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Influence of baking conditions and formulation on furanic derivatives, 3-methylbutanal and hexanal and other quality characteristics of lab-made and commercial biscuits

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A B S T R A C T

Biscuit baking can cause the formation of heat-related toxic compounds, mainly through the Maillard reaction, including some volatile organic compounds (VOCs) that are potentially carcinogenic to humans.

This study investigates the effects of different baking conditions and recipes on quality characteristics (moisture, water activity, colour, texture) and on the concentration of some VOCs (furfural, furfuryl acetate, 5-methylfurfural, furfuryl alcohol, 3-methylbutanal, hexanal) in biscuits. Specifically, lab-made biscuits baked under static and ventilated conditions and three commercial biscuit types categorised as shortbreads with eggs, with chocolate chips and dry petits were evaluated.

Concerning the lab-made biscuits, the ventilated mode resulted in faster baking and a slightly lower concentration of investigated VOCs compared to the static mode. Besides the process conditions, the recipe also played a role in the final quality and target volatiles, whose concentrations were lower in dry petits than in shortbreads, which are characterised by higher sugar and fat contents.

1. Introduction

Biscuits are among the most consumed bakery products in the world thanks to their pleasant sensory properties and long shelf-life (Romani & Rodriguez-Estrada, 2016). Their global consumption has increased by 31.6% during the COVID-19 outbreak (Cookie Report—TOP Agency, 2022); therefore, the importance of studying the overall quality and safety characteristics of these products is constantly increasing.

Even though the preparation of biscuits is quite simple, as it consists of mixing the ingredients, shaping, baking and packaging (Drakos et al., 2019; Konstantas et al., 2019), several complex physical and chemical mechanisms occur during the baking process, including heat and mass transfers and non-enzymatic browning reactions (Devu et al., 2022; Shahrbabki et al., 2018; Sruthi et al., 2021). These phenomena are responsible of volume expansion, water evaporation, texture development, surface browning and organoleptic characteristics of the final products. The extent of these physico-chemical occurrences depends on many process variables including formulation, time–temperature conditions and heat transfer mode.

The most important non-enzymatic reactions are the Maillard reactions, which take place during the baking step and mainly contribute to the typical sensory profile of the biscuits, but can also lead to the formation of some compounds that can be toxic to humans (ALjahdali & Carbonero, 2017). These include the furanic compounds, a class of heterocyclic organic compounds with five-membered-rings. They can be present in various beverages and food, such as coffee, fruit juices, baby foods, breakfast cereals, crackers, crispbreads, biscuits etc. and are responsible for the typical flavour of the final products as they have a high volatility (Kettlitz et al., 2019). Given the potential toxicity to human health of these compounds, the interest in studying this class of molecules is steadily increasing. Indeed, in 1995, furan was classified as Group 2B by the International Agency for Research on Cancer (IARC) and defined as “possibly carcinogenic to humans, based on sufficient evidence in experimental animals” (IARC, 1995). Moreover, two of the

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most studied furanic derivatives, 5-hydroxymethylfurfural (HMF) and furfuryl alcohol (FFA), have shown to be responsible for toxic effects on living organisms because of their conversion to 2-sulfoxymethylfuran and 5-sulfoxymethylfurfural, respectively, by sulfotransferases (SULT). These derivatives can react with DNA or proteins of the human body and cause mutations (Petisca et al., 2013; Shen et al., 2016). FFA, in fact, has been classified as Group 2B by IARC and defined as possibly carcinogenic to humans based on sufficient evidence of carcinogenicity in experimental animals and no data or inadequate evidence in human beings (IARC, 2019).

Data on population exposure to furan and its derivatives have prompted the researchers to focus their attention on the presence of these compounds in several food (Rahn & Yeretzian, 2019). Although for furanic compounds, unlike other toxicants such as acrylamide, there are no specific reference values to be respected in food, it is important to keep their concentration As Low as Reasonably Achievable (ALARA concept) (Barrios-Rodríguez et al., 2022). In addition to toxic furanic compounds, other important volatile organic compounds (VOCs) can be formed during baking, including 3-methylbutanal and hexanal. The first compound imparts a malty note, is a product of the lipid oxidation and can be considered a potential volatile oxidation marker for food products (Cardenia et al., 2015). Hexanal is the product of lipid oxidation and is responsible for the rancid taste. It is often used as a marker for secondary lipid oxidation (Purcaro et al., 2008) and as a food quality parameter for biscuits (Romani et al., 2015).

The aim of this study was to investigate: i) the influence of different baking conditions on some quality attributes (moisture, water activity, colour and texture), and on the concentration of some VOCs (furfural, furfuryl acetate, 5-methylfurfural, furfuryl alcohol, 3-methylbutanal, hexanal) in lab-made biscuits during baking; ii) the influence of the formulation of three types of commercial biscuits (shortbread with eggs, shortbread with chocolate chips and dry petits) on the above-mentioned quality attributes and investigated VOCs concentrations. This in order to investigate whether baking conditions and/or formulation have a greater influence on the formation of these compounds.

2. Materials and methods

2.1. Biscuit samples

2.1.1. Experimental biscuits

Experimental shortbread with eggs were prepared in lab using the formulation and preparation described by Schouten et al. (2022) with 500 g of wheat flour, 125 g of sucrose, 125 g of pasteurized eggs, 100 g of butter, 100 g of milk and 15 g of leavening agent containing sodium diphosphate and carbonates. All the ingredients were purchased from the local market (Cesena, FC, Italy). All ingredients were mixed and kneaded using a household blender (mod. Bimby Robot TM31, Vorwerk, Wuppertal, Germany) at speed position 5 (approx. 3000 rpm) for 1 min and 30 s. After 80 s, the speed control was reversed to give the walls of the kneading bowl time to clean. The dough was then kneaded by hand for about 30 s. The biscuits dough had a moisture content of 24.50 ± 0.04% and an a_w value of 0.92 ± 0.01. The dough was left to rest in the refrigerator at 4 °C for 20 min and rolled out to a thickness of 3.0 mm using a dough sheeter (mod. SFSI 42040050 T, GAM International, Santarcangelo di Romagna, Italy). The raw biscuits were obtained using a round stainless-steel mould with a diameter of 6 cm and placed on a baking tray covered with baking paper. The biscuits were baked in static mode (A) and in ventilate mode (B) in an electric oven (Procombi Plus, AEG-Electrolux, Berlin, Germany) at 175 °C for 18, 20, 22, 24 and 26 min from underbaked to overbaked levels according to preliminary tests. For each baking method and time, 3 batches of 10 biscuits were produced (in total 30 biscuits per time for each baking mode). After cooling at room temperature, a portion of each sample was evaluated for moisture, water activity, colour and texture while a portion was finely ground and stored under vacuum in the dark for subsequent chemical analysis.

