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Influence of different baking powders on physico-chemical, sensory and volatile compounds in biscuits and their impact on textural modifications during soaking

Giada Canali[1] · Federica Balestra[2] · Virginia Glicerina[1] · Federica Pasini[1] · Maria Fiorenza Caboni[1,2] · Santina Romani[1,2]

Abstract The aim of this study was to investigate the effects of different type of leavening agents (ammonium bicarbonate—A, cream of tartar—B, grape-based leavening agent—C, baking powder—D) on the structural (also after soaking), physico-chemical and other quality characteristics of biscuits made with the same formulation. The major changes observed between samples were related to their textural properties, volatile profile and sensory characteristics. The presence of ammonium bicarbonate in sample A, gave arise to a biscuit with the most homogeneous surface appearance, the less hard structure and highest fracturability and spread, that promoted their greater milk uptake during soaking. The B and C biscuits, made with baking powder having tartaric acid, were characterized by a crisper texture and lower sensory overall acceptability. The presence in formulation of baking powders made with cream of tartar (B) and ammonium bicarbonate (A) mostly influenced the biscuit volatile profiles. Biscuit sample obtained with the most common baking powder (diphosphates powder) showed in general intermediate characteristics between A and B–C samples. Results obtained in this study demonstrate that a lot of final properties of biscuits can be strongly influenced by the type of leavening agents used in formulation.

Keywords Aroma profile · Soaking process · Quality · Peleg model

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✉ Virginia Glicerina
virginia.glicerina2@unibo.it

[1] Interdepartmental Centre for Agri-Food Industrial Research, Alma Mater Studiorum, University of Bologna, Via Quinto Bucci 336, 47521 Cesena, FC, Italy

[2] Department of Agricultural and Food Science, Alma Mater Studiorum, University of Bologna, Piazza Goidanich 60, 47521 Cesena, FC, Italy

Introduction

Biscuits or cookies, as named in the USA, are a popular food product, consumed by a wide range of people, due to their various sensory characteristics, long shelf-life and relatively low cost (Romani et al. 2012). The main difference between biscuits and other cereal bakery products is a 5% lower moisture content in the former. Generally, at least 60% of biscuits formulation is represented by wheat flour. Other ingredients normally used are fat or shortening, sugar, eggs, milk and leavening agents, in different proportions depending on product type (Drakos et al. 2019). Biscuits can be classified in different ways depending: on their final textural characteristics, on the used forming method of dough (e.g. fermented, developed, laminated, cut, moulded, extruded, deposited, wire cut, coextruded) and on their formulation (Manley 2001; Zydenbos et al. 2004). Regarding formulation, biscuits can be divided into: hard dough products, characterized by 6–10% of fat and 10–15% of sugar; short dough, with low sugar and fat content, in which the fat amount is about 12–19% and sugar 14–20%; short dough, with high sugar and fat content, in which the fat amount is around 18–22% and sugar 15–30%; soft dough, with fat around 30%and sugar about 33–60% (Lanza 2006). Hard dough biscuits are generally crispy and crunchy with an open texture (e.g. *tea biscuits, garibaldi fruit ones, hardtack*, etc.), short dough ones are brittle and less plastic than the previous ones (e.g.

shortbread, shortcake, Italian frollini, etc.); Soft dough ones are characterized by a soft texture that makes them fragile and subjected to breakage (e.g. *sponge biscuits, meringues*, etc.) (Lanza 2006).

Biscuits process steps, such as weighing and mixing of ingredients, dough sheeting, dough sheet relaxation, shape forming, leavening, baking, cooling and packaging, are important in determining the characteristics of the final product (Cronin and Preis 2000). Several studies dealing with biscuits and reported in scientific literature (Manohar and Rao 2002; Caponio et al. 2008; Drakos et al. 2019) evaluated the influence of type and quantity of the main ingredients, the effect of mixing time and operating methods, the influence of additives, emulsifiers, fat level and type on the quality of the final product.

Leavening and baking are the main steps that affect the structure development of baked products. During leavening an increase in the volume of the product takes place because of air and other gases, such as CO_2, produced by leavening agents simultaneously to a reduction in product density associated with the development of an open porous structure (Lanza 2006; Gökmen et al. 2008). Moreover, during baking a reduction of moisture up to 4% and a change in the surface color is observed. In fact, during this step sugars together with proteins, due to Maillard reaction, tend to produce browning and specific volatile compounds (Bastos et al. 2012). The high temperature needed for this reaction is reached only in the outer zones of the product, once it is sufficiently dried with a low water activity. The duration of this phase depends on the initial water content of the dough and it is ruled by heat and mass transfer phenomena (Asselman et al. 2007).

