Extruded flour improves batter pick-up, coating crispness and aroma profile

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Abstract

Fried battered foods are widely consumed worldwide. In this study, the influence of the replacement of native wheat flour by extruded flours (7.5 and 15%) subjected to different extrusion severities on chicken nuggets was assessed. Microstructure, pick-up, moisture and fat content, texture, colour, volatile profile, and consumer acceptability were evaluated. Extruded flour replacement resulted in batters with improved pick-up with increasing percentage and severity of extrusion treatment. Extruded flour also contributed to a moisture increase, while oil remained practically unchanged. Textural properties highlighted a higher crispness of batters made with high-severity treatment extruded flours. Volatile compounds analyses revealed lower amount of lipid oxidation (lower rancidity notes) and higher furfuryl alcohol content (pleasant aroma and darker crust) in fried batters containing extruded flour. Consumers testing showed that batters made with intermediate-severity treatment extruded flour presented the best acceptability. These results confirm that extruded flour inclusion improves the quality of deep-fried batters.

Keywords: batter**;** extrusion**;** coating**;** pick-up**;** texture; aroma; volatile

1 Introduction

Fried battered foods are prominent in the diet of consumers all over the world. In the past, fried battered products have typically been prepared and consumed at home, but in more recent decades they have become very popular in the market of prepared food (Sanz, Salvador, & Fiszman, 2004b). Their growing demand is due to the convenience with which they can now be stored frozen at home and finished in a quick way for immediate consumption (Martínez, Sanz, & Gómez, 2015). A batter can be defined as a liquid dough, basically made of flour, water, leavening agent, and other minor ingredients, into which a product is dipped prior to cooking, normally by frying (Fiszman & Salvador, 2003).

Batter coatings provide a crunchy texture as well as pleasant flavour, and good appearance to foods, and they act as a barrier against the loss of moisture which, in turn, protects the natural juices of foods (Dogan, Sahin, & Sumnu, 2005b; Mohamed, Hamid, & Hamid, 1998). Therefore, batter coatings ensure that the final product is tender and juicy on the inside and crispy on the outside (Fiszman & Salvador, 2003). According to Loewe (1990), batter quality is based on the uniformity and thickness of the coating and its adhesion to the product, along with its general appearance, colour, crunchiness, and taste. In attempts to attain high quality batters, gums and hydrocolloids have been added to the formulations to improve some of these properties (Albert et al., 2009; Primo-Martin et al., 2010; Sanz, Salvador, & Fiszman, 2004a,b). The use of hydrocolloids is mainly related to their ability to absorb and retain water, acting as thickeners or regulators of viscosity. Hydrocolloids also contribute to batter properties by improving adhesion to the food substrate and stability to the freeze-thaw process, ultimately enhancing the textural attributes, maintaining freshness and reducing oil absorption during frying (Duxburry, 1998; Varela & Fiszman, 2011). With a similar aim, pregelatinised starches have been included in batter formulation, increasing coating pick-up (Altunakar, Sahin, & Sumnu, 2004; Mohamed et al., 1998).

Hydrothermal treatments of flours, such as extrusion, combining high temperatures and mechanical shearing at relatively low moisture levels and resident times can cause starch gelatinisation. Other than starch pregelatinisation, extrusion also leads to starch fragmentation, protein denaturation, enzyme (in)activation and Maillard reactions, whose extent is dependent on the severity of the extrusion process (Camire, Camire, & Krumhar, 1990). The intensity of the extrusion treatment, which is related to temperature, moisture content and screw speed, modify flour behaviour by changing its hydration as well as its thermal and pasting properties (Martínez, Rosell, & Gómez, 2014). In this way, extruded flours with a higher degree of pregelatinisation have greater thickening power in cold temperatures than traditional flours, along with higher water absorption and retention capacities (Hagenimana, Ding, & Fang, 2006; Martínez et al., 2014), yielding similar functionality to that of the hydrocolloids or pregelatinised starches. In fact, the use of extruded flours has been proposed to modify the rheology of batter systems (Martínez et al., 2015), demonstrating that the results depend highly on the severity of the extrusion conditions. A further advantage of extruded flours is that extrusion is a physical treatment that allows for the alteration of flour functionality while still maintaining a clean label (Jacobs & Delcour, 1998). Additionally, the process to obtain pregelatinised flour is more economic with a lower environmental impact compared to that of starches (Eckhoff & Watson, 2009). Despite the fact that pregelatinised starches have been used to improve the quality of battered products, to the best of our knowledge, the effect of the addition of extruded flours with different pregelatinisation degrees into these products has never been assessed. Furthermore, although batters coatings are thought to provide the covered food matrix with a pleasant flavour and aroma, no studies on the analysis of the volatile compounds of fried batter products have been performed so far. Moreover, considering that the kind of flour employed strongly influences the volatile profile in other thermally processed foods, such as breads (Pico, Tapia, Bernal, & Gómez, 2017b), it is expected that the type of flour used in the elaboration of fried batter products would also influence its final aroma. In addition, it is worth mentioning that, in this work, different thermally modified flours are used in the batter formula, during which processing Maillard and caramelisation reactions are expected to occur (Camire et al., 1990), which can, in turn, further modify the volatile profile of the fried product.

In this study, the influence of partial wheat flour replacement by extruded wheat flours subjected to different extrusion conditions (mild, intermediate, and high) on the quality of batter coatings was evaluated. Wheat flour in the batters was partially replaced by extruded flour at 7.5 and 15% substitution levels and the microstructure, pick-up, textural properties, and colour of the resulting fried, battered nuggets were analysed. In addition, it is important to mention that in this study, for the first time, volatile compounds in batter systems before and after frying are evaluated. A consumer test was also carried out to evaluate the sensory acceptability of the different coatings.

2 Materials and methods

2.1 Materials

Native wheat flour (moisture and protein contents of 11.73 g/100 g and 11.20 g/100 g, respectively) was supplied by Harinera Castellana (Medina del Campo, Valladolid, Spain). Extruded modified wheat flours (flours 1, 2 and 3) were provided by Molendum Ingredients (Zamora, Spain), who performed the extrusion treatment using a Bühler Basf single screw extruder (Bühler S.A., Uzwil, Switzerland). The length-to-diameter (L/D) ratio for the extruder was 20:1. Initial wheat flour was subjected to different extrusion treatments, which included modifications in barrel temperature, feed rate and moisture content of the mass feed. Three types of extruded flours, numbered from 1 to 3 indicating the severity of the extrusion treatment (1, mild; 2, intermediate and 3, high), were obtained. Both flours 1 and 2 were extruded at a maximum barrel temperature of 120 ºC and a screw speed of 397 rpm. However, flour 1 was extruded with a feed rate of 700 kg/h and a feed moisture content of 60 L/h, while flour 2 was extruded with a feed rate of 400 kg/h and a feed moisture content of 60 L/h. Flour 3 was extruded at a maximum barrel temperature of 160 ºC and a feed moisture content of 50 L/h, with a feed rate of 500 kg/h and with a screw speed of 340 rpm. Then extruded flours were ground with a compression roller to a particle size below 200 microns.

