1 **PHYSICO-CHEMICAL CHANGES IN BREADS FROM BAKE OFF**
2 **TECHNOLOGIES DURING STORAGE**

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10 **Running title:** Frozen partially baked breads staling

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Abstract

Quality of several bread specialties from frozen partially baked breads was assessed to define main quality features. Loss of crust freshness shortly after baking was also determined. Quality parameters that characterize bread crust and crumb were determined by instrumental methods in nine different (regarding to formulation and bake off duration) bread types obtained from frozen partially baked breads. Principal component analysis (PCA) allowed discriminating among bread specialties. Quality parameters that enable the differentiation of wheat bread types were crust mechanical properties together with specific volume, crumb hardness and structure. Crust flaking barely represented a problem in the studied types of bread. Crust mechanical properties were rapidly lost during the first 4 hours after baking and the rate of the process was greatly dependent on the bread type. The force to promote crust fracture underwent increase up to 6 hours after baking and those changes occurred in the A_w range of 0.50-0.74 or moisture content 9-15g/100g.

Key words: bread, instrumental analysis, crust properties, frozen part-baked.

1. Introduction

Bread has an important contribution in a well balanced diet, due to its content on starch and complex carbohydrates. Partially baked bread is an alternative bakery product that is gaining market share every year. It offers several advantages at retails, such as materials and equipment reductions, less production space is required, greater variety of bread, and also at the consumers, which have fresh bread available at any time of the day (Rosell, 2009). Breadmaking technology with partial baking, also referred as bake off technology (BOT), consists in two baking stages (Fik & Surowka, 2002; Bárcenas & Rosell, 2006a,b). First baking gives a product (partially baked bread) with aerated crumb but without crunchy and colored crust. After that bread can be kept refrigerated or frozen. Second or full baking favors water evaporation from the surface layers, and in consequence the crust development, and the Maillard reaction responsible for the coloration and the release of flavors takes place during this stage (Le Bail, Monteau, Margerie, Lucas, Chargelegue & Reverdy, 2005; Farahnaky & Majzoobi, 2008). Similarly to conventional breadmaking, main quality attributes of bread obtained from BOT process are texture and taste. Breads obtained from frozen partially baked breads are frequently connected to negative aspects, mainly associated to mechanical changes in the crust that led to flaking and very rapid crispiness loss (Le Bail et al., 2005). However, studies reported on those effects have been focused on French baguette, which avoid making a general statement about this type of technology.

Industrial practice suggests that the freezing step is responsible of crust flaking, one of the major quality problems of frozen part-baked breads (Hamdami, Pham, Le Bail & Monteau, 2007). Crust flaking resulting from the detachment of some part of the crust

constitutes an important drawback, which has been related to excessive drying of the bread surface at the end of the post-baking chilling and freezing process (Lucas, Quéllec, Le Bail & Davenel, 2005). Crust flaking has been related to concentration of water as ice below the crust during freezing and the mechanical damages due to the intense thermo-mechanical shock during chilling-freezing and final baking (Hamdami et al., 2007).

Additionally, other problem associated to all bakery products is their relative very short shelf life, since certain physical and chemical changes affecting to crust and crumb occur during their storage, which are known as staling (He & Hoskeney, 1990). Breads from frozen partially baked bread are particularly sensitive to changes related to crust crispiness. Crust characteristics are decisive for the purchasing attitude of consumers of this type of product, which is based on the subjective fingers sensory perception (Lambert et al., 2009). A crispy texture is originated when starch and gluten matrix are in glassy state and it has been associated with low moisture content and water activity (Stokes & Donald, 2000). Crispiness is a complex attribute resulting from multiple sensations and influenced by numerous physical parameters, combining molecular, structural and manufacturing process as well as storage conditions (Roudaut, Dacremont, Valles Pamies, Colas & Le Meste, 2002; for additional information see review of Luyten, Plijter & van Vliet, 2004). Crispiness is perceived only for a short time after baking and it is the main attribute that causes consumer rejection (Duizer, 2001). Lately, different studies have been focused on extending the crust mechanical properties by either modifying the bread formulation or the cooling conditions (Primo-Martín, Beukelaer, Hamer & van Vliet, 2008a,b). However, scarce information has been

reported on the variation of the crust mechanical properties of breads obtained from partially baked technologies.