Baking powders, predominantly composed by inorganic salts, are important ingredients in biscuit recipes. In particular, chemical leavening agents, in the form of different bicarbonates (ammonium and sodium ones), are widely used in baked goods. In chemically leavened products, CO_2 can be obtained by bicarbonates decomposition thanks to the high temperatures produced during baking and through the chemical reaction of bicarbonates with leavening acids (Lajoie and Thomas 1991; Vetter 2003). However, in the case of sodium bicarbonates, the carbonate formed by its decomposition is very alkaline, giving arise to products with high pH and unpleasant taste, known as "soda bite", together with other undesirable effects, for example on crumb color. Moreover, the yield of carbon dioxide obtained through chemical reaction of sodium bicarbonate with acids is greater than that obtained by heat decomposition. For these reasons, sodium bicarbonate is not used alone but in combination with an acid, unlike ammonium one that in low moisture products leaves no residues when it decomposes by heat (Taylor et al. 2012).

During baking CO_2 as is released, expanding the dough and imparting specific characteristics to the final product (Sharma et al. 2017). The amount and type of leavening agents to add in biscuit formulation should be selected on the basis of the final product characteristics required such as: taste, rheological and textural properties, color and aromatic profile. Among them texture, aroma and color, influencing consumer acceptance and preferences, are considered key attributes. In particular, the choice and the combinations of different bicarbonate with different acids and flour components will result in release of leavening gases of several profiles which will be appropriate for different bakery products (Brose and Becker 2001). For what concern texture characteristics, biscuits take up moisture very quickly when immersed in a liquid medium, such as semi-skimmed milk, leading to undesirable changes in texture. During immersion it is possible to observe a hardness and a crunchiness loss parallel to a plastic deformation, probably related to the plasticization and softening milk effect on the starch/protein matrix, which alters the mechanical strength of the product (Sacchetti et al. 2003; Romani et al. 2016).

To our knowledge, only one preliminary research (Sharma et al. 2017) was performed on the influence of different baking powders in combination with different level of acid calcium phosphate on some cookies and cake quality characteristics.

The aim of this research was to investigate the effects of four different commercial leavening agents, on chemico-physical, textural, sensory and aroma profile of biscuits considering also their structural changes during soaking. These leaving agents, normally employed in bakery and confectionary companies and recommended for biscuits manufacturing, were in specific: ammonium bicarbonate, cream of tartar, grape-based leavening agent and a baking powder.

Materials and methods

Materials

The experimental variables used to differentiate the biscuit formulations were the type and the amount of the commercial leavening agent: ammonium bicarbonate (F.lli Rebecchi, Valtrebbia, Italy), cream of tartar (Giovanni Randi Spa, Forlì, Italy), a grape based leavening agent (F.lli Rebecchi, Valtrebbia, Italy) and a standard baking powder (Cameo, Bielefeld, Germany). The composition of commercial baking powders, as on label, and samples name are reported as follows:

- Sample A (Ammonium bicarbonate), made up from ammonium bicarbonate (E503).
- Sample B (Cream of tartar), made up from potassium tartrate from grapes, sodium bicarbonates and corn starch.
- Sample C (Grape based leavening agent), made up from grape juice (42%), sodium bicarbonate and corn-starch.
- Sample D (Baking powder), made up from diphosphates, sodium bicarbonates and corn-starch.

The amount of leavening agents added in biscuit formulations as reported in their label, were: 20 g for A, 36 g for B, 30 g for C and 20 g for D, while the tartaric acid content of cream of tartar and grape based leavening agent, as analysed by HPLC, were respectively, 22.3 g/100 g in B sample and 36.3 g/100 g in C one.

Biscuits formulation was set-up after preliminary trials by using a traditional recipe. All ingredients were purchased from a local market.

The amounts of wheat flour (1000 g), sugar (250 g), eggs (200 g), butter (200 g) and milk (100 g) were kept constant in all formulations; the quantity of each leavening agent was chosen following the doses recommended by their producers in the label, verified after preliminary trials.

Biscuits preparation

All the ingredients were weighed and mixed in a household mixer (Bimby Robot TM31, Vorwerk, Germany) in the following order: wheat flour, sugar, butter, eggs, milk and leavening agent. After 80 s of mixing the dough was kneaded for few s to compact it and then sheeted to a thickness of about 3.0 mm by a pasta filler machine (Mod. SFSI 42040050T, GAM International, Italy), cut by using a stainless-steel circular mould (3.5 cm diameter) and placed on a tray. Biscuits were baked in an electric thermo-conventional oven (Apollo PS1 Steamer, AEG, Porcia, Italy) at 170 °C for 14 min. After baking, biscuits were removed from the oven, placed on a grid and kept cooling at 23 ± 1 °C for 1 h. For each formulation three batches of about 50 biscuits were produced. Biscuit samples were named as previous reported.