2.1.2. Commercial biscuits

Commercial biscuits categorised by three different types, such as shortbread with eggs, shortbread with chocolate chips and dry petit biscuits, were purchased on the local market (Cesena, FC, Italy). Three different brands were analysed for each biscuit category, the samples characteristics, as reported in the labels, and abbreviation codes are reported in Table 1. For each brand and biscuit type, 30 biscuits were evaluated taking 10 biscuits from three different packages belonging to the same production batch. A portion of samples was evaluated for moisture, water activity, colour and texture while a portion was finely ground and stored under vacuum in the dark for subsequent chemical analysis.

2.2. Quality attributes determinations

2.2.1. Moisture and water activity

The moisture content (%) and water activity (a_w) of experimental and commercial biscuits were evaluated on ground samples. Moisture content was determined gravimetrically by placing 3 g of the sample in an oven at 105 °C until the weight remained constant. The a_w was

Table 1
Category, sample code and labelled ingredients of the three types of commercial biscuit samples each belonging to three different brands.

Biscuit category	Sample code	Labelled ingredients
Shortbreads with Eggs	SE1	Wheat flour, sugar, sunflower oil, fresh eggs, raising agents (ammonium hydrogen carbonate, sodium hydrogen carbonate monopotassium tartrate), milk, salt, honey, milk protein, glucose-fructose syrup
	SE2	Wheat flour, sugar, sunflower oil, fresh eggs, raising agents (sodium hydrogen carbonate, ammonium hydrogen carbonate), milk, salt, milk protein, honey
	SE3	Wheat flour, sugar, sunflower oil, fresh eggs, glucose syrup, raising agents (sodium hydrogen carbonate, ammonium hydrogen carbonate), milk, salt, milk protein, honey
Shortbreads with Chocolate chips	SC1	Wheat flour, sugar, chocolate chips (cocoa paste, sugar, dextrose, soya lecithin), maize oil, butter, glucose-fructose syrup, raising agents (sodium hydrogen carbonate, ammonium hydrogen carbonate), wheat starch, dextrose, salt, sunflower lecithin, flavourings
	SC2	Wheat flour, sugar, chocolate chips (sugar, cocoa paste, low-fat cocoa powder, cocoa butter, soya lecithin), sunflower oil, butter, glucose-fructose syrup, wheat starch, raising agents (sodium hydrogen carbonate, ammonium hydrogen carbonate), salt, vanillin flavouring
	SC3	Wheat flour, sugar, chocolate chips (sugar, cocoa paste, low-fat cocoa powder, cocoa butter, soya lecithin), sunflower oil, butter, glucose-fructose syrup, raising agents (ammonium hydrogen carbonate, sodium hydrogen carbonate), salt, flavourings
Dry Petit	DP1	Wheat flour, sugar, maize oil, wheat starch, milk powder, glucose-fructose syrup, barley and maize malt extract, fresh eggs, raising agents (ammonium hydrogen carbonate, sodium hydrogen carbonate, monopotassium tartrate), salt, sunflower lecithin, flavourings
	DP2	Wheat flour, sugar, sunflower oil, glucose syrup, milk powder, raising agents (ammonium hydrogen carbonate, sodium hydrogen carbonate), dextrose, salt, flavourings
	DP3	Wheat flour, sugar, sunflower oil, raising agents (ammonium carbonate, sodium carbonate, potassium tartrate), wheat starch, salt, flavourings

determined using an AQUALAB dew point hygrometer (Meter 4TE, Pullman, Washington, USA) at 25 °C. Three replicates per sample were performed.

2.2.2. Colour and visual appearance

The colour of experimental and commercial biscuits was measured using a Colorflex Tristimulus Spectrophotocolorimeter (HunterLab, Sunset Hills Road Reston, Virginia, USA), illuminant D65 (6500 K) and geometry 45°/0°. Before each set of measurements, the instrument was properly calibrated with a standard white ceramic tile and a black glass. The colour was given in the standard scale CIE L* (lightness), a* (green–red) and b* (blue-yellow) and the measurements were made on the top side of 5 biscuits for each sample.

To capture the visual/colour appearance of experimental and commercial biscuits a computer vision system (CVS) was used. The samples were placed inside a dark chamber over a black background in controlled lighting conditions with four daylight fluorescent lamps (TL-D Deluxe, Natural Daylight, 18 W/965, Philips, USA) with a colour temperature of 6500 K. The RGB images of the samples were acquired using a colour digital camera (D7000, Nikon, Japan) equipped with 105 mm lens (AF-S Micro Nikkor, Nikon, Japan) and positioned vertically.

2.2.3. Texture and thickness

Texture measurement on experimental and commercial biscuits was carried out at room temperature using the TA -HDi500 texture analyser (Stable Micro System, Surrey, UK) equipped with a three-points bending ring, a horizontal stainless-steel probe and a 25 kg load cell. The pre-test speed was 5.00 mm/s, the test speed was 1.00 mm/s, the post-test speed was 10.00 mm/s, the probe distance 5 mm and the distance of the two beams was 30 mm. The recorded parameters were expressed as hardness (N), which was calculated from the maximum force values, crispness index, which was calculated from the linear distance between the first and the last recorded peak value.

As the commercial biscuit samples had different thicknesses (mm), this parameter was evaluated using a digital vernier calliper (CDJB15, Borletti, Italy).

Force-distance curves and thickness were obtained from 5 biscuits for each sample.

2.3. VOCs determination

2.3.1. Standards and chemicals

Pure standards of 3-methylbutanal (CAS 590-86-3), hexanal (CAS 66-25-1), furfural (CAS 98-01-1), furfuryl acetate (CAS 623-17-6), 5-methylfurfural (CAS 620-02-0), furfuryl alcohol (CAS 90-00-0), 2-methylpentanal (CAS 123-15-9) and HPLC-grade ethanol were purchased from Sigma Aldrich (Milan, Italy). The stock solutions of target volatile compounds were prepared by mixing 10 mg of pure standards with 10 mL of HPLC-grade ethanol (1000 $\mu g \cdot mL^{-1}$). Standard working solutions at different concentration were prepared by diluting the stock solutions in ultrapure water. The ultra-pure water was obtained through Milli-Q SP Reagent Water System (Millipore, Bedford, MA, USA). 2-Methylpentanal were used as Internal Standard (IS) and it was added in each standard solution at a concentration of 100 $\mu g \cdot mL^{-1}$. The calibration curve was prepared by plotting the standard solution concentrations by the respective Response Factor (RF). RF is the ratio between the peak area of analyte and the peak area of IS.