Industrially made and rectangular shaped chicken pieces (30 x 50 x 10 mm) used as the food matrix were kindly provided by Productos Florida (Almazora, Castellón, Spain). Sodium bicarbonate (Manuel Riesgo, S.A., Madrid, Spain) and sodium pyrophosphate (Adín S.A. Paterna, Spain) were used as leavening agents. Sunflower oil and salt were purchased in the local market.

For the volatile profiles characterisation, 2-acetyl-1-pyrroline (2-ACPY, number 16 in Table 2) was purchased from Eptes (Vevey, Switzerland) and the other pure standards labelled from 1 to 15 and from 17 to 43 in Table 2 were obtained from Sigma-Aldrich (Steinheim, Germany). Dichloromethane was obtained from Scharlab (Barcelona, Spain) and methanol from VWR International (Fontenay-sous-Bois, France). Argon, nitrogen and helium were acquired from Carburos Metálicos (Barcelona, Spain).

2.2 Methods

2.2.1. Batter preparation and frying

The batter formulations and preparation was the same as that reported in Martínez et al. (2015). Briefly, the batter formulation was composed of native wheat flour (control) or a mixture of native wheat flour and extruded flour (91.40 g/100 g), sodium pyrophosphate (1.78 g/100 g), sodium bicarbonate (1.32 g/100 g), and salt (5.50 g/100 g). The levels of wheat extruded flour replacement in the flour mixture were 7.5 and 15 g/100 g. Batters were prepared with the three different types of wheat extruded flour (denoted as flour 1, 2, and 3). The pre-blended powders were mixed with water (20 ºC) in a Kenwood Major Classic mixer (Kenwood Ltd, UK) at second speed for 2 min. The water/dry mix proportion was always 1.2:1. Batters containing extruded flours were labeled according to the type of extruded flour included (Flour 1, 2 or 3) and the percentage of wheat flour replacement (7.5 or 15%). Apparent viscosity of the batters at 20 ºC was determined according to Martínez et al. (2015). Briefly, apparent viscosity versus shear rate data was recorded using a rheometer (Haake RheoStress 1, Thermo Fischer Scientific, Scheverte, Germany) with a titanium parallel plate geometry sensor PP60 Ti (60 mm diameter, and 1 mm gap). The test was programmed to increase the shear rate from 1 to 100 s^{-1} for 100 s after a resting time of 5 min. Measurements were made in duplicate and the obtained curves are depicted in supplementary material 1. Viscoelastic and thermal properties of the 7 raw batters were also studied in a previous study (Martínez et al., 2015). The individual chicken pieces were pre-dusted with wheat flour, immersed in the batter, and allowed to drip for 30 s. Immediately afterwards, four battered chicken pieces were fried at a time in a deep fat fryer Taurus Profesional-3 (Taurus, Lleida, Spain) at 190 ± 2 ºC for 3.5 min. The fryer was filled with fresh sunflower oil and preheated at 190 ± 2 ºC for 1 h before frying. Fried chicken pieces were removed from the fryer and let to cool down on a tray covered with tissue paper for 10 min before further analysis. Each of the 7 batters (elaborations) was made in duplicate.

2.2.2 Microstructure

Two fried batters of control batter and batters made with 15% of extruded flours were separated from the nuggets and cut into 2 x 2 cm pieces. Photographs of the external surface of the coating were taken with a Steoroscopic Zoom Microscope Nikon SMZ-1500 (Nikon, Tokyo, Japan) for observation of the coating microstructure.

2.2.3 Batter pick-up determination

In batter-coated products, the term pick-up is generally used to denote the amount of batter adhered to the piece of food. The pick-up will be a determining factor in the yield and the quality of the final product (Albert et al., 2009). Therefore, the amount of batter adhered to the chicken nugget (g/100 g) was considered the batter pick-up value, calculated as:

$$\text{Batter pick-up} = \frac{B}{FP} \text{ x } 100$$

where B is the weight of batter coating the food matrix after cooking and FP is the weight of the whole fried product (chicken nugget and coating), after cooking (Baixauli, Sanz, Salvador, & Fiszman, 2003). Determinations were made on four nuggets per each elaboration.

2.2.4 Moisture and fat content

For moisture and fat determination, the coating was removed from the food matrix. Moisture content was determined according to approved method 44-15.02 (AACC, 2015). For fat determination, the dried sample was subsequently ground using a Super Junior S coffee grinder (Moulinex, Ecully, France). Crude fat content [g/100 g, in a dry basis (db)] was determined by extraction with petroleum ether using an extractor system Ankom XT10 (Ankom Technology Inc., Macedon, NY) following official procedure Am 5-04 (AOCS, 2005). Moisture and fat contents were determined in triplicate.

2.2.5 Colour determination

Colour was measured using a Minolta CN-508i spectrophotometer (Minolta Co., Ltd, Japan) with the D65 standard illuminant and the 2º standard observer; the results were expressed in the CIE L*a*b* colour space. Colour measurements were made on the external surface of the fried battered product. Two measurements were made on the crust of four batters from each elaboration.

2.2.6 Instrumental texture analysis

Four nuggets per each elaboration were measured to obtain mechanical data. A TA-XT2 Texture Analyser (Stable Micro Systems, Godalming, UK) was used with a 25 kg load cell to evaluate the texture of the fried coatings 10 min after frying. A penetrometry test consisting of a single compression was performed using a P/6 aluminum cylinder probe. The test settings were: test speed 1 mm/s, trigger force 5 g, distance 10 mm. Force (N) vs. displacement (mm) curves were plotted.