Methods

Tartaric acid analysis

Tartaric acid was measured, according with the official method (AOAC 2000) with some modifications, to identify its amount in cream of tartar and in the grape-based leavening agent. One g of sample was weighed into a beaker and 50 mL of water and 7 mL of chloridric acid solution (37 g/L) were added. The solution was heated till boiling, transferred in a flask and the volume adjusted to 500 mL with water. After filtration (2 μm filter) the clear extract (20 μL) was injected onto an HPLC Agilent 1260 Infinity II series (Agilent Technologies, Santa Clara, USA). Separation of the tartaric acid was performed using a Hi Plex H (150 × 4.6 mm, 3.5 μm) LC-column. The LC system was equipped with a detector UV/VIS and a standard cell of 4000 kPa G7114A1260 VWD. Mobile phase prepared mixing 2.45 g/L of orto-phosphoric acid solution (2 mol/L). The flow rate was set to 0.8 mL/ min, with 5000 kPa of work pressure.

Data acquisition was performed using the ChemStation software (Agilent Technologies), acquiring chromatograms at a detection wavelength of 214 nm and the tartaric acid was identified on the basis of its retention time, compared to its standard.

Moisture content, water activity and pH

Moisture content of cooked biscuits was determined following the AOAC method (2000). For each sample five biscuits were ground, about three grams of product, exactly weighed, was placed in an oven at 105 °C until constant weight. Five replicates were performed for each sample.

The water activity values of grounded biscuits were obtained by using a dew point hygrometer, AcquaLab-Water Activity Meter (mod. SERIES 3TE. Decagon Device, Inc., Nelson Court, NE). Three replicates were obtained for each sample.

The pH value was estimated directly by using a digital pH meter (Crison, Alella, Spain) according to the standard method of the AOAC (2000). Five replicates were performed for each sample.

Color measurements

Color of biscuits surfaces was measured using a tristimulus spectrophotocolorimeter (mod. ColorFlex, HunterLab, USA). Color was determined using the CIE L*a*b* scale and illuminant D65. The evaluated parameters were lightness (L*), redness (a*) and yellowness indexes (b*).

Numerical values of a* and b* were converted into hue angle (h°) and chroma (C*) that represent the hue and the saturation index: $h° = [\arctan (b^*/a^*)/2 \quad \pi] \times 360$, $C^* = [(a^*)^2 + (b^*)^2]^{1/2}$ (Glicerina et al. 2013, 2018). Twenty biscuits were analysed for each sample.

Textural and physical characteristics

A texture analyzer TA-HDi500 (Stable Micro Systems, Surrey, UK), equipped with a HDP 3-point bending rig and a cell of 25 kg, was used to determine biscuit fracture strength. The distance of two beams was 30 mm and the

other settings were: pre-test speed of 5.00 mm s^{-1}, test speed of 1.00 mm s^{-1}, post-test speed of 10.00 mm s^{-1} and distance of 5 mm. The downward movement was advanced till the biscuits was broken. The maximum peak of the obtained curve indicates the biscuit hardness. The distance between the origin of curve till the point where the biscuit breaks represents the fracturability. If this distance is short biscuit have a high fracturability. In order to better understand the fracturability results it was expressed as 1/break point distance (Romani et al. 2012). Twenty replicates were analysed for each sample.

Other physical characteristics of biscuit samples such as diameter, thickness, spread ratio and weight were measured following the methods reported by Umesha et al. (2015). The diameter of biscuits was obtained as the average value measured on twenty biscuits for each sample using a digital caliper (CDJB15, Borletti, Italia). The same set of samples was rotated of 90° and the diameter was re-measured. Thickness was measured as average value of twenty individual biscuits for each sample. The spread ratio value was calculated by dividing the diameter by thickness data. High spread ratio value corresponds to low expansion of the biscuit after baking (Umesha et al. 2015). The average weight value of twenty individual biscuits was obtained by using a digital scale.

Soaking process and texture analysis

Biscuit characteristics after soaking were determined following the procedure reported by Sacchetti et al. (2003) and De Brier et al. (2015) with slight modifications. Five biscuits for each sample were weighed; each biscuit was placed in a beaker with 50 mL of semi-skimmed milk at 23 ± 1 °C and soaked for 30, 60, 90 and 120 s. The highest time of soaking was chosen after preliminary trials in order to avoid the disintegration of biscuit in the milk. After each soaking time, biscuits were drained on a grid for thirty s per side to remove excess of milk, then was further weighed. The increased weight was evaluated considering the weight of biscuit before and after soaking process as follows (1):

$$\text{Weight increase } \% = \left(\frac{W_f - W_i}{W_i}\right) \times 100, \quad (1)$$

W_i is the biscuit weight before soaking, and W_f is the biscuit weight after soaking.