2.3.2. Headspace solid phase microextraction (HS-SPME)

The analysis of furanic compounds was performed following a procedure previously developed by Acquaticci et al. (2023) and quantified through a headspace-solid phase microextraction-gas chromatography-mass spectrometry (HS-SPME-GC–MS). Briefly, immediately after the packaging opening 1 g of sample was then weighted into a 20 mL vial and rapidly the internal standard 100 $\mu g \cdot mL^{-1}$ was added into. Immediately after the vial was sealed with a screw cap with a PTFE septum. The analysis has been performed by using a PAL RSI 85 autosampler (Zwingen, Switzerland) which was able to yield strong analysis repeatability. The incubation of the sample was performed at 60 °C for 20 min under agitation (250 rpm, 5 s of on-time and 2 s of off-time). The divinybenzene/carbon-wide range/polydimethylsiloxane fibre (DVB/C-WR/PDMS) from Supelco (Bellefonte, PA, USA) was selected for this work.

The fibre was conditioning for 20 min at 250 °C and then it was inserted inside the headspace of sample vial with a speed of 20 $mm \cdot s^{-1}$ and a penetration depth of 40 mm. The extraction was performed at 60 °C for 40 min and then the fibre was inserted into injector port at a speed of 100 $mm \cdot s^{-1}$ and a penetration depth of 40 mm. The desorption occurred at 250 °C for 3 min. After desorption, the fibre was conditioning at 250 °C for 15 min.

2.3.3. GC–MS analysis

The GC–MS analysis was carried out by a 7890B gas chromatograph from Agilent equipped with a PAL RSI 85 autosampler and a 5977B mass spectrometer Agilent (Santa Clara, California, USA). The ionization source was a high efficiency electron ionization source (EI).

The injector temperature was set at 250 °C and the liner used was recommended for SPME injection namely Inlet liner, Ultra Inert, splitless, straight, 0.75 mm id, (5190–4048) from Agilent. The gas carrier was helium at a flow rate of 1 $mL \cdot min^{-1}$. The separation of target molecules was established on a DB-WAX capillary column (60 m, 250 μm i.d., 0.25 μm film thickness) with this ramp of temperature: 35 °C held for 2 min, 35–240 °C at 8 °C/min and 240 °C held for 3 min. The run time was about 32 min.

The transfer line was set at 250 °C and the temperature of the ionization source and the mass analyser were set at 230 and 150 °C, respectively. The gain factor was set at 0.1 that corresponded at 1413 V.

The ion species optimization for compounds were carried out by injecting a standard solution (10 $\mu g \cdot mL^{-1}$) in SCAN mode (35–450 *m/z*). The identification of compounds was performed by comparison with NIST library (US National Institute of Standards and Technology). The acquisitions were carried out in 'Selected Ion Monitoring' (SIM) mode and detection was divided into time windows. The most abundant ions were used for quantitation while the other to confirm the presence of the analytes. The GC–MS parameters including the retention time (Rt) and time windows are reported in **Table S1** (in **Supplementary Materials**). Data results have been managed by MSD ChemStation Software (Agilent, Version G1701DA D.01.00). Samples were analysed in triplicate. Acceptable relative standard deviation (%RSD) were set up below 20%.

2.3.4. HS-SPME-GC–MS analytical method optimization and validation

A new HS-SPME-GC–MS analytical method for the quantification of six VOCs was developed, optimized and validated. Three fibres, four types of salt, three incubation times and temperatures and three extraction times were evaluated. Results showed that 1 g of sample, without the addition of salts, incubated for 15 min at 60 °C and extracted for 30 min with a DVB/C-WR/PDMS fibre were the best conditions for the analysis. **Table S2** (in **Supplementary Materials**) showed the parameters optimized. The chromatographic separation was good for all peaks and **Fig. S1** (in **Supplementary Materials**) showed an example of a chromatogram of a standard mixture of the six VOCs quantified with this method.

This new method was validated by studying linearity, sensitivity and repeatability (**Table S3** in **Supplementary Materials**). Linearity was studied by injecting five different concentrations of the six VOCs and plotting the calibration curves with the respective determination coefficients (R^2). All compounds showed a good linearity as the R^2 was equal to or greater than 0.990. Repeatability was expressed as relative standard deviation (%RSD) and intra-day repeatability was assessed by injecting 5 replicates of mix standards five times in a day while inter-day repeatability was 3 replicates of mix standards once a day for three days. The intra-day repeatability ranged from 3.9 to 18.3% while inter-day repeatability was 5.4–19.9% for all target volatile compounds.

2.4. Statistical analysis

All the obtained results were reported as average value ± standard deviation. Significant differences between data were calculated by unidirectional analysis of variance (ANOVA) followed by Tukey's post-hoc comparison test, with a significance level of $p < 0.05$. The statistical package STATISTICA 8.0 software (StatSoft, Tulsa, UK) was used.

3. Results

3.1. Main quality attributes of biscuit samples

The results of quality attributes, measured in experimental biscuits formulated with a traditional shortbread recipe and baked for different times at static and ventilate modes, are listed in Table 2.

Moisture content of biscuits and bakery goods is a very important parameter that characterises different products, influences the behaviour of chemical reactions during baking (including VOCs formation) and the shelf-life of the final products during storage (Faridi, 1994; Mandala et al., 2006). The moisture content of all experimental biscuits baked under different conditions ranged from 8.0 to 2.7% from the lowest to the longest baking time. After 22 min baking, all samples reached a moisture content of less than 5% within the standard range of commercial biscuits. As expected, the baking process promoted a dehydration of the biscuits, however, the samples baked in static mode (A) for 20 and 22 min had significantly higher moisture and a_w values than those baked in ventilated one (B). This is because in static mode, heat is generated by electrical resistance on the top and bottom of the oven chamber (natural convection), whereas in ventilate mode, heat is generated by a fan and distributed by forced convection, heating the chamber faster and more evenly (Marcotte, 2007; Sakin et al., 2009; Walker, 2016). However, after 24 and 26 min of baking no significant moisture and a_w differences between the two heat-transfer methods were detected.

Another quality parameter that characterises the biscuit product and is also related to different baking process conditions is its surface colour (Manley, 2011). For the same baking time, the two heat-transfer modes favoured, as expected, the development of different colour characteristics on the biscuits top surface. When comparing the L* values, the samples baked in the ventilate mode consistently showed a significantly darker colour than those baked in the static one. For both experimental biscuits, the colour parameter a* increased significantly as baking proceeded. However, for the A biscuits, the a* values increased more slowly compared to the B ones until 22 min. These differences in the colour parameters are again due to the different heat distribution in the oven for the two baking methods, as reported above (Schouten et al., 2022). The differences found in biscuit samples colour baked at different conditions were also perceptible from the visual appearance as shown in **Fig. S2(a)** (in **Supplementary Materials**).