The aim of this study was to select quality parameters that permit a rapid characterization and differentiation of different bread specialties obtained by applying bake-off technologies. For that purpose, diverse instrumental quality parameters related to crust and crumb were assessed. Short term storage behavior was also followed to evaluate crust changes during staling in different bread specialties obtained from BOT processes.

2. Materials and methods

Nine partially baked breads were selected to represent a range of different bread specialties currently available in the Spanish market. Part-baked frozen breads were directly provided by the producers (Forns Valencians S.A., Valencia, Spain) and stored at -18° C until use. A description of the bread ingredients is given in Table 1.

Full baking process and storage

Loaves removed from the freezer were thawed at ambient temperature (18-20° C) for 50 min, and baked in an electric oven (Eurofours, France). Baking conditions (provided by the company) varied with the specialty and were as follows: 180° C for 11 min in the case of pulguita, small ciabatta, small brioche, rustic and brioche, 180° C for 14 min for ximos, and 180° C during 18 min for white loaf, baguette and ciabatta.

After baking, breads were cooled down and stored in a cabinet with controlled both temperature (18°C) and relative humidity (57%) till further characterization. Duplicates of each sample were baked in separate days.

Fresh loaves (0.5 h after baking) and stored loaves (2, 4, 6 and 24 h) were tested for specific volume, water activity, moisture content, crust and crumb color, width/height ratio of the slices, textural characteristics, crust mass ratio, crust flaking, crust section and crumb cell analysis.

Physicochemical analysis

Chemical composition was determined following ICC standard methods (1994) for moisture (ICC 110/1), protein (ICC 105/2), fat (ICC 136) and ash (ICC 104/1). Carbohydrates were determined by difference.

Bread volume was determined by the rapeseed displacement method. The rest of the physicochemical parameters were independently determined in the crust (according to crust section analysis described below) and crumb, which were separated using a razor blade. Color parameters of the crumb and crust were measured at three different locations by using a Minolta colorimeter (Chroma Meter CR-400/410, Konica Minolta, Japan) after standardization with a white calibration plate ($L^* = 96.9$, $a^* = -0.04$, $b^* = 1.84$). The color was recorded using CIE- L^* a^* b^* uniform color space (CIE-Lab), where L^* indicates lightness, a^* indicates hue on a green (-) to red (+) axis, and b^* indicates hue on a blue (-) to yellow (+) axis. Moisture content of the crust and crumb was determined following the ICC Method (110/1, 1994). Water activity (A_w) of crust and crumb samples was measured using an Aqua Lab Series 3 (Decagon devices, Pullman, USA) at 22°C. Crust mass ratio was calculated as the ratio of crust weight and crumb weight.

Puncture tests

The peak force and the peak deformation point of the crust were calculated by punching the sample at three different points of bread surface: in the middle of the crust area and at 2 cm distance on both sides. The average value was determined for each bread variety. Experiments were assessed using a texture analyzer (TA XT plus, Stable Micro Systems, Surrey, UK). A cylindrical probe of 4 mm diameter was used at 40 mm/s cross-speed. This speed was initially chosen by Primo-Martin et al. (2008a) to simulate biting with the front teeth (Vincent, 1998). The failure force was calculated as the peak force observed according to studies by Jackman and Staley (1992). The failure deformation, defined as the deformation at the peak point, was also calculated. Three loaves were used for each bread variety.

Crumb hardness test

Crumb hardness was carried out in a texture analyzer (TA XT plus, Stable Micro Systems, Surrey, UK). A 1 cm-thick slice was compressed with a 25 mm-diameter cylindrical stainless steel probe, up to 50% penetration of its original height at a crosshead speed of 1 mm/s speed.

Crust flaking evaluation

Crust flaking test was carried out in specific crushing system developed by Le Bail et al. (2005). Bread was crushed on its flanks and on its base by 30% of its diameter and height in crushing system. Pieces of the crust were collected and weighed, after that a digital picture of crust pieces was taken. Total number of flakes, total area in mm², average size in mm² and area fraction in percent were determined Using an Image J software.