After soaking each biscuit was measured by texture analyzer with a HDP 3-point bending rig, in order to obtain the maximum force at failure Fp (N).

The Fp textural parameter was modelled according to Peleg's model modified by Palou et al. (1994), as reported below (2):

$$\mathrm{Fp_t} = \left(\frac{Fp_0 - t}{k1 + k2t}\right), \quad (2)$$

where Fp_t is the force at failure at time t, Fp_0 is the initial force at failure at time 0, t the soaking time (s), k_1 is the Peleg's rate constant and k_2 the Peleg's capacity constant; the minus (−) sign correspond to loss hardness kinetics.

According to this model, the equilibrium force F_{pE} (i.e. when $t \to \infty$), is given by:

$$\mathrm{F_{pE}} = \left(Fp_0 + \frac{1}{k2}\right). \quad (3)$$

Similarly, the initial force rate dFp_t/dt, at t = 0 is given by $1/k_1$.

Analysis of volatile compounds

The aroma profiles of biscuits were evaluated by headspace solid phase microextraction-gas chromatography/mass spectrometry (HS-SPME-GC/MS), using a GC-MSeQP2010 Plus (Shimadzu, Tokyo, Japan) equipped with an AOC 5000 autosampler (Shimadzu, Tokyo, Japan), as already reported by Marzocchi et al. (2017). About 3 g of ground biscuit were weighed into a 10 mL amber vial, with septum and aluminium caps, and equilibrated at 40 °C for 30 min; a 2 cm × 0.11 mm (i.d.), 50/30 mm divinylbenzene/carboxen/polydimethylsiloxane (DVB/Carboxen/PDMS) SPME fiber (Supelco, Bellefonte, PA, USA) was then inserted through the septum into the vial at 40 °C for another 10 min. Afterwards, the SPME fiber was desorbed at 240 °C for 7 min in the split mode (1:10). An Rtx-Wax fused-silica capillary column (30 m × 0.25 mm i.d. × 1.0 mmf.t.) (Phenomenex, Torrance, CA, USA) was used for the chromatographic separation. The oven was programmed from 40 °C (kept for 10 min) to 200 °C at 3 °C/min and maintain it for 3 min, then increased from 200 to 240 °C at 10 °C/min and kept at the final temperature for 5 min. The injector, transfer line and the ion source temperatures were set at 240 °C, 240 °C and 200 °C respectively. Helium was used as the carrier gas at an inlet pressure and constant flow rate of 1.5 mL/min.

The filament emission current was 70 eV. A mass range from m/z 30 to 250 was scanned from 3.5 to 70 min. The acquisition was carried out in Total Ion Current (TIC) mode, using the GCMS solution software, version 2.50 SU1 (Shimadzu, Tokyo, Japan). Each extraction was carried out two times for each biscuit sample. Identification of volatile compounds was performed by comparing their mass spectra with those reported in literature and the NIST Mass Spectral Database (NIST 08, National Institute of Standards and Technology, Gaithersburg, MD, USA). The VOCs content was indicated as area counts.

Consumer testing

One hundred and ten consumers (female and male, aged from 25 to 45 years) were recruited to evaluate the four samples. Consumers were screened based on their consumption of baked goods (at least once in the last 2 weeks), and they had to be 18 years or older. They were asked to rate their preference using a 9-point hedonic scale (1 = extremely dislike; 9 = extremely like). The attributes rated were the following: surface color (from light yellow to dark brown); surface appearance (homogeneity in terms of colour and smooth top); smell (typical sweet odour associated with caramel and roasted notes); taste (typical sweet and caramel taste) and overall acceptability (considering all the previous parameters) (Kaur et al. 2015; Pasqualone et al. 2019). The test was performed in a laboratory, conducted in individual booths (ISO 8589 2007). Biscuit samples were prepared the same day of the sensory evaluation and served to the tasters in a randomized order.

Statistical analyses

Analysis of variance (ANOVA) and the test of mean comparisons according to Fisher's least significant difference (LSD) with a 0.05 level of significance were applied to find out significant differences among the different samples.

For the application of the Peleg's model on experimental data non-linear regression analysis was performed with Eq. (2) as the model function, using the Gauss–Newton algorithm for the least-square estimation of the non-linear parameters (Hartley 1961). The fitting efficiency was evaluated by the coefficient of determination (R^2) and the root mean square deviation (*RMSD*); the latter according to Eq. (3) (Engels et al. 1987):

$$\mathrm{RMSD} = \frac{1}{n} = \sqrt{\sum_{i=1}^{n}(E_i - P_i)^2}, \quad (4)$$

where n represent the number of observations, Ei are the experimental values and Pi the model predictions.

