01^{BC}	0.89 ± 0.01^{A}	0.87 ± 0.0 1 ^{BC}	0.86± 0.01^{C}
Alveograph parameters					
Tenacity/extensibility ratio	2.4 ± 0.1^{BC}	2.7 ± 0.1^{A}	2.2 ± 0.2^{C}	2.6 ± 0.1^{AB}	2.7 ± 0.1^{A}
Deformation energy ×10^{-4} (J)	223 ± 4^{A}	208 ± 5^{B}	210 ± 5^{B}	212 ± 4^{B}	193 ± 4^{C}
Farinograph parameters					
Water absorption at 500 B.U. (g/100 g)	62.3 ± 0.4^{A}	60.5± 0.6^{B}	59.3 ± 0.2^{C}	59.6± 0.2^{C}	59.7 ± 0.2^{BC}
Dough development time (s)	93 ± 4^{C}	108 ± 1^{B}	111 ± 2^{AB}	114 ± 1^{A}	111 ± 4^{AB}
Dough stability (s)	327 ± 4^{A}	171 ± 5^{D}	180 ± 4^{CD}	306 ± 6^{B}	187 ± 4^{C}
Softening index (B.U.)	46 ± 1^{C}	65 ± 1^{A}	66 ± 2^{A}	59 ± 1^{B}	59 ± 1^{B}
Color indices					
Yellow index (b*)	21.0 ± 0.1^{A}	19.2 ± 0.2^{B}	18.4 ± 0.2^{C}	18.0 ± 0.1 ^{C}	18.1 ± 0.1^{C}
Red index (a*)	-2.3 ± 0.1^{C}	-1.9 ± 0.1^{B}	-1.6± 0.3^{AB}	-1.5 ± 0.1^{A}	-1.4 ± 0.1^{A}
Brown index (100 - L*)	10.7 ± 0.1^{C}	10.6± 0.1^{C}	11.0 ± 0.2^{BC}	11.2 ± 0.1^{B}	11.6 ± 0.1^{A}

Different letters in the same row indicate significant differences at p<0.05.
B.U. = Brabender Units.

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Table 3

Main physicochemical characteristics of Dittaino PDO bread. Three bread loaves were collected at each sampling.

Parameter	**Sampling**				
	A	**B**	**C**	**D**	**E**
Crumb moisture (g/100 g)	46.2± 0.4^{A}	45.6 ± 0.6^{A}	41.9 ± 0.2^{BC}	41.4 ± 0.4^{C}	42.5 ± 0.3^{B}
Crumb a_w	0.964 ± 0.002^{A}	0.963 ± 0.003^{A}	0.929 ± 0.003^{B}	0.924 ± 0.003^{B}	0.930 ± 0.002^{B}
Carotenoid pigments (mg/kg)	3.65 ± 0.14^{A}	2.87 ± 0.19^{B}	2.36 ± 0.11^{C}	2.41 ± 0.15^{C}	2.30 ± 0.13^{C}
Specific volume (mL/g)	3.03 ± 0.12^{A}	2.57 ± 0.15^{B}	2.97 ± 0.17^{A}	2.89± 0.16^{AB}	2.28 ± 0.21^{C}
Crumb color indices					
Yellow index (*b**)	21.6 ± 0.5^{A}	20.2± 0.6^{B}	19.3 ± 0.5^{B}	19.9± 0.7^{B}	19.2 ± 0.4^{B}
Red index (*a**)	-2.5 ± 0.1^{A}	-3.1 ± 0.1^{B}	-2.6 ± 0.1^{A}	-3.0± 0.2^{B}	-3.2 ± 0.2^{B}
Brown index (100 - *L**)	27.4 ± 0.5^{B}	25.6 ± 0.4^{C}	25.3 ± 0.9^{C}	24.2 ± 1.3^{C}	29.2 ± 1.0^{A}
Crust color indices					
Yellow index (*b**)	26.8 ± 0.2^{A}	26.0 ± 1.9^{AB}	28.5 ± 0.7^{A}	23.6 ± 0.9^{B}	26.9 ± 0.6^{A}
Red index (*a**)	10.1 ± 0.6^{B}	12.8 ± 0.8^{A}	13.1 ± 0.5^{A}	9.2 ± 0.1^{B}	12.8 ± 0.8^{A}
Brown index (100 - *L**)	55.6 ± 2.5^{A}	54.6 ± 2.1^{A}	52.8 ± 2.0^{A}	47.1 ± 3.0^{B}	49.7 ± 1.8^{AB}
Textural parameters					
Hardness (N)	17.3 ± 0.5^{C}	27.1 ± 1.0^{A}	16.4 ± 0.4^{C}	23.7 ± 0.6^{B}	26.0 ± 0.1^{A}
Springiness (mm)	5.7 ± 0.2^{A}	4.5± 0.4^{C}	5.6 ± 0.1^{A}	5.3 ± 0.1^{AB}	4.6 ± 0.5^{BC}
Resilience	0.95 ± 0.01^{A}	0.89 ± 0.02^{B}	0.96 ± 0.01^{A}	0.87 ± 0.02^{B}	0.89 ± 0.02^{B}
Chewiness (N × mm)	93.7 ± 7.1^{B}	108.5 ± 4.8^{A}	88.2 ± 5.3^{B}	109.2 ± 5.7^{A}	106.4 ± 8.8^{AB}