2.2.7 Volatile compounds analysis

2.2.7.1 Preparation of standard solutions

2-ACPY solutions were prepared in dichloromethane, as 2-ACPY is only stable in dichloromethane and ethyl acetate. It was necessary to work in every moment under inert atmosphere of argon due to the lack of stability of the compound to the oxygen and moisture. For this reason, dichloromethane was dried in a SDS PS-MD-5 purification system from Düperthal Sicherheitstechnik (Karlstein am Main, Germany).

For the other 42 volatile compounds marked from 1 to 15 and from 17 to 43 in Table 2, working solutions of each volatile compound were prepared in methanol. All the solutions were stored in a freezer at -21 °C.

2.2.7.2 Volatile compounds analysis by SPME-GC/QTOF

Both the unfried (UB) and fried batters (FB) were frozen with liquid nitrogen and ground in an Ika grinder model M20 (Staufen, Germany) for 10 s, achieving a powder.

The solid-phase microextraction (SPME) conditions were previously optimised and validated by the research group for the analysis of volatile compounds in bread crust (Pico, Antolín, Román, Gómez, & Bernal, 2018). 0.75 g (± 0.0050 g) of each batter powder (unfried or fried) was weighed into a 20 mL vial. The selected fibre was 50/30 µm DVB/CAR/PDMS (Sigma Aldrich, Gillingham, UK). The sample was incubated for 5 min at 60 °C (without the fibre) and then extracted for 51 min at 60 °C, without agitation. After that, the fibre was inserted into the GC injector port for thermal desorption during 5 min at 270 °C, with an injection volume of 1µL. Finally, the fibre was conditioned for 30 min at 270 °C after each analysis. Each sample was analysed in triplicate.

GC/QTOF analysis conditions were the same as used in Pico, del Nozal, Bernal, and Gómez, (2017a). All the volatile compounds were identified by comparison of their retention times and accurate mass spectra (with four decimal places) with standards as well as using their Mass Spectra Library (NIST MS Search 2.2 & MS Interpreter).

2.2.8 Consumer testing

Hedonic sensory evaluation of battered products was conducted with 94 volunteers, who were regular battered products eaters between 16-65 years of age and from various socioeconomic backgrounds. Consumer tests were carried out at the Sensory Science Laboratory of the Agricultural Engineering College at the University of Valladolid, Palencia (Spain) in individual booths. Although complete nugget pieces were provided to the consumers, they were only asked (both oral and written instructions) to evaluate the chicken nuggets based on their coatings. Batters were evaluated on the basis of acceptance of their appearance, odour, taste, texture, and overall liking on a nine-point hedonic scale. The scale of values ranged from “extremely like” to “extremely dislike” corresponding with highest and lowest scores of “9” and “1”, respectively. Samples were presented on white plastic dishes coded with four-digit random numbers and served in random order. Water was available for rinsing. Samples were tested 10 min after being fried. Since sensory evaluation was carried out with non-trained panelists, and then, a high number of samples can be excessive and saturate consumer’s perception, only four samples were chosen: control sample and the three batters with 15% of replacement by extruded flour. The samples with the highest level of replacement were chosen based on the greater differences in their batter psychochemical characteristics compared to the control.

2.2.9 Statistical analyses

Differences between the batters were studied by analysis of variance (ANOVA). Fisher’s least significant differences (LSD) method was used to describe means with 95% confidence intervals. The statistical analysis was performed with Statgraphics Centurion XVI software (Statpoint Technologies, Inc., Warrenton, USA). To assess the variation of the volatile profiles from unfried and fried batters regarding the use of native or extruded flours, a PCA was conducted with the peak areas average of each batter sample (unfried or fried) prepared by duplicate and analysed by triplicate (n=6). The PCA was performed with the software LatentiX version 2.00 (Latent5, Copenhagen, Denmark), with data standardised prior to the analysis.

3 Results and Discussion

3.1 Batter pick-up determination

Coating pick-up is an important quality parameter of battered products in the food industry, with values varying between 30–50 g/100 g. In this study, pick-up values (30.95-46.39 g/100 g) increased with increases in both the intensity of extrusion treatment of the wheat flour (flour 1< flour 2< flour 3) and the level of replacement of extruded flour in the batter (Table 1). No significant differences in pick-up were observed between control batter and batters made with extruded flour 1, the one subjected to the mildest extrusion treatment. However, higher pick-up values (i.e., greater thickness of the batter layer) were obtained for batters made with 15% extruded flour 2 and, especially, for 7.5 and 15% of extruded flour 3 (subjected to the most severe extrusion treatment), with higher pick-up observed with higher replacement level. It is known that coating pick-up is positively correlated with batter viscosity in the sense that as viscosity rises more batter remains adhered to the sample (Dogan, Sahin, & Sumnu, 2005a; Fiszman & Salvador, 2003). In this study, increasing extruded flour amount and severity of extrusion treatment in the batter formula resulted in an increase in the apparent viscosity of the batters, as seen in supplementary material 1. Thus, the higher pick-up values are in agreement with the higher apparent viscosity of the batters as well as with the higher consistency index found by Martínez et al. (2015) in raw batters when using the same extruded wheat flours. These results are explained by the presence of pregelatinised starch in extruded flours, whose content increases with the severity of the extrusion treatment (flour 3> flour 2> flour 1) [Martínez et al., 2014; 2015]. In this way, a greater content of pregelatinised starch would contribute to a higher water absorption capacity and viscosity of the more severely extruded wheat flours, as seen by Martínez et al. (2014), yielding higher values of coating pick-up.

3.2 Moisture and fat content

Moisture and fat content for the different batter formulations are shown in Table 1. With regard to moisture retention, no differences were observed in moisture content between the 7.5% level of extruded flours subjected to mild treatments (flour 1 and flour 2) and the control. However, for the rest of the extruded flours, higher moisture content was observed with increasing severity of extrusion treatment and with increasing level of replacement in the most pregelatinised flour (flour 3). Again, this higher moisture content would be related to the superior water retention capacity of the more severely extruded flours (i.e., less free water able to evaporate during frying) leading to better barrier properties. Similarly, Altunakar et al. (2004), using different starches for batter formulation, reported that the highest pick-up and moisture values were found with pregelatinised tapioca starch, relating this event to its higher water binding capacity and film forming ability. Another plausible explanations for the moisture content could also be related to differences between the external structure of the batters. Formation of bubbles and vented holes was observed on the outer surface of all types of fried batters. Notably, batter solely containing native wheat flour seemed to show higher number of gas cells and more deeper and vented holes, which may indicate a greater ease in steam migration and gas escape during frying (supplementary material 2). Thus, higher water evaporation is associated with larger number of voids, cracks, and crevices on the batter surface (Dana & Saguy, 2006).