The statistical package STSG Statistica for Windows, version 6.0 (Statsoft Inc., Tulsa, OK, USA) was used.

Results and discussions

Moisture, water activity and pH

In Table 1 the moisture content, water activity and pH values of the different biscuit samples are reported. D and C biscuits showed significantly ($P < 0.05$) higher moisture (around 0.5%) and water activity values compared to A and B ones. Despite the significantly differences in moisture data between biscuits, all analysed samples showed very low moisture content, this means that the cooking time of 14 min permitted to reach a similar cooking level.

Table 1 Moisture content, water activity (a_w) and pH values of biscuits made with different type of leavening agents in formulations (A, ammonium bicarbonate; B, cream of tartar; C, grape-based; D, baking powder)

Sample	Moisture (%)	a_w	pH
A	7.535 ± 0.280b	0.566 ± 0.018 c	7.36 ± 0.05b
B	7.392 ± 0.111 b	0.566 ± 0.006 c	7.29 ± 0.07b
C	7.913 ± 0.162 a	0.587 ± 0.001 b	7.63 ± 0.10a
D	8.070 ± 0.123 a	0.603 ± 0.006 a	7.65 ± 0.12a

a–c Values followed by different letters differ significantly at $P < 0.05$ level

The highest moisture and a_w values measured after baking in C and D samples, could be due to an incomplete or slow dissolution of sodium bicarbonate probably characterized by different purity, granulometry and as reported in Table 1 by higher pH than in A and B ones. As reported by Li et al. (2016), higher the pH of sodium bicarbonate solution, as in C and D samples, lower will be the salt dissolution, that in this form tends to hold more water (Ozdemir et al. 2007). The sodium bicarbonate present in the B powder probably underwent a fast dissolution and a complete reaction (Lanza 2006). Moreover, the lower moisture and water activity values also observed in A sample can be reasonably attributed to the complete dissolution of ammonium bicarbonate (Manley 2001).

Color measurements

As reported in Supplementary Figure S1, D and C samples showed the lower lightness (L*) and hue angle (h°) but higher b* values than biscuits made with cream of tartar (B) and ammonium bicarbonate (A).

The less lightness and a redder and less yellow hue of D and C biscuits could be a consequence of an uncompleted dissolution of leavening agents, that probably contributed to increase the pH of matrices, as previous observed in Table 1, promoting Maillard reaction during baking (Manley 2001; Konings et al. 2007). C and D samples showed in fact orange spots on their surface after cooking (data not shown), that according with literature (Manley 2001) are also related to a poor dissolution of sodium bicarbonate and high pH values (Gökmen et al. 2008). A and B samples did not show any spot in their surfaces, probably due to a fast dissolution and complete reaction of the salts in related leavening powders, as previous stated (Lanza 2006).

Considering a* index and Chroma results these parameters were not able to well discriminate biscuit's color as the others.

Textural and physical characteristics

In Table 2 the values of textural and physical characteristics (diameter, thickness, spread ratio and weight) measured on the different samples are reported.

B and D samples showed the significantly ($P < 0.05$) highest hardness values, followed by C biscuit. The A sample had the lowest hardness and fracturability values, indeed, the internal structure of this biscuit was particularly spread than the others, that had higher and significantly similar fracturability values.

Concerning the others physical properties, sample A showed a significantly ($P < 0.05$) higher diameter compared to the others; whereas B, C and D had similar diameter values. Moreover, A biscuit showed the significantly highest value of thickness, the lowest weight and spread ratio values. According with Umesha et al. (2015), the lowest value of spread ratio the highest spread of the product. These results confirm hardness and fracturability ones obtained for A. The peculiar physical characteristic values of the A sample can be ascribable to ammonium bicarbonate. This baking agent decomposes during baking in CO_2 and ammonium, that contribute to promote a high spread and development of the internal structure of biscuits (Manley 2001; Vetter 2003). The C biscuit showed the significantly highest spread ratio value and the lowest thickness one. B and D samples presented physical characteristics similar each other. Cross section photos of biscuits are given in Supplementary Fig. S2.