Different letters in the same row indicate significant differences at $p<0.05$.

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Table 4

Volatile compounds (peak areas expressed as total ion chromatogram × 10^6) of Dittaino PDO bread. Three bread loaves were collected at each sampling.

	Sampling				
	A	B	C	D	E
Alcohols					
Ethanol	245.76 ± 19.24^{A}	257.13 ± 11.06^{A}	196.07 ± 9.18^{B}	137.98 ± 27.61^{C}	146.42 ± 7.41^{C}
Propanol	n.d.	1.03 ± 0.13	n.d.	n.d.	n.d.
3-Methyl-1-butanol	3.08 ± 1.54^{C}	3.17 ± 0.81^{C}	1.67 ± 0.75^{C}	106.13 ± 3.44^{B}	135.03 ± 3.95^{A}
Pentanol	0.95 ± 0.19^{C}	1.07 ± 0.11^{C}	0.79 ± 0.18^{C}	6.48 ± 0.27^{B}	8.86 ± 0.34^{A}
Hexanol	6.14 ± 0.82^{C}	7.75 ± 1.43^{C}	7.57 ± 1.25^{C}	37.28 ± 2.58^{B}	45.22 ± 3.21^{A}
Heptanol	1.32 ± 0.87^{B}	n.d.	n.d.	3.33 ± 0.30^{A}	n.d.
1-Octen-3-ol	n.d.	1.08 ± 0.21^{C}	n.d.	2.19 ± 0.31^{A}	1.62 ± 0.05^{B}
2-Phenylethanol	5.58 ± 0.03^{A}	2.72 ± 1.03^{C}	5.08 ± 1.25^{AB}	4.11 ± 1.03^{BC}	4.97 ± 0.09^{B}
Aldehydes					
2-Methylpropanal	1.33 ± 0.05^{B}	1.27 ± 0.06^{B}	1.04 ± 0.02^{C}	1.25 ± 0.05^{B}	2.34 ± 0.22^{A}
Butanal	1.67 ± 0.16^{B}	2.08 ± 0.17^{A}	0.73 ± 0.33^{C}	n.d.	n.d.
2-Methylbutanal	3.73 ± 0.15^{C}	4.03 ± 0.32^{B}	4.36 ± 0.21^{B}	4.11 ± 0.14^{B}	4.85 ± 0.04^{A}
3-Methylbutanal	10.66 ± 0.10^{C}	10.88 ± 2.11^{BC}	11.76 ± 2.33^{BC}	18.68 ± 2.44^{B}	25.14 ± 1.40^{A}
Pentanal	1.01 ± 0.07^{C}	1.12 ± 0.15^{C}	0.76 ± 0.13^{C}	4.68 ± 0.44^{B}	6.32 ± 0.20^{A}
Hexanal	13.76 ± 1.95^{C}	18.09 ± 1.56^{B}	13.69 ± 3.67^{C}	49.32 ± 0.94^{A}	51.50 ± 1.74^{A}
2-Hexenal	1.94 ± 0.87^{B}	1.33 ± 0.14^{B}	5.01 ± 2.45^{A}	1.27 + 0.08^{B}	n.d.
Heptanal	5.20 ± 1.12	4.86 ± 1.13	5.77 ± 1.63	5.02 ± 1.18	5.27 ± 1.61
2-Heptenal	2.47 ± 0.40	2.85 ± 0.21	2.99 ± 0.58	2.64 ± 0.33	2.57 ± 0.31
Octanal	3.49 ± 0.48	3.69 ± 1.29	3.23 ± 0.23	n.d.	n.d.
2-Octenal	2.37 ± 0.25^{A}	2.38 ± 0.39^{A}	2.35 ± 0.11^{A}	1.52 ± 0.18^{B}	1.38 ± 0.28^{B}
Nonanal	14.87 ± 3.37^{A}	7.70 ± 0.25^{B}	13.01 ± 2.42^{A}	3.78 ± 0.19^{C}	2.49 ± 0.18^{C}
2-Nonenal	4.26 ± 0.22^{B}	3.67 ± 0.22^{C}	5.19 ± 0.17^{A}	3.04 ± 0.14^{D}	1.98 ± 0.05^{E}