Regarding fat content, whose values ranged from 14.62 to 16.01 g/100 g, no significant differences were found for the oil content of the different batters. During the frying process, oil absorption occurs as moisture is evaporated from the food (Dana & Saguy, 2006). Then, the higher moisture retention in batters with higher percentage of extruded flour should have been associated with lower fat content in the fried batter. It is noteworthy that, despite the fact that some authors have found a reduction in oil absorption content in the presence of hydrocolloids due to the increase in moisture retention (Sanz et al., 2004a,b), values of fat content with the hydrocolloid incorporation were similar to those of this study (~15-16 g/100 g, db). Furthermore, Primo-Martín et al. (2010) found that the relationship between moisture and oil content was not always held true, indicating that other mechanisms, apart from water replacement, are responsible for the oil absorption phenomenon. Oil uptake is a surface phenomenon, and its uptake and distribution are determined by the crust microstructure, ultimately leading to the restriction of oil absorption to the immediate crust and product surface (Dana & Saguy, 2006). In agreement with this, Varela, Salvador, and Fiszman (2008) measured the oil content in the crust and core of battered chicken nuggets and reported that oil content in the core did not significantly change. Dana & Saguy (2006) already reported that one of the approaches to reduce oil absorption after frying is to wipe the product surface with absorbent paper. Therefore, a plausible mechanism is that oil may have migrated from inside the crust after frying to its exterior towards the absorbent paper used to keep the fried nuggets before further analysis rather than inside the crust towards the nugget core. This oil migration would be more prone to occur in the control sample, since the greater number of deep and vented external holes (see supplementary material 2) and the thinner crust (poor physical barrier to oil migration) could facilitate superficial oil loss after frying. Conversely, the higher coating pick up, which means enhanced crust formation and efficient coverage of the surface, might have controlled the diffusion of oil into and out of the product when more pregelatinised extruded flour was used in the batter formulation.

3.3 Colour determination

The colour of a battered product is directly related to its external appearance, and, therefore to its acceptance by consumers. Colour parameters of the different batters are shown in Table 1. All crusts presented the typical dark golden colour (see supplementary material 2) caused by Maillard reactions and sugar caramelisation at high temperatures (Loewe, 1990). In general, the use of extruded flours in the formulation brought about darker (lower L* values) and more reddish coatings (higher a* values) with minimal differences in the yellowish (b* values). Results also indicated a darkening of the crust (lower L* and higher a* values) with increasing both the percentage of the extruded flour and the intensity of its treatment. This occurrence should be attributed to starch dextrinisation during extrusion processing, resulting in the formation of reducing sugars that will participate in Maillard and sugar caramelisation. In fact, Martínez et al. (2014) reported that free sugar content increased in parallel with the extrusion severity, although a minimum intensity was necessary for promoting those changes, as the scarce differences in L* values between control sample and mildly extruded flour confirmed. Furthermore, not only free sugars are produced during extrusion cooking but also the process conditions used (high temperatures in combination with shear strain and low water content) are known to favour the Maillard reaction between reducing sugars and proteins (Camire et al., 1990). This may have also accounted for a darker colour of the extruded flour, and, in turn, of the coating made with this flour. Therefore, it seems that the level of reducing sugars in batter formulations contributed to Maillard reactions on the crust during frying, and, in turn, on the greater colour development of batters containing extruded flours.

3.4 Instrumental texture analysis

The texture profiles of the different batters are depicted in Fig. 1a and 1b. In these figures, it can be seen that the curve of control sample, presenting a low jagged profile with few peak forces, had a similar shape to those batters containing flour 1. Whereas a highly jagged profile characterised by many force peaks and drops was found for batters containing flour 2 and 3 (intermediate and high extrusion level), especially when they were presented in a higher percentage in the formula (Fig. 1b). This more jagged profile is associated with numerous fracture events, and, then, it describes the behaviour of a crispy or crunchy product (Albert et al., 2009).

It would be expected that the higher moisture content of batter with extruded flours would decrease the crispness (less number of peaks). In fact, previous works have already related a higher moisture content of the crust to a decreased perception of crispness (Baixauli et al., 2003; Primo-Martin et al., 2010). However, samples with similar moisture contents in the crust can have very distinct crispness characteristics since crispness would depend on the water distribution within the crust, a more difficult parameter to measure in these products with a sandwich-like structure (Varela et al., 2008).

In is worth noting that the penetration curves presented two regions: the jagged zone, which was observed in the first part of the profile, and a second half, which hardly presented any fracture events. This could be attributed to the fact that the water distribution in the crust is not homogeneous, which may also help explain why moisture loss does not seem to be correlated with crispness. In fact, the moisture loss is more extreme in the outer layers, which is in agreement with the jagged profile observed here, while the internal regions of the batter are moist and tough rather than crisp (Luyten, Plijter, & Van Vliet, 2004; Varela et al., 2008). Therefore, it could be hypothesised that the higher moisture content with increasing percentages of severely treated extruded flour is mainly due to higher moisture retention in the inner part of their thicker crust (see pick-up values, Table 1); where moisture is more protected from evaporation as a result of the good film forming ability and viscosity of extruded flours. Furthermore, since crusts of battered products have a porous morphology, consisting of films of solid material surrounding air cells (Primo-Martín et al., 2010), the fracture behaviour depends on the distribution and homogeneity of the solid material and on the amount and structure of the air cells (Gibson & Ashby, 1988). Additionally, the greater barrier properties of these viscous extruded flours could have also contributed to enhanced retention of the gas produced by the leavening agents in the batters, bringing about a more aerated/expanded coating, and, thus, explaining the highly jagged profile of the batters. In agreement with this affirmation, Altunakar et al. (2004) reported that starch addition, especially when pregelatinised, enhanced gas entrapment inside the fried product, increasing the volume of the coating and improving texture, due to its greater pick-up and film forming ability.

On the other hand, higher peak force values were obtained for more severely treated extruded flours (flour 2 and 3), especially for 15% level of replacement. Conversely, flour 1 (7.5 and 15% level), with mild extrusion treatment, hardly showed differences in the height of peak force compared to the control sample, with values being only slightly higher at the end of the curve. In this case, the higher force values indicated more resistance to penetration in a less fragile covering (i.e., the presence of a harder coating), which may be associated with the higher pick-up and, possibly greater thickness of batters made with increasing percentages of extruded flours.