Hardness and crispness are useful structural properties in the assessment of biscuits quality and sensorial acceptability and are also associated with the perception of freshness (Karaoğlu & Kotancilar, 2009; Lara et al., 2011). The hardness and crispiness of the experimental biscuits increased in all samples with increasing baking time, which can be attributed to the reduction in moisture content. At all baking times, except for hardness at 22 and 24 min and crispness at 26 min, the A biscuits had significantly lower texture parameters than the B samples. According to Palazoğlu et al. (2015) and Schouten et al. (2022) the hot air flow circulating in the oven accelerates the formation of the crust on the surface mainly when the oven was set in convection mode.

Based on the overall main quality attributes results, the optimal baking times for biscuits baked in static and ventilate heat-transfer modes were 22 min and 20 min, respectively.

The results of moisture, a_w, texture and colour of the different tested commercial biscuits are reported in Table 3.

The moisture content of the different commercial biscuit samples ranged from 0.8 to 3.2% within the standard range of this type of bakery product. DP samples, as expected, had significantly lower moisture content than shortbread types (SE and SC). Some significant differences were found between the moisture data of same type of biscuit belonging to different brands. The greatest differences were found in the dry petit biscuits, where sample DP1 had a significantly lower moisture content than analogues DP2 and DP3. This result can be attributed to the emulsifier lecithin, which was only used in the formulation of sample DP1. Shortbreads with eggs (SE1, SE2, SE3) and shortbreads with chocolate chips (SC1, SC2, SC3) presented on the average similar moisture values of 2.6 ± 0.3% and 2.8 ± 0.4% respectively. The differences between shortbreads and DP could also be attributed to the different baking conditions. Indeed, it is known that industrially produced shortbread biscuits require baking conditions characterised by low temperatures and long baking times, while petit dry biscuits require higher baking temperatures and shorter baking times to obtain their characteristic final moisture content (Manley, 2011). As expected, the recorded a_w values were proportional and consistent with the moisture ones measured on the same samples. Thus, the DP biscuit category had a significantly lower average a_w of 0.14 ± 0.05 than the other biscuit types (0.22 ± 0.03 for SE and 0.23 ± 0.03 for SC). The differences in moisture and a_w results can be attributed to the different ingredients used in shortbreads and DP biscuit formulations (Pittia & Paparella, 2016). For example, the presence of starch, milk proteins and hydrocolloids limit the availability and migration of water as they can act as 'binders' (water holding or water binding capacity). Comparing the labelled ingredients, shortbreads biscuits had in general a richer formulation than DP ones required to achieve their typical final organoleptic characteristics.

Concerning the surface colour of the biscuits, all commercial samples

Table 2
Main quality attributes of experimental dough and biscuits baked in static (A) and ventilate (B) modes for 18, 20, 22, 24, 26 min.

Biscuit sample	Moisture (%)	Water activity (a_w)	L*	a*	Hardness (N)	Crispness (linear distance)
Raw dough	24.5 ± 0.0 ^{A}	0.92 ± 0.01 ^{A}	80.9 ± 0.3 ^{A}	4.2 ± 0.7 ^{E}	–	–
A18	8.0 ± 0.2 $^{a, B}$	0.55 ± 0.01 $^{a, B}$	77.0 ± 0.8 $^{a, B}$	8.9 ± 0.6 $^{f, D}$	45.0 ± 3.1 $^{e, F}$	59.1 ± 2.3 $^{e, G}$
A20	5.8 ± 0.2 $^{b, C}$	0.48 ± 0.02 $^{b, C}$	74.7 ± 0.8 $^{b, C}$	10.3 ± 0.6 $^{e, C}$	46.7 ± 2.4 $^{e, F}$	80.2 ± 3.0 $^{d, E}$
A22	4.5 ± 0.1 $^{c, E}$	0.38 ± 0.03 $^{c, E}$	71.4 ± 0.8 $^{c, D}$	12.1 ± 0.5 $^{d, B}$	65.0 ± 3.0 $^{d, C}$	106.3 ± 4.1 $^{c, D}$
A24	3.7 ± 0.0 $^{d, G}$	0.26 ± 0.02 $^{d, G}$	70.0 ± 0.7 $^{d, D}$	12.8 ± 0.4 $^{bc, B}$	66.6 ± 2.5 $^{bc, C}$	127.1 ± 3.1 $^{b, C}$
A26	2.8 ± 0.1 $^{e, H}$	0.23 ± 0.01 $^{e, H}$	68.0 ± 0.5 $^{e, E}$	13.3 ± 0.3 $^{ab, A}$	70.1 ± 4.1 $^{a, B}$	169.2 ± 2.7 $^{a, A}$
B18	7.7 ± 0.2 $^{a, B}$	0.56 ± 0.02 $^{a, B}$	74.6 ± 0.7 $^{a, C}$	10.3 ± 0.6 $^{e, C}$	53.1 ± 1.2 $^{e, E}$	69.1 ± 1.8 $^{e, F}$
B20	5.1 ± 0.2 $^{b, D}$	0.43 ± 0.02 $^{b, D}$	70.0 ± 0.8 $^{b, D}$	12.7 ± 0.5 $^{cd, B}$	57.2 ± 3.1 $^{d, D}$	108.0 ± 1.9 $^{d, D}$
B22	4.1 ± 0.2 $^{c, F}$	0.35 ± 0.04 $^{c, F}$	68.6 ± 0.7 $^{c, E}$	13.1 ± 0.3 $^{abc, A}$	65.4 ± 2.1 $^{c, C}$	129.3 ± 2.1 $^{c, C}$
B24	3.6 ± 0.1 $^{d, G}$	0.25 ± 0.03 $^{d, G}$	67.9 ± 0.7 $^{d, E}$	13.2 ± 0.4 $^{abc, A}$	67.3 ± 2.1 $^{bc, BC}$	146.2 ± 3.1 $^{b, B}$
B26	2.7 ± 0.0 $^{e, H}$	0.21 ± 0.02 $^{e, H}$	66.7 ± 0.4 $^{e, F}$	13.5 ± 0.2 $^{a, A}$	81.2 ± 3.2 $^{a, A}$	170.9 ± 3.5 $^{a, A}$

Different lower-case letters in the same column indicate significant differences among the samples belonging to the same baking mode ($p < 0.05$). Different capital letters in the same column indicate significant differences among the samples ($p < 0.05$).

Table 3
Main quality attributes of the different commercial biscuit types from three different brands (SE - Shortbreads with Eggs; SC - Shortbreads with Chocolate chips; DP - Dry Petit biscuits).