Crust section and crumb cell analysis

Crust section and crumb cell analysis were performed by scanning longitudinal and cross section of bread sample, 10 mm thick, on flatbed scanner (HP Scanjet 4400c). The crust section was calculated from the scanned samples at the upper and bottom side using an image analysis program (UTHSCSA Image Tool software). For crumb cell, the images were analyzed by Image J software according to Gonzales-Barron and Butler (2006), number of cell, average cell per mm², average diameter per mm² and circularity were calculated.

Statistical analysis

For each quality parameter, a one way analysis of variance (ANOVA) was applied using Statgraphics Plus V 7.1 (Statistical Graphics Corporation, UK), to assess significant differences ($p < 0.05$) among samples that might allow discrimination among them. Principal component analysis (PCA) was also performed to determine the number of principal components that significantly ($p < 0.05$) discriminated breads.

3. Results and discussion

Characterization of fresh breads

Nine bread specialties with rather similar recipes (Table 1) were used to evaluate crust behavior during staling. Physico-chemical characteristics were initially determined, in order to find the main discriminating factors among the specialties that could help to understand possible differences during storage.

Table 2 shows the macronutrients composition of the nine bread specialties that agrees with common bread proximate composition. Fat content was higher in the bread varieties (pulguita, ximos, small brioche and brioche) that contained lard, and

specialties with higher protein content were brioches and small brioches that had milk powder added.

Several technological parameters have been assessed to identify the most discriminating parameter that allows the characterization of the breads. Preliminary analysis of data collected using ANOVA showed that all physicochemical characteristics significantly ($p < 0.05$) discriminated between the bread types tested (Table 3 and 4). The highest specific volume was observed in the brioche type followed by rustic bread (Table 3). Crust properties have been considered essential features for bread quality assessment (Luyten et al., 2004), because of that, special emphasis was put in assessing crust physico-chemical properties. Significant differences ($p < 0.05$) were observed in both the crust and crumb moisture content, which ranged from 4.8 to 11.5 g/100g and from 31.3 to 45.3g/100g, respectively. Similarly, water activity in the crust and in the crumb showed significant differences among bread types; it varied from 0.34 to 0.58 in the crust and from 0.95 to 0.98 in the crumb. Those values agree with previous findings for breads (Baik & Chinachoti, 2000). Moisture content and A_w greatly contribute to crust mechanical properties (Katz & Labuza, 1981).

Regarding crust flaking, significant ($p < 0.05$) differences were observed among the bread types, but in all cases very low flaking (less than 2%) was observed. Crust flaking has been mainly investigated on partially baked French baguette stored under frozen conditions (Le Bail et al., 2005). Only the small ciabatta yielded high amount of flakes, which also showed the highest size. Crumb structure analysis of breads confirmed that bread specialties had significantly ($p < 0.05$) different structure, having very big void spaces or gas cells the ciabatta, white loaf, rustic bread and small ciabatta. Those types of breads also showed higher crumb hardness (Table 4), suggesting coarser crumb

structure with thicker cell walls. The presence of rye flour in their formulation could be responsible of the different crumb structure; although baguette would be an exception, because it contains rye flour and did not exhibited the same crumb behavior.

Crust mechanical properties were assessed by a puncture test, where sufficient strain energy was applied to penetrate in the crispy crust (Table 4). The failure force was related to the hardness necessary to break a brittle material in the mouth during the first bite. A force-deflection curve was obtained, showing a force increase up to a major rupture and a drop to zero when pieces fall away (Vincent, 1998). Higher values of failure force were observed for ciabatta and small ciabatta, followed by baguette and white loaf, which required higher force to promote crust fracture. Presumably, the presence of additional gluten and freeze dried sourdough in the recipe of ciabatta and small ciabatta could be responsible of their harder crust. Considering that crispy texture is generated when materials in a glassy state interrupted with empty spaces (cells or air pockets leading to heterogeneous structure) are subjected to enough energy (Vincent, 1998), higher force in the fresh bread suggests more rigid and firm crust with less cracks in it. On the opposite side, ximos together with brioches, small brioches and pulguita exhibited the lowest failure force with low values of failure deformation, due to thinner crust section (values not showed), likely due to the presence of added fat in their formulation that acts as plasticizer. Failure deformation is referred to the applied deformation that has been related to the teeth displacement (Vincent, 1998). High values of failure deformation have been related to less stiff crust texture when the effect of different hydrocolloids on bread physical properties were studied (Mandala, 2005). However, in the present study where different bread types were evaluated, breads with thick crust gave high values of failure deformation suggesting stiffer crust structure.