3.5 Volatile compounds analysis

43 volatile compounds were determined (Table 2), belonging to most of the chemical classes reported in bakery products (Birch, Petersen, & Hansen, 2014; Pico, Bernal, & Gómez, 2015). For a first exploration of the impact of the use of native or extruded flours (15% replacement) in the elaboration of the batter, a bidimensional PCA was performed (Fig. 2). Regarding the PC1 (60.92 % of the variance), there was a clear separation between unfried batters (positive component) and fried batters (negative component). The main reason was that the fried batters presented higher abundance of the volatile compounds, with the highest content of 33 of the 43 studied volatile compounds. On one hand, the high temperatures applied during frying encouraged the generation of higher abundances of volatile compounds from Maillard reactions (Loewe, 1990), such as 2-methylpyrazine, 2-ethylpyrazine, dimethylpyrazines, 2,3,5-trimethylpyrazine, 2-ethyl-3-methylpyrazine, 2-acetylpyrroline as well as 2-acetylpyrrol or furan derivatives, including furfural, 5-methylfurfural, furfuryl alcohol or 4-hydroxy-2,5-dimethyl-3(2H)-furanone (see Table 2). These high temperatures also promoted caramelisation reactions (Loewe, 1990), which led to the generation of more furfural and 5-methylfurfural from pentose degradation (Ait Ameur, Rega, Giampaoli, Trystram, & Birlouez-Aragon, 2008). Acetoin, acetic acid, benzaldehyde, benzyl alcohol or phenylacetaldehyde were volatile compounds that could have been generated from several origins, being one of them Maillard, and they were also higher in the fried batters than in the corresponding unfried batters. Concretely, acetoin, acetic acid, phenylacetaldehyde can be also generated by fermentation, benzyl alcohol from lipids oxidation and benzaldehyde from both fermentation and lipids oxidation (Pico et al., 2015). The incidence of volatile compounds from fermentation without added yeast is not surprising, since these volatile compounds have been reported in raw flours (Pico et

al., 2017b). Moreover, the small amount of endogenous yeasts, the small amount of free sugars as well as the presence of endogenous α-amylases in flours, can lead to a soft fermentation during mixing even in absence of exogenous yeasts (Martínez-Anaya, 1996). On the other hand, the use of oil for frying justified the higher abundances of volatile compounds from lipids oxidation (LOx), such as nonanal, 1-octen-3-ol, 2-ethyl-1-hexanol, 2-(E)-nonenal or 2,4-(E,E)-decadienal (see Table 2). Nevertheless, LOx and volatile compounds from Maillard reactions of low boiling point (i.e. hexanal, pentanol, 1-hexanol from LOx and 2,3-butanedione, 1-methylpyrrol or pirazine from Maillard) were in lower amount in the fried batters, which can be explained by an evaporation during frying.

Regarding the PC2, there was a clear separation between the control sample and the sample made with the most severely extruded flour (flour 3, both for the unfried and fried batters, not being observable a clear trend for the batters containing flours 1 and 2 (due to their proximity to x axis). In the case of the unfried batters, the LOx and volatile compounds from Maillard reactions mentioned above were, generally, in higher concentration in the unfried batter made with extruded flour 3 (Table 2). This can be explained by the extrusion process, which caused severe starch dextrinisation and protein denaturation (Yaylayan, Fichtali, & van de Voort, 1992), with the subsequent formation of reducing sugars and free amino acids, respectively, that will participate in sugar caramelisation and/or Maillard (Camire et al., 1990), as was explained above. Moreover, the high temperatures produced during extrusion process (especially in flour 3; see 2.1 section) not only encouraged the higher generation of volatiles from Maillard reactions and caramelisation, but also the higher generation of LOx, due to the cleavage of the hydroperoxides produced during the mixing by the lipoxygenases (Camire et al., 1990; Pico et al., 2015). In relation to the fried batters, the opposite effect was observed, and it is the control sample the one with higher content in LOx and volatile compounds from Maillard reactions and caramelisation (Table 2). As mentioned above, during the extrusion process there is a starch dextrinisation that could somehow change the interaction between the volatile compounds and the matrix, hindering the release of volatile compounds during SPME analyses. In fact, even in low amounts, cyclodextrins have been reported during extrusion process of wheat flour (Román, Dura, Martínez, Rosell, & Gómez, 2016), which are known to be encapsulators of flavours (Astray, Gonzalez-Barreiro, Mejuro, Rial-Otero, & Simal-Gándara, 2009) forming stable inclusion complexes (Astray, Mejuto, Morales, Rial-Otero, & Simal-Gándara, 2010). Moreover, as it was observed in the fat content analyses (section 3.2), the fried batter with 15% of flour 3 absorbed slightly less fat than the control sample due to starch pregelatinisation during extrusion (although with small differences), explaining the lower content of hexanal, pentanol, 1-hexanol, nonanal, 1-octen-3-ol, 2-ethyl-1-hexanol, 2-(E)-nonenal and 2,4-(E,E)-decadienal in batter made with flour 3. However, when comparing the LOx between the different fried batters containing extruded flours, flour 3 presented the highest amount of all the LOx, which is justified by the highest content in LOx of the corresponding unfried batter (Table 2). Concerning to the Maillard reaction volatile compounds, it would be expected that the highest content of pyrazines in the control sample would lead to a darker colour of the fried control batter. Pyrazines have been reported as important Maillard compounds in bread crust (Paraskevopoulou, Chrysanthou, & Koutidou, 2012), that should contribute to its colour (Cho & Peterson, 2010). However, it was fried batter containing extruded flour, which presented the darkest colour (Table 1), due to the starch dextrinisation and the release of free sugars. In view of these results, the content of the furan derivatives (furfural, 5-methylfurfural, furfuryl alcohol) was higher in fried batter with flour 3, which can be generated by

Maillard reactions but also by caramelisation (Ait Ameur et al., 2008) and, hence, can also contribute to the colour of the heated food (Hofmann, 1998). Concretely, furfuryl alcohol has been reported to polymerise in acidic conditions to aliphatic polymers that give a brown colouration to the bread (Okaru & Lachenmeier, 2017).

In conclusion, the extrusion process resulted in fried batters that should present lower fatty and rancid notes (Pico et al., 2015) due to their lower amount of LOx and fat absorption. There was also a decrease in the content of pyrazines of the fried batter with extruded flour, thus, its darkest color should be due to the highest content in furfuryl alcohol from Maillard reactions and caramelisation.