Biscuit samples	Moisture (%)	Water activity (a_w)	L*	a*	Hardness (N)	Crispness (linear distance)	Thickness (mm)
SE1	2.6 ± 0.1 ^{b}	0.23 ± 0.02 ^{a}	60.5 ± 1.1 ^{a}	14.6 ± 0.3 ^{a}	41.6 ± 6.1 ^{b}	54.0 ± 10.0 ^{b}	9.9 ± 0.3 ^{b}
SE2	2.3 ± 0.1 ^{c}	0.19 ± 0.02 ^{b}	60.2 ± 1.5 ^{a}	13.9 ± 0.5 ^{b}	45.7 ± 4.6 ^{b}	52.5 ± 7.7 ^{b}	9.7 ± 0.4 ^{b}
SE3	3.0 ± 0.2 ^{a}	0.24 ± 0.02 ^{a}	60.3 ± 1.9 ^{a}	13.3 ± 0.5 ^{b}	65.8 ± 8.7 ^{a}	76.1 ± 10.5 ^{a}	11.7 ± 0.3 ^{a}
Mean	*2.6 ± 0.3* ^{B}	*0.22 ± 0.03* ^{A}	*60.3 ± 0.1* ^{B}	*13.9 ± 0.6* ^{A}	*51.0 ± 12.9* ^{B}	*60.9 ± 13.2* ^{B}	*10.4 ± 1.1* ^{B}
SC1	2.5 ± 0.1 ^{b}	0.21 ± 0.02 ^{b}	59.6 ± 1.9 ^{b}	11.9 ± 0.4 ^{a}	95.1 ± 9.3 ^{a}	95.0 ± 9.3 ^{a}	10.2 ± 0.3 ^{c}
SC2	2.7 ± 0.1 ^{b}	0.22 ± 0.01 ^{b}	55.7 ± 1.7 ^{c}	12.0 ± 0.4 ^{a}	59.9 ± 8.4 ^{c}	59.9 ± 8.5 ^{c}	11.5 ± 0.5 ^{b}
SC3	3.2 ± 0.3 ^{a}	0.26 ± 0.03 ^{a}	63.1 ± 2.6 ^{a}	11.5 ± 0.9 ^{b}	73.0 ± 11.3 ^{b}	73.4 ± 11.4 ^{b}	13.5 ± 0.4 ^{a}
Mean	*2.8 ± 0.4* ^{C}	*0.23 ± 0.03* ^{A}	*59.5 ± 3.7* ^{B}	*11.8 ± 0.3* ^{B}	*76.0 ± 17.8* ^{A}	*76.0 ± 17.7* ^{A}	*11.7 ± 1.7* ^{A}
DP1	0.8 ± 0.1 ^{b}	0.08 ± 0.02 ^{c}	68.1 ± 0.8 ^{a}	12.2 ± 0.4 ^{b}	30.7 ± 5.5 ^{a}	31.6 ± 5.6 ^{a}	5.4 ± 0.3 ^{b}
DP2	2.3 ± 0.2 ^{a}	0.19 ± 0.02 ^{a}	58.9 ± 1.1 ^{c}	16.3 ± 0.5 ^{a}	28.1 ± 5.5 ^{a}	28.5 ± 5.7 ^{ab}	6.1 ± 0.3 ^{a}
DP3	2.2 ± 0.2 ^{a}	0.15 ± 0.01 ^{b}	67.0 ± 1.7 ^{b}	11.3 ± 0.9 ^{c}	22.6 ± 4.5 ^{b}	25.5 ± 6.2 ^{b}	5.4 ± 0.3 ^{b}
Mean	*2.0 ± 1.1* ^{A}	*0.14 ± 0.05* ^{B}	*64.7 ± 5.0* ^{A}	*13.3 ± 2.7* ^{A}	*27.1 ± 4.1* ^{C}	*28.5 ± 3.0* ^{C}	*5.6 ± 0.4* ^{C}

Different letters in the same column indicate significant differences among the samples belonging to the same biscuit category ($p < 0.05$). Different capital letters in the same column indicate significant differences among the mean values of SE, SC and DP ($p < 0.05$).

examined had L* higher than 50 with average of 66.5 ± 1.5, 64.5 ± 1.8 and 71.3 ± 0.9 for SE, SC and DP biscuits, respectively. The lightest colour of DP biscuits can be attributed to the usually lower fat and sugar content in the recipe, which can lead to a slower development of Maillard reactions. On the other hand, the darker colour of SC biscuits can be attributed to the presence of whole chocolate chips and their partial melting during baking. Furthermore, the biscuits SC and DP showed significant differences between the different brands, especially the samples SC2 and DP2 were the darkest within their respective categories. These differences can be attributed to possible differences in the baking methods and parameters adopted by the companies of each biscuit brand. As far as the colour parameter a* is concerned, the samples of the categories SC and DP had significantly lower values, indicating less red and yellow colouring of the top of the biscuit compared to SE. This result could be attributed to the use of fresh eggs in the formulation of the SE biscuits, an ingredient not used in the other categories, with the exception of sample DP1, which can however be assumed to contain a relatively small amount of this ingredient. The main described colour differences between the commercial biscuit types and brands were also recognisable from the visual appearance shown in **Fig. S2(b)** (in **Supplementary Materials**).

The texture of commercial biscuits is also closely influenced by the biscuits category. The commercial samples analysed revealed an average hardness and crispness of 51.0 ± 12.9 N and 60.9 ± 13.2; 76.0 ± 17.8 N and 76.0 ± 17.7; 27.1 ± 4.1 N and 28.5 ± 3.0 for SE, SC and DP biscuits, respectively. The hardest and crispiest samples were shortbreads with chocolate chips, particularly SC1 and SC3, followed by SE biscuits, while the least hard and crispy were DP biscuits. The inclusion of chocolate chips, which results in a more compact and irregular structure in the biscuits, can be ascribed with the highest values of the texture parameters of SC. The lower values of the texture parameters of DP can be attributed to the lower moisture content and the lower proportion of fats and sugars in the recipe, which makes the consistency of this type of biscuit more alveolate and less compact than shortbread biscuits. In addition, the lower hardness and crispness of the DP biscuits could also be explained by the fact that they were thinner (5.6 ± 0.4 mm on average) than the others (10.4 ± 1.1 mm for SE and 11.7 ± 1.7 mm for SC). Thickness also varied greatly between brands, especially the champions SE3, SC3 and DP2 presented a greater thickness within their category. The behaviour of the chemical reactions during baking, and therefore the aforementioned quality parameter, could also have been influenced by the thickness and shape of the biscuits, which can vary depending on the type and brand (Pereira et al., 2013).

3.2. VOCs concentration of biscuit samples

Experimental and commercial biscuit samples were analysed using the optimised and validated HS-SPME-GC–MS method to quantify six VOCs such as 3-methylbutanal, hexanal and four furanic compounds such as furfural, furfuryl acetate, 5-methylfurfural and furfuryl alcohol.