Significant quality parameters analyzed by PCA indicated that six principal components significantly ($p < 0.05$) discriminated between bread specialties, which accounted for 95 % of the variability in the original data (data not showed). This analysis described 32% and 22% of variation on principal components 1 (PC1) and 2 (PC2), respectively (Figures 1 and 2). Component 1 was defined by b^* color parameter of the crust, flakes count, specific volume and number of cells along the positive axis, which were present in brioche, small brioche, baguette, and ximos. Along the negative axis, PC1 was described by cell diameter, hardness, area and failure force that were present in ciabatta. Parameters like crust moisture, flakes area and A_w of crumb were present in small ciabatta bread. Conversely, the component 2 was mainly defined by the crust color parameters a^* and L^* , along the positive and negative axis, respectively. Fresh samples were located in different positions (Figure 2), brioche, small brioche, baguette and white loaf breads were positively located along PC1 and PC2. On the other hand, the breads located along the negative axis of PC1 were ciabatta and small ciabatta. Ximos bread and small ciabatta were located along the negative axis of PC2. Therefore, PCA allowed discriminating among bread specialties and it showed that brioches, small brioches and baguette were similar in terms of specific volume, flaking behavior and crumb structure, although they had different ingredients in their formulation. In addition, bread types with similar ingredient composition, like baguette, white loaf and rustic, could be discriminated with the quality parameters from instrumental analysis.

Descriptive sensory attributes have been reported for discriminating among different bread types (Heenan, Dufour, Hamid, Harvey & Delahunty, 2008). In that study, porous appearance and odour attributes were the most important descriptors. The same authors indicated that freshness perception varied widely with consumers, making necessary to

segregate in consumer clusters to obtain a reliable approach for predicting freshness. Main quality parameters obtained from instrumental analysis that influence the consumers' acceptability have been recently defined (Curic, Novotni, Skevin, Collar, Le Bail & Rosell, 2008). The instrumental methods that describe bread appearance, structure and texture were selected to identify and quantify the main discrepancies of wheat bread produced by different breadmaking processes (Curic et al., 2008). In the present study wider range of instrumental parameters for characterizing crust and crumb has been considered and they made possible to differentiate the different bread types obtained from frozen partially baked breads.

Characterization of stored breads

Moisture content increases as bread ages, due to water sorption from the atmosphere and by mass transport from neighboring components of the crumb. The overall result was an increase of crumb hardness and moreover, the crust initially dry and crispy became soft and rubbery (Katz et al., 1981). Water distribution between crust and crumb also contributes largely to the organoleptic perception of freshness (Baik et al., 2000). Therefore, the influence of storage time on the water activity, moisture content and mechanical crust properties were determined in different bread specialties.

The analysis of the breads during storage revealed that moisture content of the crust increased in all types of bread along storage, and the most rapid enhancement was observed during the first 4 hours after baking (Figure 3, Table 4). Fresh samples had an average moisture content of 4.84-11.50 g/100g and after 24-hour storage their moisture content ranged from 10.07- 17.66 g/100g. The slope of the curves that was an indication of the speed of water uptake showed great variation depending on the bread type.

Taking into account that all breads have been stored in the same conditions, and thus they will have similar water sorption from the atmosphere, divergence observed in the curve slopes must be due to variation in the moisture gradient between crust and crumb. However, no relationship was found between the slope of the curves and the gradient of water content between the inner crumb and the outer crust in the different fresh bread types (Table 3). It has been reported that although factors like water content/activity play a significant role, other features like molecular structure, porosity, and crust boundary (Luyten et al., 2004), greatly influenced the water migration through the crumb to the crust, and remains unclear the contribution of each factor.

Water activity in the crust also increased during storage (Figure 4). Again, the most pronounced changes were observed during the first 4 hours after baking (Table 4). The major increase was observed in pulguita, small ciabatta and ciabatta breads. Values for water activity after 24-hours storage varied between 0.59 (brioche) and 0.78 (pulguita). A crispy texture has been associated with low values of moisture content and water activity, when starch and gluten matrix are in a glassy state and thus cell walls more susceptible to fracture (Stokes & Donald, 2000).