3.6 Consumer testing

Sensory evaluation of control batter and batters made with 15% extruded flour incorporation are presented in Table 3. The nuggets coated with batters containing severely treated extruded flours (flour 2 and 3) were found to have a significantly better appearance than the control, which can be related to their darker colour and greater thickness of the coating (higher batter pick-up). Regarding the odour and taste of the batters, although volatile compounds analyses demonstrated aroma differences between batters these differences were not enough to promote significant changes in consumer perceptions since no significant differences were found for these parameters in any of the samples. Considering the texture, although batters made with extruded flours did not present significant differences from the control, the batter made with 15% of flour 2 showed a higher score than that of flour 3. These results can be related to the greater thickness of the batter made with the most severely treated extruded flour (flour 3) and to the different textural profiles of these batters. Thus, the good crispiness of batter made with flour 3 may have been masked by the higher force required to break the crust in the mouth leading to a worse evaluation of this batter compared to that containing flour 2.

All these differences may contribute to the fact that the batter with incorporation of flour 2, with intermediate extrusion treatment, was the most highly rated by panelists in terms of overall acceptability, albeit it did not significant differences with flour 1. However, it should be noted that all batters were highly rated based on the overall acceptability and the highest differences were less than 0.5 points on a scale of 1 to 9. More specifically, although significant, differences between batters made with flour 2 and flour 3 were less than 0.4, and therefore, on an industrial scale, the use of fully gelatinised flour 3 may be preferable in order to achieve a higher pick-up at the expense of a slight loss of organoleptic quality.

4 Conclusions

Results demonstrated that it is possible to use extruded flours in batter formula to obtain batters of quality that satisfy industrial needs. More specifically, partial replacement of native wheat flour by wheat extruded flours in batter formula yielded higher coating pick-up, better external appearance, and good crispy textural properties. Regarding the volatile profiles of the fried batters, the extrusion leads to a decrease in the rancid volatile compounds from lipids oxidation compared to the control sample. In addition, batters made with extruded flour showed good consumer acceptability, which, in some cases, was better than in the control sample.

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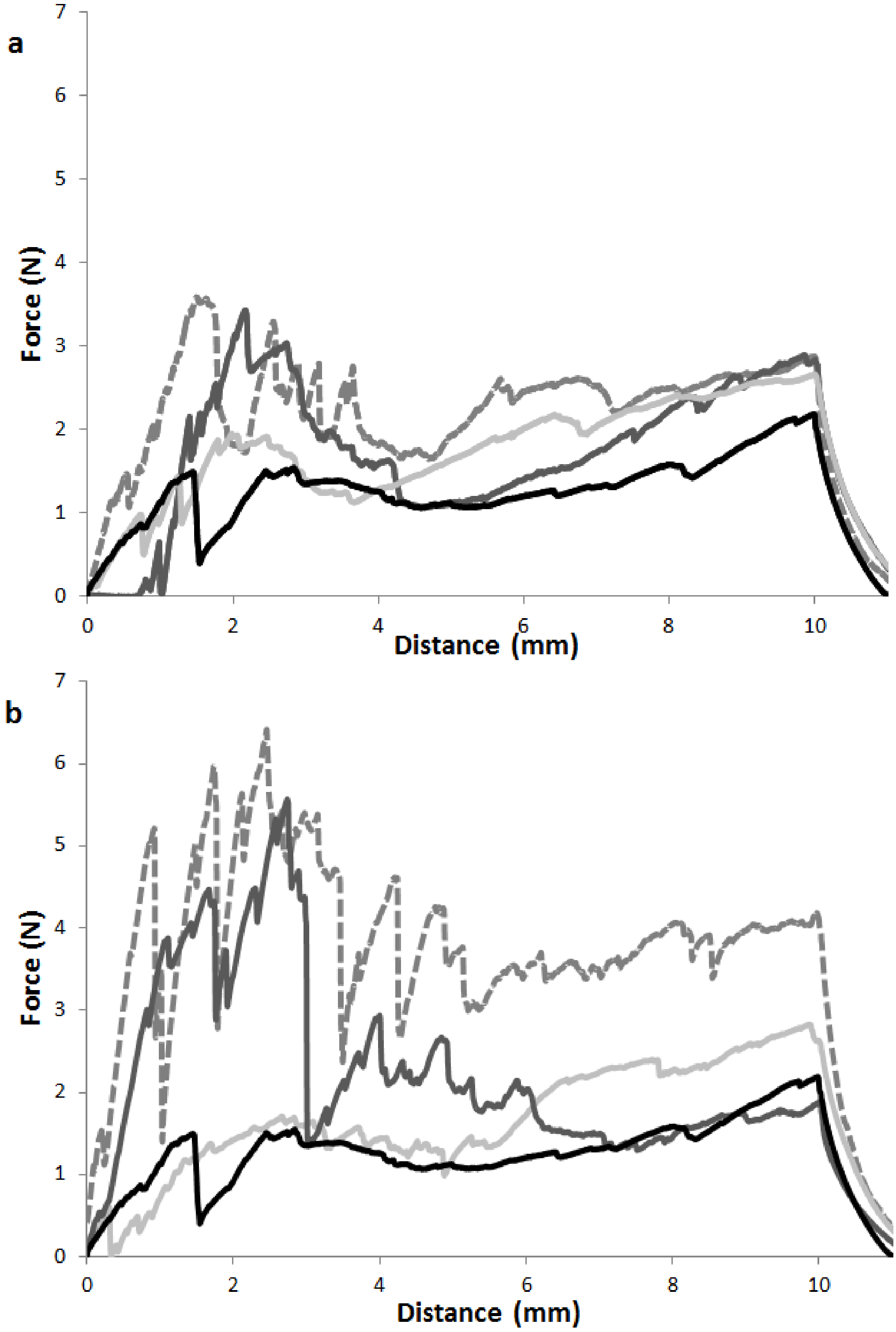


Fig. 1 Texture profile of wheat batters made with 7.5% (a) and 15% (b) replacement by extruded flour. Control sample (black line), Flour 1(light grey line), Flour 2 (dark grey line) and Flour 3 (dashed grey line).