Soaking process and texture analysis

As shown in Fig. 1, all biscuit samples underwent a characteristic weight increase behaviour, due to milk uptake during soaking, with an initial faster rate, followed by a decrease of the adsorption rate only in B, C and D ones, with increasing soaking time. The trend of weight increase in biscuit A was significantly different from the others, this sample in fact adsorbed the highest amount of milk for the entire soaking time period, reaching, after 120 s around 115% of its initial weight. Moreover, during drip process biscuits B, C and D held a film of milk on their surface, while in A the milk was at each soaking time completely absorbed. This confirm, as visually observed, that the internal structure of A sample was characterized by a more homogeneous structure in terms of pore size, predominantly smaller than in the other samples, as also reported in the case of waffle made with ammonium bicarbonate (Demirkesen et al. 2014; Huber and Schoenlechner 2017). Small pores (Karathanos and Saravacos 1993) are characterized by high specific surface area, that consequently causes a greater milk uptake. Moreover, in the presence of small pores the diffusion of fluids, in this case milk, occurs within the solid matrix rather than their outside and remaining entrapped promotes a softening effect and so a reduction of the hardness (Sacchetti et al. 2003; Demirkesen et al. 2014). Studies demonstrate in fact, that bakery products with lower number of pores having higher size were characterized by higher hardness values (Demirkesen et al. 2014).

Highlighting the hardness modification of samples during soaking, the maximum forces reached at each soaking time (Fp) were evaluated and data modelled. The applied Peleg's equation showed a good fit of the experimental data, as confirmed by high R^2 values and low $RMSD <$ 0.50% (Table 3), underlying its suitability in describing texture decrease phenomena, as also reported by Sacchetti et al. (2003).

As showed in Fig. 2 milk uptake produced the loss of the typical crunchy texture in all samples, that was normally reflected by the shape change of the typical force–displacement curve obtained by textural analysis after the different immersion times. B and C samples maintained the highest Fp values during the whole soaking period (Fig. 2), A biscuit the lowest. As reported in Table 3, $1/k_1$ Peleg constant results, referred to the initial rate of hardness loss

Table 2 Textural and physical characteristics of biscuit samples made with different type of baking agents (A, ammonium bicarbonate; B, cream of tartar; C grape-based; D, baking powder)

Sample	Hardness (N)	Fracturability (1/mm)	Diameter (mm)	Thickness (mm)	Weight (g)	Spread ratio[A]
A	26.281 ± 0.003c	0.561 ± 0.179b	44.253 ± 1.269a	12.359 ± 0.634a	6.728 ± 0.601c	3.591 ± 0.168c
B	57.744 ± 0.011a	0.841 ± 0.412a	42.211 ± 0.831b	10.138 ± 0.827b	7.501 ± 0.428a	4.189 ± 0.336b
C	46.892 ± 0.013b	0.994 ± 0.271a	42.060 ± 0.849b	9.500 ± 0.550c	7.323 ± 0.490a	4.412 ± 0.491a
D	55.711 ± 0.011a	0.812 ± 0.250a	42.252 ± 1.310b	10.401 ± 0.651b	7.069 ± 0.441b	4.071 ± 0.242b

a–d Values followed by different letters differ significantly at $P < 0.05$ level

[A]Diameter (mm)/thickness (mm) of biscuit

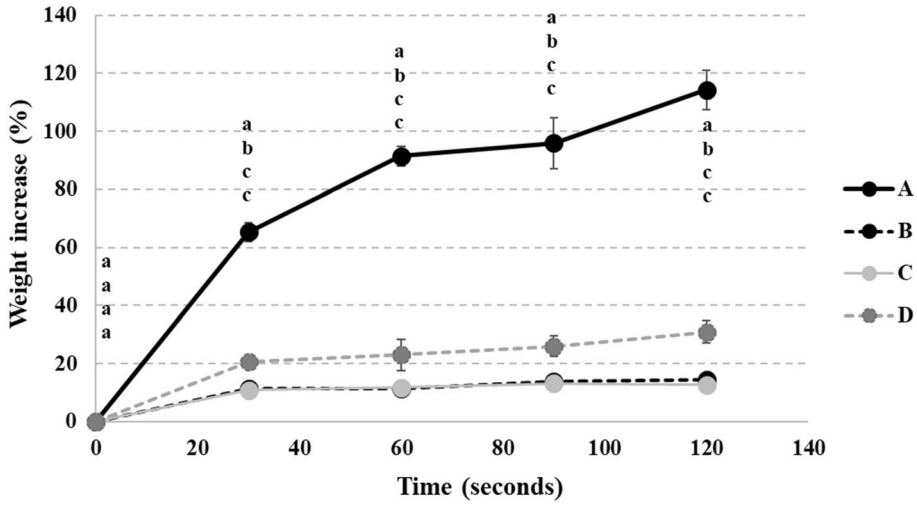

Fig. 1 Weight increase (%) of biscuits samples during soaking time in semi-skimmed milk made with different type of baking agents (A, ammonium bicarbonate; B, cream of tartar; C, grape-based; D, baking powder). a–c Values followed by different letters within the samples differ significantly at $P < 0.05$ level

Table 3 Average Peleg constants ($1/k_1$–$1/k_2$) of hardness loss of biscuit samples during soaking; significance (*p*), root mean square deviation (RMSD %), goodness of fit of Peleg model (R^2) and hardness loss (%) measured at 30 s of soaking (ΔFp) (A, ammonium bicarbonate; B, cream of tartar; C grape-based; D, baking powder)