The effect of static and ventilate heat-transfer mode for different time during baking on the concentration of all monitored VOCs in the experimental biscuit samples was evaluated and the results are reported in Table 4.

The highest average values of 3-methylbutanal and hexanal were found in experimental biscuits baked in static mode (A) with respect to those baked in ventilate one (B). The concentration of 3-methylbutanal and hexanal decreased in both samples as the baking time increased. The highest concentration of both compounds was found in samples baked in static mode for 18 and 20 min and the lowest in biscuits baked in a ventilate mode for 24 and 26 min, with statistically significative differences. Biscuits baked in static mode for 18 min displayed the significantly highest concentration of hexanal compared to other samples. On the other hand, no statistically significant differences were found between the concentration of hexanal in biscuits baked in ventilate mode. Where significant, the decrease of 3-methylbutanal and hexanal with increasing baking time can be attributed to the evaporation process as these compounds have a boiling point of 94 °C and 129 °C, respectively (Hertel et al., 2007). In a previous study, it was shown that the internal temperature of the biscuit reached 100 °C after no more than 10 min in both baking modes; moreover, the temperature was reached slightly faster in the ventilate mode than in the static one (Schouten et al., 2022). This could also explain the lower concentration of both compounds in the B biscuits compared to A ones.

Concerning the furanic compounds, the concentration of furfural remained almost the same in both samples at all baking times. Biscuits baked in a static mode for 18 and 20 min had significantly higher concentrations of furfural than B samples baked for the same times. On the average, biscuits baked in ventilate mode had a lower amount of furfural with respect to those baked in static mode.

The concentrations of furfuryl acetate and 5-methylfurfural, decreased slightly with increasing baking time in static mode biscuits without statistically significant differences. Similarly, in the samples baked in ventilate mode, the concentration of these two compounds did not change significantly during the baking times. In particular, the A biscuits baked for 18 and 20 min had a higher concentration of these furanic compounds than the same samples baked for longer times. Compared to the A biscuits, the B samples baked in ventilate mode had lower amounts of furfuryl acetate and 5-methylfurfural, regardless of the baking time, although without statistically significant differences.

The concentration of furfuryl alcohol did not change in the A biscuits, but it decreased significantly in the B ones during the prolonged baking process. This is probably due to the increasing temperature on the surface of the B biscuits, as the boiling point of this compound is

Table 4
Concentrations of the monitored volatile organic compounds (VOCs) in experimental biscuit samples baked in static (A) and ventilate (B) mode for different times. VOCs concentrations are expressed in mg·kg^{-1} and reported as average ± standard deviation (n = 3).

Biscuit sample	3-methylbutanal	Hexanal	Furfural	Furfuryl acetate	5-methylfurfural	Furfuryl alcohol	Total furanic compounds
A18	8.1 ± 0.34 $^{a, A}$	0.18 ± 0.02 $^{a, A}$	2.7 ± 0.07 $^{a, A}$	0.5 ± 0.04 $^{a, A}$	1.1 ± 0.04 $^{a, A}$	4.2 ± 0.35 $^{a, B}$	8.6 ± 0.24 $^{a, A}$
A20	3.7 ± 0.16 $^{b, A}$	0.10 ± 0.01 $^{b, A}$	2.4 ± 0.24 $^{a, A}$	0.5 ± 0.05 $^{a, A}$	0.9 ± 0.1 $^{a, A}$	3.7 ± 0.20 $^{a, A}$	7.5 ± 0.20 $^{b, A}$
A22	2.6 ± 0.26 $^{c, A}$	0.08 ± 0.01 $^{b, A}$	1.9 ± 0.23 $^{b, A}$	0.4 ± 0.00 $^{a, A}$	0.7 ± 0.1 $^{a, A}$	3.8 ± 0.56 $^{a, A}$	6.7 ± 0.42 $^{c, A}$
A24	2.0 ± 0.37 $^{c, A}$	0.07 ± 0.01 $^{b, A}$	2.1 ± 0.16$^{b, A}$	0.4 ± 0.04 $^{a, A}$	0.7 ± 0.07 $^{a, A}$	3.6 ± 0.08 $^{a, A}$	6.8 ± 0.21 $^{bc, A}$
A26	2.5 ± 0.31 $^{c, A}$	0.06 ± 0.00 $^{c, A}$	2.1 ± 0.08 $^{b, A}$	0.4 ± 0.02 $^{a, A}$	0.7 ± 0.06 $^{a, A}$	3.9 ± 0.24 $^{a, A}$	7.1 ± 0.26 $^{bc, A}$
B18	2.7 ± 0.37 $^{a, B}$	0.07 ± 0.01 $^{a, B}$	1.7 ± 0.03 $^{a, B}$	0.3 ± 0.01 $^{a, A}$	0.6 ± 0.02 $^{a, A}$	5.8 ± 0.78 $^{a, A}$	8.3 ± 0.77 $^{a, A}$
B20	2.7 ± 0.50 $^{a, B}$	0.09 ± 0.01 $^{a, A}$	1.7 ± 0.14 $^{a, B}$	0.3 ± 0.03 $^{a, A}$	0.5 ± 0.05 $^{a, A}$	4.3 ± 0.23 $^{b, A}$	6.8 ± 0.29 $^{b, B}$
B22	2.3 ± 0.25 $^{a, A}$	0.08 ± 0.01 $^{a, A}$	1.7 ± 0.11 $^{a, A}$	0.3 ± 0.01 $^{a, A}$	0.5 ± 0.03 $^{a, A}$	3.9 ± 0.51 $^{c, A}$	6.5 ± 0.66 $^{b, A}$
B24	1.7 ± 0.17 $^{b, A}$	0.05 ± 0.01 $^{a, A}$	1.7 ± 0.06 $^{a, A}$	0.3 ± 0.02 $^{a, A}$	0.5 ± 0.03 $^{a, A}$	3.4 ± 0.40 $^{d, A}$	5.9 ± 0.49 $^{b, A}$
B26	1.5 ± 0.12 $^{b, B}$	0.06 ± 0.00 $^{a, A}$	1.7 ± 0.15 $^{a, A}$	0.3 ± 0.04 $^{a, A}$	0.5 ± 0.07 $^{a, A}$	3.1 ± 0.05 $^{e, A}$	5.7 ± 0.27 $^{b, B}$

Different lower-case letters in the same column indicate significant differences among the samples belonging to the same baking mode ($p < 0.05$).
Different capital letters between A and B at the same baking time indicate significant differences among the samples ($p < 0.05$).

around 171 °C. Biscuits baked in ventilate mode (B) for 18 min had the highest significant concentration of furfuryl alcohol. In the biscuit matrix, this compound can be converted to 4-hydroxycyclopent-2-enone (4-HCP) in the presence of water at high temperature (Hronec et al., 2014). In this case, the concentration of furfuryl alcohol in the biscuits baked in ventilate mode (B) was slightly higher during the first baking times than in those baked in static mode (A). This is probably mainly due to the overall higher dehydration reached by the B sample during the first 20 min of baking, as the hot air removes the water from the surface of this sample more quickly. On the contrary, the overall higher moisture content of the A sample during baking may have resulted in a lower concentration of furfuryl alcohol.