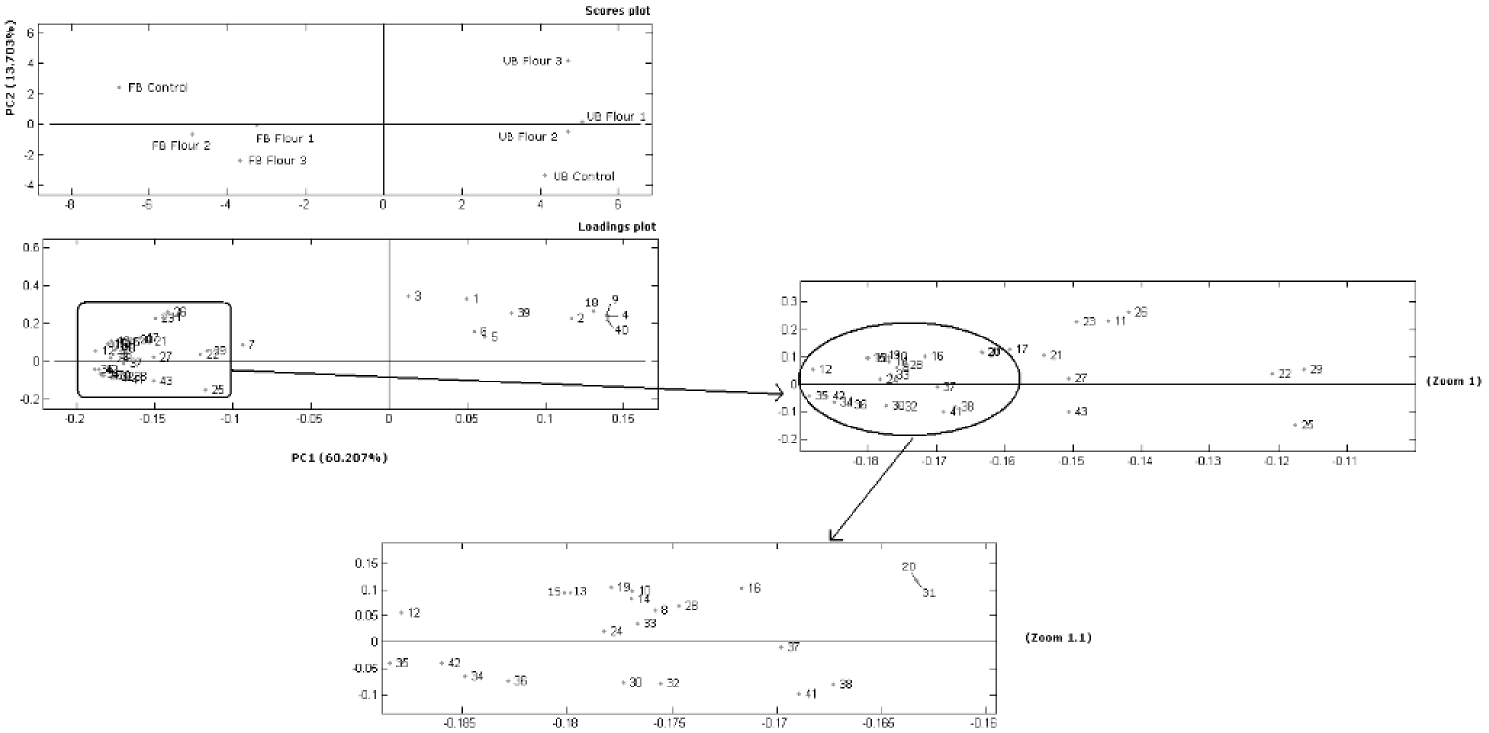


Fig. 2 PCA of the 43 volatile compounds studied in the different unfried (UB) and fried batters (FB). Control batters were elaborated with native flour, while the rest of batters were prepared with 15% of extruded flours of different extrusion levels (Flour 1= mild, Flour 2 = intermediate, Flour 3 = high).

Table 1. Values of batter pick-up, moisture, and fat content and colour parameters of coatings made with different substitution levels and types of extruded flours

Sample	**Pick-up (g/ 100 g)**	**Moisture content (g/100 g)**	**Fat content (g/100 g, db)**	**L***	**a***	**b***
Control	31.49a ± 0.76	14.56a ± 0.12	15.11a ± 0.26	55.25d ± 1.80	5.24a ± 1.33	24.16b ± 1.54
Flour 1 (7.5%)	30.95a ± 2.68	12.42a ± 2.38	14.63 a ± 0.82	53.11cd ± 2.80	6.59ab ± 2.01	20.19a ± 1.71
Flour 2 (7.5%)	32.87ab ± 2.24	15.50ab ± 0.76	15.49 a ± 0.28	51.79bcd ± 3.40	9.47c ± 0.59	20.59a ± 2.46
Flour 3 (7.5%)	41.69c ± 3.05	22.90d ± 0.89	14.41 a ± 0.45	48.35b ± 5.94	9.40c ± 1.10	19.96a ± 3.69
Flour 1 (15%)	32.22ab ± 1.31	18.51bc ± 0.01	16.01 a ± 0.05	52.99cd ± 3.39	7.63b ± 2.34	20.59a ± 1.86
Flour 2 (15%)	36.73b ± 1.91	21.55cd ± 0.30	15.36 a ± 0.86	50.87bc ± 3.28	9.46c ± 1.34	18.80 a ± 4.52
Flour 3 (15%)	46.39d ± 1.89	26.99e ± 0.52	14.62 a ± 0.24	43.72a ± 4.62	9.59c ± 1.61	17.77 a ± 3.77

Values ± standard deviation followed by the same letters within each parameter indicate no significant differences ($p \leq 0.05$).

Table 2. Volatile compounds studied in the different unfried (UB) and fried batters (FB). Control batters were elaborated with native flour, while the rest of batters were prepared with 15% of extruded flours of different extrusion levels (Flour 1= mild, Flour 2 = intermediate, Flour 3 = high).