The crust failure force during storage (Figure 5, Table 4) was much higher in the aged breads, due to moisture redistribution (water migration from crumb to crust) that leads to a toughen crust (He & Hoskeney, 1990). The highest increase in the crust rupture force was observed for white loaf and ciabatta. In general, the failure force showed a steady increase in all bread specialties up to 6 hours storage, reaching an asymptotic plateau, that suggests the completely loss of crispy texture. Crispness loss during storage seems to be greatly dependent on the water migration from crumb to crust and from the atmosphere. Primo-Martin et al. (2007) stated that amylopectin retrogradation, which is

the main process responsible for the staling of bread crumb, cannot be responsible for crispness deterioration of the crust as amylopectin retrogradation upon storage of breads. Those authors confirmed by X-ray and differential scanning calorimetry (DSC) that starch retrogradation of the crust could only be measured in the crust after 2 days storage.

Changes in the curve trend occurred between 0.50-0.74 A_w or 9-15% moisture content, depending on the bread type. These results agree with previous findings of Primo-Martín, Sozer, Hamer & Van Vliet (2009), who reported that a significant increase of the force required breaking the crust was observed at A_w of 0.65 or higher when using a crust model with similar structure to bread crust.

The instrumental methods applied for bread quality assessment allowed discriminating among bread specialties from frozen partially baked breads. Crust mechanical properties together with specific volume, crumb hardness and structure were the quality parameters that permitted bread differentiation. In general, crust flaking did not represent a problem in the types of bread tested.

Regarding the evolution of the crust physicochemical properties, they changed shortly after baking. Specifically, water content/activity showed a steady increase during the first 4 hours after baking. In addition, the force to promote crust fracture varied up to 6 hours and those changes occurred in the A_w range of 0.50-0.74 or moisture content 9-15%.

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Figure captions

Figure 1. Correlation loadings plot from principal component analysis showing the technological quality parameters of nine bread specialties.

Figure 2. Scores plot from principal component analysis of the nine bread types evaluated for technological quality.

Figure 3. Changes in moisture content of the crust from nine bread types during storage. Symbols: ■: pulguita; ◆: small ciabatta; ▲: ximos; ●: small brioches; □: rustic; ○: brioche; Δ: white loaf; ◇: baguette; —: ciabatta.

Figure 4. Water activity in bread crust as a function of storage time for different specialty breads. Symbols: ■: pulguita; ◆: small ciabatta; ▲: ximos; ●: small brioches; □: rustic; ○: brioche; Δ: white loaf; ◇: baguette; —: ciabatta.

Figure 5. Evolution of failure force of the crust for different specialty breads during storage. Symbols: ■: pulguita; ◆: small ciabatta; ▲: ximos; ●: small brioches; □: rustic; ○: brioche; Δ: white loaf; ◇: baguette; —: ciabatta.

Table 1. Bread product sample and listed ingredients (information supplied by producer). Special ingredients for each type of bread appeared highlighted.

Product	Ingredients
Pulguita	Wheat flour, lard , yeast, salt, bread improver
Small ciabatta	Wheat flour, rye flour, salt, bread improver, freeze dried sourdough , yeast and gluten
Ximos	Wheat flour, lard , yeast, salt, bread improver
Small brioches	Wheat flour, sugar, lard, milk powder , yeast, salt, bread improver
Rustic	Wheat flour, rye flour, yeast, salt, bread improver
Brioche	Wheat flour, sugar, lard, milk powder , yeast, salt, bread improver
White loaf	Wheat flour, rye flour, yeast, salt, bread improver
Baguette	Wheat flour, rye flour, yeast, salt, bread improver
Ciabatta	Wheat flour, rye flour, salt, bread improver, freeze dried sourdough , yeast and gluten

Table 2. Chemical proximate composition (expressed as g/100g as is) of nine breads obtained from frozen partially baked breads.

	Carbohydrates	Fats	Proteins	Minerals
Product				
Pulguitas	60.0	2.73	6.39	0.70
Small				
ciabatta	59.1	0.72	5.41	0.50
Ximos	60.1	2.74	6.41	0.70
Small				
brioche	55.1	3.59	7.32	0.60
Rustic	63.6	0.48	7.06	0.50
Brioche	55.1	3.59	7.32	0.60
White loaf	62.4	0.48	7.05	0.50
Baguette	62.5	0.48	7.20	0.70
Ciabatta	58.3	0.71	4.67	0.50
<i>Mean</i>	59.6	1.72	6.54	0.59
<i>SD</i>	3.1	1.40	0.94	0.09

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Table 3. Technological quality parameters of nine bread specialties obtained from frozen partially baked breads.