When comparing biscuit samples baked under the selected optimal conditions (22 min for static and 20 min for ventilate baking) for furanic compounds linked to health risks (Petisca et al., 2013; Shen et al., 2016), there was no statistically significant difference for furfuryl alcohol, while there were statistically significant differences ($p < 0.05$) between samples for furfuryl acetate and furfural.

Overall, the average total value of all furanic compounds (i.e., furfural, furfuryl acetate, 5-methylfurfural and furfuryl alcohol) was significantly higher in the A biscuits than in those baked in ventilate mode after 20 and 26 min (Table 4). However, there was no statistically significant difference between the biscuits baked under the optimal baking conditions (22 min for A and 20 min for B samples). For both baking modes, total furanic compounds decreased with increasing baking time, in agreement with a study conducted on rye bread crusts by Ozolina et al. (2011).

In order to evaluate the effect of different formulation on the investigated VOCs concentration different commercial biscuit types from different brands were also analysed and the obtained results are reported in Table 5.

The significantly highest average concentration of 3-methylbutanal was found in shortbread with chocolate chips (4.6 mg·kg^{-1}). This result is likely due to the presence of butter in the SC samples, an ingredient not present in the biscuit types SE and DP. In the category SC, brand SC3 had the highest concentration of this compound probably due to the highest presence of fat ingredients. The 3-methylbutanal is responsible for the malty flavour of buckwheat honey and is a product of the Strecker reaction by deamination and decarboxylation of amino acids by dicarbonyls produced in the Maillard reactions (Van Boekel, 2006).

As far as hexanal concentration is concerned, the highest significant average value was found in SE (3.1 mg·kg^{-1}) and in particular in SE1 brand. Hexanal is the product of the oxidation of linoleic acid (Purcaro et al., 2008) and contributes to buckwheat flavour (Janeš et al., 2009). It is an important indicator of food quality after a long storage period, as the aldehydes produced by the action of lipoxygenase are responsible for undesirable odours. Therefore, the highest value in SE biscuits can be attributed to the presence of fresh eggs, vegetable sunflower oil and honey, which may contribute to higher hexanal levels. The sample SE1, which contains the highest concentration of hexanal, probably contains a higher percentage of honey in the formulation than the SE samples of other brands. According to Castro-Vázquez et al. (2009), the concentration of hexanal in honey also depends on its plant origin which cannot be determined by the label of the biscuits evaluated.

The highest concentrations of the total furanic compounds studied were found in SE biscuits, followed by SC and finally DP ones; with the exception of furfuryl alcohol, the concentration of which was higher in SC than in the others commercial biscuits types (Table 5). The average concentration of furfural in DP (0.5 mg·kg^{-1}) was in agreement with values found in biscuits in a previous study (0.65 mg·kg^{-1}) (Mesias et al., 2021). To the best of the knowledge, there are no other studies analysing the other furanic compounds in similar commercial biscuits. The higher concentration of furanic compounds found in shortbread

Table 5
Concentrations of the monitored volatile organic compounds (VOCs) in the different commercial biscuit types from three different brands (SE - Shortbreads with Eggs; SC - Shortbreads with Chocolate chips; DP - Dry Petit biscuits). VOCs concentrations are expressed in mg·kg^{-1} and reported as average ± standard deviation (n = 3).

Biscuit sample	3-methylbutanal	Hexanal	Furfural	Furfuryl acetate	5-methylfurfural	Furfuryl alcohol	Total furanic compounds
SE1	1.3 ± 0.25 ^{a}	5.9 ± 1.16 ^{a}	2.6 ± 0.47 ^{a}	1.4 ± 0.27 ^{a}	1.4 ± 0.28 ^{a}	1.9 ± 0.37 ^{a}	7.3 ± 1.40 ^{a}
SE2	0.9 ± 0.05 ^{a}	2.2 ± 0.44 ^{b}	1.6 ± 0.14^{b}	0.7 ± 0.09 ^{b}	0.8 ± 0.09 ^{b}	1.3 ± 0.09 ^{a}	4.4 ± 0.41 ^{b}
SE3	1.1 ± 0.15 ^{a}	1.0 ± 0.05 ^{c}	0.9 ± 0.02 ^{c}	0.3 ± 0.01 ^{c}	0.5 ± 0.01 ^{c}	1.9 ± 0.09 ^{a}	3.7 ± 0.06 ^{b}
Mean	*1.1 ± 0.13* ^{B}	*3.1 ± 0.52* ^{A}	*1.6 ± 0.34* ^{A}	*0.8 ± 0.12* ^{A}	*0.9 ± 0.12* ^{A}	*1.6 ± 0.13* ^{B}	*5.1*
SC1	1.5 ± 0.02 ^{a}	0.9 ± 0.13 ^{a}	1.3 ± 0.17 ^{a}	0.4 ± 0.07 ^{a}	0.6 ± 0.09 ^{a}	1.9 ± 0.11 ^{a}	4.2 ± 0.43 ^{a}
SC2	3.8 ± 0.75 ^{a}	0.6 ± 0.10 ^{a}	1.2 ± 0.10 ^{b}	0.3 ± 0.05 ^{a}	0.5 ± 0.04 ^{a}	3.1 ± 0.13 ^{b}	5.2 ± 0.06 ^{a}
SC3	8.9 ± 1.71 ^{b}	0.4 ± 0.07 ^{a}	0.9 ± 0.02 ^{c}	0.2 ± 0.04 ^{b}	0.4 ± 0.01 ^{a}	2.3 ± 0.31 ^{a}	3.9 ± 0.29 ^{a}
Mean	*4.6 ± 0.44* ^{A}	*0.7 ± 0.10* ^{B}	*1.1 ± 0.12* ^{A}	*0.3 ± 0.05* ^{B}	*0.5 ± 0.05* ^{B}	*2.4 ± 0.30* ^{A}	*4.4*
DP1	1.1 ± 0.22 ^{a}	0.1 ± 0.01 ^{a}	0.4 ± 0.08 ^{a}	0.1 ± 0.01 ^{a}	0.3 ± 0.06 ^{a}	1.0 ± 0.19 ^{a}	1.8 ± 0.34 ^{a}
DP2	1.1 ± 0.09 ^{a}	0.1 ± 0.01 ^{a}	0.6 ± 0.02 ^{a}	0.1 ± 0.01 ^{a}	0.3 ± 0.01 ^{a}	2.9 ± 0.22 ^{b}	4.0 ± 0.22 ^{b}
DP3	0.7 ± 0.03 ^{a}	0.2 ± 0.02 ^{a}	0.4 ± 0.01 ^{a}	0.05 ± 0.01 ^{a}	0.2 ± 0.00 ^{a}	0.9 ± 0.09 ^{c}	1.7 ± 0.08 ^{a}
Mean	*0.9 ± 0.09* ^{B}	*0.2 ± 0.01* ^{B}	*0.5 ± 0.02* ^{B}	*0.1 ± 0.00* ^{C}	*0.3 ± 0.01* ^{C}	*1.8 ± 0.15* ^{B}	*2.5*

Different lower-case letters in the same column indicate significant differences among the samples belonging to the same biscuit category ($p < 0.05$). Different capital letters in the same column indicate significant differences among the mean values of SE, SC and DP ($p < 0.05$).