Samples	$1/k_1$	$1/k_2$	*p*	RMSD (%)	R^2	ΔFp (%)
A	25.645	41.667	0.001	0.320	0.999	90.441
B	4.542	30.303	0.002	0.340	0.999	54.562
C	5.881	32.258	0.003	0.280	0.999	58.764
D	16.670	35.714	0.002	0.300	0.999	68.687

($N\ s^{-1}$), confirm that sample A underwent the highest hardness decrease respect to B and C that presented the lowest $1/k_1$ values. D biscuit exhibited an intermediate behaviour. Moreover, sample A showed the highest hardness loss (ΔFp %) during the first 30 s of soaking (around 90%) (Table 3); samples B and C showed a similar trend between them, with the less ΔFp values (of about 55%). Also, for what concern $1/k_2$ constant rate sample A showed the highest value.

Analysis of volatile compounds

In Table 4 the aroma compounds detected in biscuit were reported.

The aroma development in bakery products can be attributed to numerous volatile compounds, strictly related to the product formulation and manufacture process (Romani and Rodriguez-Estrada 2016).

Concerning alcohol amount 1-propanol was detected only in sample C. This compound can be probably attribute to the leavening agent composition of this sample, made up

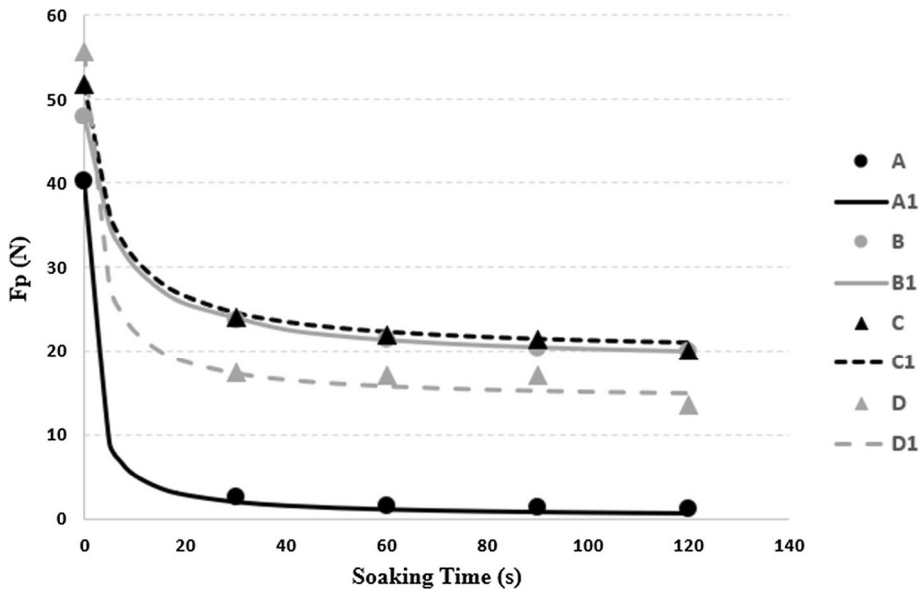

Fig. 2 Fitting of the Peleg model to peak force at failure (Fp) of biscuits made with different type of baking agents (A, ammonium bicarbonate; B, cream of tartar; C grape-based; D, baking powder), in which symbols represent experimental data and lines the fitted ones

Table 4 Volatile compounds obtained by SPME–GC–MS of the biscuit samples made with different type of baking agents (A, ammonium bicarbonate; B, cream of tartar; C grape-based; D, baking powders)

Compounds	A	B	C	D
Alcohols				
1-Propanol	nd	nd	3.5 ± 0.9a	nd
Aldehydes				
2-Methylbutanal	nd	8.1 ± 0.3a	3.9 ± 1.1b	2.8 ± 0.0b
3-Methylbutanal	nd	14.6 ± 0.4a	8.2 ± 1.5b	5.7 ± 0.0b
Hexanal	19.8 ± 1.8a	9.0 ± 1.1c	11.3 ± 0.9b,c	15.4 ± 1.0a,b
Nonanal	3.7 ± 0.3a	nd	nd	nd
Benzaldehyde	2.9 ± 0.4a	nd	nd	nd
Ketones				
2-Butanone	nd	2.1 ± 0.2a	nd	nd
2-Pentanone	nd	2.9 ± 0.2a	nd	nd
2,3-Butanedione	nd	4.6 ± 0.4a	nd	3.7 ± 0.4a
2-Heptanone	nd	14.4 ± 1.6a	12.0 ± 0.9a	14.0 ± 0.1a
3-Hydroxy-2-butanone	nd	4.6 ± 0.3a	4.0 ± 0.2a	4.0 ± 0.1a
1-Hydroxy-2-propanone	6.8 ± 0.5d	138.5 ± 0.2b	102.1 ± 5.9c	155.5 ± 4.0a
2-Nonanone	1.1 ± 0.1b	nd	4.4 ± 0.1a	4.5 ± 0.3a
Furan compounds				
2-Furanmethanol	2.3 ± 0.0c	4.5 ± 0.5a, b	3.6 ± 0.9b,c	6.1 ± 0.1a
Pyrazines				
2-Methylpyrazine	4.9 ± 0.4a	1.6 ± 0.1b	nd	nd
Sulfur compounds				
Benzothiazole	23.5 ± 5.5a	32.0 ± 5.5a	34.9 ± 0.6a	25.5 ± 3.9a