Benzaldehyde	10.77 ± 0.32^{B}	10.00 ± 0.37^{B}	10.41 ± 0.87^{B}	12.19 ± 0.58^{A}	12.80 ± 0.75^{A}
Phenylacetaldehyde	1.08 ± 0.11^{B}	0.95 ± 0.08^{B}	2.52 ± 0.19^{A}	1.03 ± 0.10^{B}	0.86 ± 0.21^{B}
Ketones					
2-Butanone	3.72 ± 0.19^{C}	3.21 ± 0.11^{D}	4.36 ± 0.17^{B}	5.22 ± 0.21^{A}	5.45 ± 0.61^{A}
2,3-Butanedione	0.57 ± 0.05^{D}	1.64 ± 0.08^{B}	0.71 ± 0.10^{C}	2.98 ± 0.26^{A}	3.03 ± 0.25^{A}
2-Pentanone	n.d.	0.55 ± 0.01	n.d.	n.d.	n.d.
2,3-Pentanedione	2.17 ± 0.19^{B}	2.51 ± 0.30^{B}	2.63 ± 0.27^{B}	2.82 ± 0.24^{B}	3.23 ± 0.09^{A}
2-Octanone	0.96 ± 0.11	0.93 ± 0.04	0.96 ± 0.10	n.d.	n.d.
Carboxylic acids					
Acetic acid	81.14 ± 4.34^{C}	90.77 ± 6.46^{C}	82.91 ± 3.81^{C}	101.29 ± 3.61^{B}	168.22 ± 0.66^{A}
Propanoic acid	17.71 ± 1.93^{A}	16.86 ± 5.32^{AB}	9.62 ± 1.36^{B}	8.54 ± 2.41^{B}	10.31 ± 2.37^{B}
Butanoic acid	n.d.	n.d.	1.35 ± 0.09	n.d.	n.d.
Hexanoic acid	4.96 ± 0.47^{A}	1.93 ± 0.16^{B}	1.94 ± 1.01^{B}	1.67 ± 0.80^{B}	1.43 ± 0.71^{B}
Heptanoic acid	0.81 ± 0.32	n.d.	n.d.	0.80 ± 0.20	n.d.
Octanoic acid	n.d.	n.d.	1.88 ± 0.82	n.d.	n.d.
Nonanoic acid	2.55 ± 0.47^{B}	3.89 ± 1.01^{A}	n.d.	2.41 ± 0.36^{B}	2.12 ± 0.27^{B}
Decanoic acid	3.28 ± 0.78^{AB}	3.76 ± 0.96^{AB}	4.27 ± 0.40^{A}	3.10 ± 0.17^{B}	4.12 ± 0.22^{A}
Esters					
Ethyl acetate	1.82 ± 0.12^{B}	2.06 ± 0.21^{B}	1.50 ± 0.24^{B}	10.96 ± 0.26^{A}	10.00 ± 2.06^{A}
Furan compounds					
2-Ethyl-4-hydroxy-5-methyl-3(2H)furanone	27.93 ± 8.79^{A}	31.10 ± 5.98^{A}	13.25 ± 4.34^{B}	10.03 ± 2.12^{B}	14.21 ± 3.22^{B}
2-Furanmethanol	11.94 ± 3.85^{ABC}	14.61 ± 5.51^{A}	15.91 ± 4.10^{A}	10.21 ± 0.14^{B}	8.21 ± 0.42^{C}
2-Furanmethanol acetate	1.05 ± 0.04	0.90 ± 0.34	1.37 ± 0.83	n.d.	n.d.
2-Furanmethanol propionate	1.12 ± 0.83^{B}	0.89 ± 0.65^{B}	2.73 ± 0.13^{A}	0.71 ± 0.11^{B}	0.56 ± 0.04^{B}
2-Furanylethanone	n.d.	n.d.	n.d.	2.31 ± 0.11^{A}	1.69 ± 0.14^{B}
3-Methylfurfural	1.87 ± 0.09	1.57 ± 0.67	n.d.	n.d.	n.d.
5-Pentylfuran	4.86 ± 0.96^{A}	4.40 ± 1.01^{A}	5.03 ± 1.10^{A}	1.70 ± 0.14^{B}	2.69 ± 0.35^{B}
Acetylfuran	2.10 ± 0.44^{B}	2.28 ± 0.63^{B}	3.33 ± 0.97^{A}	n.d.	n.d.
Furfural	36.29 ± 1.74^{B}	54.32 ± 2.70^{A}	29.38 ± 2.16^{C}	15.83 ± 1.37^{D}	16.98 ± 1.16^{D}