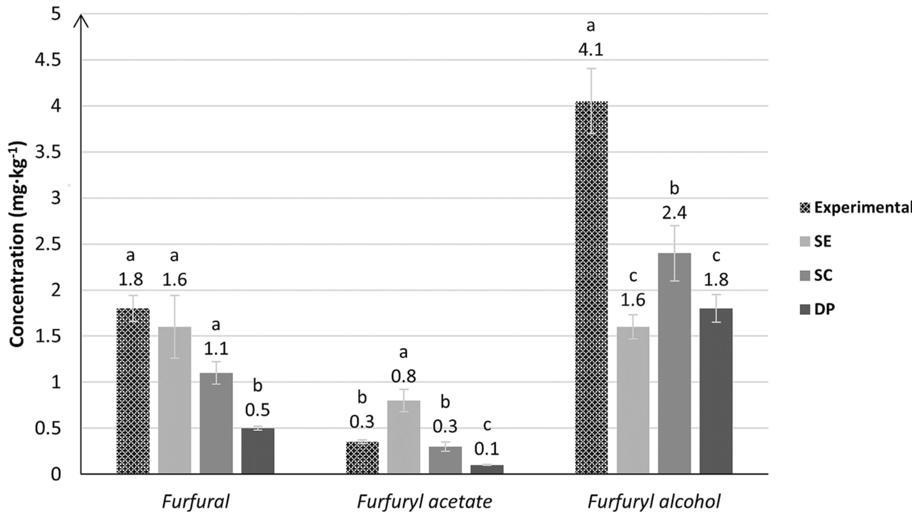

Fig. 1. Average values of furfural, furfuryl acetate and furfuryl alcohol found in the experimental biscuits baked under optimal conditions (at 175 °C static for 22 min and ventilate for 20 min) and in commercial Shortbreads with Eggs (SE), Shortbreads with Chocolate chips (SC) and Dry Petit (DP) biscuit types. Different letters in the same compounds indicate significant differences among samples at $p < 0.05$ level.

biscuits compared to DP ones is probably related to the generally richer recipe. Indeed, the presence of different amounts and types of ingredients influencing the a_w, pH values and the interaction between the main substrates in the dough may change the rate of Maillard reactions taking place during baking and thus the amount of furanic compounds. In addition, the different time, temperature and method conditions normally used for the baking of each commercial biscuit category could be also responsible for the different concentrations of the VOCs (Manley, 2011; Shakoor et al., 2022).

With regard to the furanic compounds considered as the most toxics, such as furfural, furfuryl acetate and furfuryl alcohol (Petisca et al., 2013; Shen et al., 2016), their averaged concentrations found in experimental biscuits baked under optimal conditions (at 175 °C for 22 min for A mode and 20 min for B mode) and in commercial biscuit types were compared as showed in Fig. 1. The most important difference was the higher average concentration of furfuryl alcohol in the experimental biscuits, which was of 61.0, 41.5 and 56.1% compared to SE, SC and DP, respectively. On the other hand, the experimental biscuits contained lower concentrations of furfuryl acetate than the SE biscuits but not compared to the other commercial biscuit types. Finally, the lowest values of furfural were found in DP and SC biscuits. As mentioned above, these results could be due to differences in the type and content of ingredients, as well as between the shaping and baking conditions adopted for the production of the different types of biscuit analysed.

4. Conclusions

Although there are no specific reference levels for toxic VOCs in food, researchers in industry and academia have made significant progress in identifying possible variables involved in the formation of this group of contaminants in order to reduce them to the lowest reasonably achievable level. The ability to analyse VOCs concentrations in biscuits and other bakery products is therefore important in controlling the levels of these heat-induced contaminants. To contribute to this global effort, the present study optimised and validated an analytical method for six VOCs to determine the concentrations of these compounds in two types of experimental biscuits baked under different conditions and in three different widely consumed commercial biscuits of different brands. The main results confirm that both the baking conditions and the different recipes of the biscuits can influence the main quality baking attributes as well as the formation of 3-methylbutanal, hexanal and furanic compounds.

The use of the oven set at ventilated mode resulted in faster baking of the biscuits. This was clearly demonstrated by the results of the main quality parameters measured on the experimental biscuits baked for different lengths of time. As a consequence, it was found that the studied VOCs concentrations in the biscuits baked in ventilated mode were lower than in those baked in static mode for some of the baking times considered. When comparing lab-made biscuits baked under the optimal baking conditions (22 min for static baking and 20 min for ventilated baking), there were statistically significant differences between the biscuits for furanic compounds associated with health risks, with the exception of furfuryl alcohol.

Commercial biscuits in the category shortbread with eggs, shortbread with chocolate chips and dry petit biscuits produced by different brands differed in terms of the most important quality characteristics studied. This can be attributed to their different recipes, but also to possible different shaping and baking conditions, which may influence the behaviour of the physical and chemical reactions during baking and thus the different concentrations of VOCs found. On average, shortbread types had the highest levels of 3-methylbutanal, hexanal and furanic compounds, which can be attributed to their recipes richer in fats and sugars (e.g., eggs, butter, chocolate), which may have contributed to higher rate of Maillard reactions compared to dry petit biscuits.

Overall, it is a challenge to completely prevent the development of heat-induced contaminants while ensuring that biscuits continue to have the desired quality characteristics given the wide variety of biscuits available on the market and baked at home.

CRediT authorship contribution statement

Laura Acquaticci: Writing – original draft, Investigation, Formal analysis. **Maria Alessia Schouten:** Writing – original draft, Investigation, Formal analysis. **Simone Angeloni:** Investigation, Methodology, Writing – review & editing. **Giovanni Caprioli:** Methodology, Writing – review & editing, Supervision. **Sauro Vittori:** Writing – review & editing, Supervision. **Santina Romani:** Conceptualization, Writing – review & editing, Supervision, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2023.137791.

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