Areas (area × 10^4) are expressed as mean ± standard deviation (n = 2)

a–d In each raw, values followed by different letters differ significantly at $P < 0.05$ level

nd non detected

of a 42% of grape juice extract (Moreira et al. 2011). Regarding aldehyde content, A sample showed a different pattern than the other biscuits: in this sample were detected nonanal and benzaldehyde, hexanal was present at significantly higher level whereas, the Stracker aldehydes (2-methylbutanal and 3-methylbutanal) were absent. According to literature, aldehyde profile can be related with the intensity and duration of kneading, but as well with the intensity of the chemical reaction during leavening (Cayot 2007). The ammonium bicarbonate used in sample A, probably react very quickly with the other ingredients giving arise to a high amount of hexanal. Moreover, rapid chemical reactions are also related to low concentration or the absence of the Stracker aldehydes (2- and -3 methylbutanal), that are also responsible for malty notes (Pasqualone et al. 2015). These two compounds were instead present at the highest concentration in biscuit B. Different ketones were detected in biscuit samples and some of them derived from Maillard reaction (2-butanone, 2,3-butanedione and 1-hydroxy- 2-propanone). Except in A biscuit, 1-hydroxy-2-propanone, that provide malty and roasted notes, was the compound found at the highest level in all biscuit samples and in particular in D one. This result seems in agreement with those obtained for color in C and D samples. Except for 2-nonanone, all considered ketones were present in sample B.

Other volatiles, associated also to Maillard reaction, were found: the 2-furanmethanol, a furan compound associated to sweet and caramel notes; the 2-methylpyrazine, a pyrazine compound presents only in A and B biscuits and, finally, the benzothiazole, a sulfur compounds normally found in all biscuit (Pasqualone et al. 2015).

It seems that each volatile profile detected in the different biscuit is related other than to the Maillard reaction also to the different leaving powders used.

Consumer testing

In Supplementary Table S1, test results are reported. The A sample reached the highest score values for what concern surface appearance. A and D samples presented the highest overall acceptability, reaching also the highest score for smell attribute. B and C samples, obtained with leavening powder partially derived from grape, were instead perceived with the significantly less scores, for overall acceptability and taste, even if, for this last parameter not

significantly different from A. Moreover, D and C samples reached the lowest and similar scores for surface appearance, that was judged not very homogeneous. From biscuit acceptance testing results, it seems that not all the differences observed in their volatile profile, were perceived by consumers. This could indicate that some aroma compounds, detected during the volatile analysis, are not perceived after biscuits cooling, limiting the discriminating ability of the test.

Nevertheless, A and D samples reached the highest score values for smell and acceptability, probably due the highest percentage of 2-methylpirazine in A, compound normally associated with nutty and roasted notes and the highest level of 1-hydroxy-2-propanone and 2-furanmethanol in D, two volatile compounds derived from Maillard reaction and responsible for malty, roasted and sweet notes (Pasqualone et al. 2019).

Conclusion

The overall obtained results confirm that different leavening powders in formulation can influence the final quality of biscuits mainly in terms of textural, physical and organoleptic characteristics.

In particular, ammonium bicarbonate tends to impart a very homogeneous surface appearance and a less hard texture with high fracturability and spread. For these peculiar physical characteristics, biscuits obtained with ammonium bicarbonate can be considered "dunking biscuits" more than the others, as demonstrated.

The presence in formulation of baking powders made with ammonium bicarbonate (A) and cream of tartar (B) influenced the biscuit volatile profiles. B and C samples made up from tartaric acid, were the biscuits less appreciated from panellists.

Biscuit D obtained with the most common baking powder (diphosphates powder) showed in general intermediate characteristics between A and B–C samples.

Results of this study demonstrate that changing the type of leavening agent in biscuit formulations it is possible to greatly influence some important quality characteristics of the final product, mainly the structural ones.

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