Product	Specific volume		Moisture content (g/100g)			Aw		Crust color			CM ratio	Flakes count	Flakes area	Cells number	Cells area
	mL/g		Crust	Crumb		Crust	Crumb	<i>L</i> *	<i>a</i> *	<i>b</i> *	g crust /100g		mm ²		mm ²
Pulguita	3.3	ab	5.3 b	39.7 d		0.57 f	0.97 cd	63.4 e	3.9 a	29.9 b	0.61 a	1230 a	504 a	386 c	0.65 b
Small ciabatta	3.0	a	11.5 h	39.4 c		0.54 e	0.97 cd	61.6 e	9.0 b	33.7 cd	0.43 a	5852 h	120981 i	177 b	2.23 e
Ximos	3.0	a	9.5 f	31.3 h		0.43 c	0.97 cd	67.4 f	8.8 b	38.7 e	0.47 ab	1819 d	2501 h	75 a	0.45 a
Small brioches	3.5	b	7.6 d	38.7 b		0.46 c	0.95 a	47.8 a	16.1 e	31.3 bc	0.48 ab	1588 b	1480 e	436 e	1.08 d
Rustic	5.6	d	6.9 c	45.3 g		0.58 f	0.98 e	56.5 d	11.2 bc	28.6 b	0.45 a	1729 c	934 d	416 d	2.28 ef
Brioche	6.0	d	8.5 e	38.7 b		0.45 c	0.96 b	53.2 bc	15.0 de	36.1 de	0.55 bc	4063 f	1497 f	607 f	0.64 b
White loaf	3.4	ab	5.3 b	43.5 e		0.34 a	0.98 de	56.5 cd	12.3 cd	36.2 de	0.47 a	3504 e	830 c	607 f	2.33 f
Baguette	4.9	c	4.8 a	44.5 f		0.38 b	0.97 c	52.6 b	14.8 de	34.7 d	0.45 a	4431 g	2068 g	375 c	0.72 c
Ciabatta	3.4	ab	10.8 g	37.3 a		0.517 d	0.968 ce	45.6 a	13.6 cde	20.9 a	0.64 d	1816 d	783 b	74 a	7.64 g
P- value	0.000		0.000	0.000		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

CM ratio: crust mass ratio: g crust/100g bread

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455 **Table 4.** Crust and crumb textural properties of nine bread specialties obtained from frozen partially baked breads.

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Product	Hardness		Failure force		Area		Failure deformation	Moisture content at 4h storage	Aw at 4h storage	Failure force at 4h storage
	N		N		N/m		mm	g/100g		N
Pulguita	1.66	bc	12.06	b	3.17	abc	5.40 c	13.5 c	0.67 c	14.41 b
Small ciabatta	2.05	e	21.62	d	4.88	de	4.93 bc	14.3 d	0.68 c	25.28 c
Ximos	1.91	de	7.85	a	2.16	a	5.13 c	13.1 c	0.68 c	11.42 a
Small brioches	1.59	b	8.33	a	2.38	ab	2.93 a	9.9 b	0.53 b	11.20 a
Rustic	2.39	f	11.52	b	3.53	bc	7.07 d	8.3 a	0.66 c	14.53 b
Brioche	1.82	cd	8.70	a	2.00	a	3.73 ab	9.1 ab	0.54 b	12.22 a
White loaf	1.96	de	17.75	c	5.46	de	6.93 d	8.7 a	0.45 a	32.19 d
Baguette	1.39	a	18.06	c	4.36	cd	6.27 cd	9.4 b	0.45 a	26.92 c
Ciabatta	3.82	g	22.54	d	5.79	e	6.70 d	15.1 e	0.74 d	37.57 e
P- value	0.000		0.000		0.000		0.000	0.000	0.000	0.000

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459 **Figure 1.**
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462 **Figure 2.**

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Figure 3.

469 **Figure 4.**

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Figure 5.