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Pyrazines					
2,3,5-Trimethylpyrazine	n.d.	1.02 ± 0.16	n.d.	n.d.	n.d.
2,6-Dimethylpyrazine	n.d.	0.57 ± 0.12^{B}	0.83 ± 0.01^{A}	n.d.	n.d.
2-Ethyl-3-methylpyrazine	n.d.	n.d.	1.26 ± 0.05	n.d.	n.d.
Ethylpyrazine	2.39 ± 0.49^{A}	2.03 ± 0.38^{A}	2.59 ± 0.69^{A}	0.96 ± 0.21^{B}	0.73 ± 0.10^{B}
Methylpyrazine	1.93 ± 0.67^{A}	2.63 ± 1.10^{A}	2.76 ± 0.97^{A}	0.71 ± 0.09^{B}	0.65 ± 0.11^{B}
Pyrazine	n.d.	0.93 ± 0.06	n.d.	n.d.	n.d.
Sulfur compounds					
Dimethyl disulfide	0.74 ± 0.09^{B}	1.44 ± 0.44^{A}	n.d.	n.d.	n.d.

Different letters within the same row indicate significant differences at $p<0.05$; n.d. = not detected.

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Table 5

Sensory characteristics of Dittaino PDO bread. Three bread loaves were collected at each sampling.

Descriptor intensity	**Sampling**				
	A	**B**	**C**	**D**	**E**
Visual appearance					
Crust color	5.9 ± 0.6^{A}	5.5 ± 0.4^{AB}	5.6 ± 0.5^{AB}	4.8 ± 0.4^{AB}	4.6 ± 0.5^{B}
Crust thickness	5.8 ± 0.4^{A}	5.4 ± 0.6^{AB}	5.7 ± 0.4^{A}	4.7 ± 0.5^{B}	4.8 ± 0.3^{B}
Crumb color	6.7 ± 0.6^{A}	6.0 ± 1.3^{AB}	5.5 ± 0.4^{B}	5.0 ± 0.5^{B}	5.3 ± 0.5^{B}
Crumb grain	5.8 ± 0.6^{A}	5.1 ± 0.4^{AB}	5.7 ± 0.5^{A}	5.0 ± 0.2^{AB}	4.7 ± 0.4^{B}
Visual-tactile and chewing characteristics					
Crumb consistency	4.6 ± 0.3^{B}	5.7 ± 0.6^{A}	4.6 ± 0.3^{B}	5.0 ± 0.6^{AB}	5.6 ± 0.1^{A}
Crumb cohesiveness	5.9 ± 0.6^{A}	5.3 ± 0.7^{AB}	5.9 ± 0.5^{A}	4.6 ± 0.4^{B}	5.0 ± 0.4^{AB}
Odor attributes					
Semolina odor (crumb)	3.2 ± 0.6	3.1 ± 0.7	2.8 ± 0.8	2.4 ± 0.5	2.2 ± 0.6
Sour odor (crumb)	2.7 ± 0.9^{B}	3.0 ± 0.2^{B}	3.1 ± 0.7^{B}	5.1 ± 0.9^{A}	5.3 ± 1.1^{A}
Toasted odor (crust)	4.7 ± 0.9^{A}	4.3 ± 0.6^{A}	4.4 ± 0.7^{A}	3.1 ± 0.4^{B}	3.3 ± 0.3^{B}
Taste attributes					
Salty taste	1.8 ± 0.9	2.0 ± 0.7	1.9 ± 0.6	2.2 ± 0.5	2.4 ± 0.7
Sweet taste	1.6 ± 0.5	1.2 ± 0.2	1.4 ± 0.2	0.8 ± 0.1	1.1 ± 0.1
Bitter taste	0.7 ± 0.1	0.7 ± 0.4	0.5 ± 0.2	0.7 ± 0.1	0.5 ± 0.1
Sour taste	2.2 ± 0.3^{B}	2.2 ± 0.2^{B}	2.3 ± 0.5^{B}	3.8 ± 0.4^{A}	3.9 ± 0.7^{A}

Different letters in the same row indicate significant differences at $p<0.05$.