	UB control	**UB Flour 1**	**UB Flour 2**	**UB Flour 3**	**FB control**	**FB Flour 1**	**FB Flour 2**	**FB Flour 3**
2,3-Butanedione (1)	0.710a ± 0.00652	1.22d ± 0.0282	1.03c ± 0.0217	1.44e ± 0.0412	0.984c ± 0.00253	1.24d ± 0.0783	0.973c ± 0.0106	0.881b ± 0.00426
Hexanal (2)	1.93ab ± 0.0110	6.60e ± 0.312	5.36d ± 0.0396	11.5f ± 1.37	1.65a ± 0.0179	2.66c ± 0.0184	2.45bc ± 0.00930	5.13d ± 0.0423
2-Methyl-1-propanol (3)	0.456a ± 0.0185	0.731d ± 0.0104	0.586b ± 0.00930	1.56g ± 0.0398	0.842e ± 0.0098	0.993f ± 0.00963	0.685c ± 0.00329	0.714d ± 0.0139
1-Methylpyrrol (4)	0.319ab ± 0.0112	5.46e ± 0.171	2.50d ± 0.0714	5.30e ± 0.343	0.643c ± 0.0266	0.420b ± 0.0114	0.387ab ± 0.00989	0.213a ± 0.0159
R-Limonene (5)	0.757a ± 0.00594	6.62f ± 0.0306	6.45f ± 0.00188	3.95d ± 0.419	2.54c ± 0.0586	4.75e ± 0.269	4.65e ± 0.292	1.84b ± 0.0439
Pyrazine (6)	0.172a ± 0.00589	0.745g ± 0.00412	0.676h ± 0.0267	0.498e ± 0.0271	0.397e ± 0.0096	0.430d ± 0.0139	0.607f ± 0.0309	0.275b ± 0.0285
2-Methyl-1-butanol (7)	2.61d ± 0.106	2.43cd ± 0.0250	0.700a ± 0.0184	1.69bc ± 0.0385	3.92f ± 0.276	2.69d ± 0.244	2.53d ± 0.0593	1.07ab ± 1.23
3-Methyl-1-butanol (8)	2.75ab ± 0.0242	3.82b ± 0.0963	1.87a ± 0.172	4.45b ± 0.161	56.9cd ± 1.31	22.4d ± 1.35	20.2c ± 0.478	35.9e ± 1.27
1-Pentanol (9)	0.787a ± 0.00728	5.19f ± 0.169	4.30e ± 0.203	6.43g ± 0.234	1.36d ± 0.0781	1.02b ± 0.0302	1.22c ± 0.0191	1.48d ± 0.00911
2-Methylpyrazine (10)	0.347a ± 0.00799	0.407a ± 0.0101	0.478a ± 0.000271	0.425a ± 0.0217	7.66f ± 0.176	5.11c ± 0.0473	5.12c ± 0.0954	1.75b ± 0.0280
Acetoin (11)	0.724a ± 0.0155	0.882b ± 0.00982	0.865b ± 0.0187	1.48d ± 0.0175	3.23g ± 0.0790	1.69e ± 0.0430	1.65e ± 0.0990	1.13c ± 0.113
2-Octanone (12)	1.03bc ± 0.0288	0.967b ± 0.00735	0.518a ± 0.0242	1.23c ± 0.0227	6.99f ± 0.250	3.76d ± 0.0186	3.91e ± 0.0643	4.62f ± 0.0416

Table 2. (continued)

	UB control	**UB Flour 1**	**UB Flour 2**	**UB Flour 3**	**FB control**	**FB Flour 1**	**FB Flour 2**	**FB Flour 3**
2,5-Dimethylpyrazine (13)	0.176a ± 0.0135	0.282a ± 0.0218	0.254a ± 0.00475	0.412b ± 0.00567	3.06d ± 0.0892	2.51e ± 0.110	2.32d ± 0.0615	1.00c ± 0.0215
2,6-Dimethylpyrazine (14)	0.253a ± 0.0110	0.420a ± 0.00143	0.420a ± 0.0191	0.641b ± 0.0114	2.03g ± 0.0498	2.06d ± 0.177	2.26e ± 0.105	0.999c ± 0.00441
2-Ethylpyrazine (15)	0.157a ± 0.00743	0.207b ± 0.00250	0.216b ± 0.00191	0.346c ± 0.00696	1.95f ± 0.0250	1.58e ± 0.0234	1.66f ± 0.0274	0.670d ± 0.00122
2-Acetyl-1-pyrroline (16)	0.344a ± 0.0254	0.609a ± 0.0175	0.663a ± 0.0191	1.42b ± 0.013	14.1f ± 0.427	4.16c ± 0.000882	5.32d ± 0.237	6.72e ± 0.165
2,3-Dimethylpyrazine (17)	0.194a ± 0.0228	0.277b ± 0.0118	0.303b ± 0.00620	0.342c ± 0.0118	1.40a ± 0.0260	0.526d ± 0.00140	0.527d ± 0.0298	0.732e ± 0.0160
1-Hexanol (18)	2.41b ± 0.197	3.60c ± 0.178	3.27c ± 0.112	9.94d ± 0.825	1.11f ± 0.0804	0.938a ± 0.0420	0.911a ± 0.0260	0.737a ± 0.0337
Nonanal (19)	1.29a ± 0.00560	1.35a ± 0.00940	1.66b ± 0.0264	2.04c ± 0.116	6.28f ± 0.0882	3.31d ± 0.108	3.34d ± 0.115	4.04e ± 0.0346
2,3,5-trimethylpyrazine (20)	0.169a ± 0.00325	0.205b ± 0.0143	0.198b ± 0.0125	0.332e ± 0.0025	0.978f ± 0.0146	0.964f ± 0.00318	0.969f ± 0.0251	0.284c ± 0.0188
2-Ethyl-3-methylpyrazine (21)	0.418c ± 0.00864	0.276a ± 0.00605	0.298ab ± 0.0121	0.403c ± 0.00924	0.794f ± 0.00116	0.625d ± 0.0331	0.748e ± 0.0187	0.315b ± 0.0348
Ethyl octanoate (22)	1.33b ± 0.0339	0.979a ± 0.0199	4.19e ± 0.0675	1.96d ± 0.0949	4.65h ± 0.0471	5.30g ± 0.172	8.65h ± 0.00180	1.63c ± 0.0358
1-Octen-3-ol (23)	1.08a ± 0.0447	2.81b ± 0.0425	3.34c ± 0.0104	4.90e ± 0.157	8.24e ± 0.00626	4.28d ± 0.0172	5.05f ± 0.162	5.50g ± 0.0542
Acetic acid (24)	0.160a ± 0.0110	0.243ab ± 0.0174	0.221a ± 0.00156	0.371b ± 0.00854	5.56a ± 0.0637	4.83d ± 0.0258	8.10f ± 0.148	2.90c ± 0.00257
Furfural (25)	0.232a ± 0.02	0.433a ± 0.05	0.342a ± 0.05	0.548a ± 0.07	3.18b ± 0.159	4.05c ± 0.0888	6.30d ± 0.103	16.8e ± 0.385
2-Ethyl-1-hexanol (26)	1.10a ± 0.0134	2.54b ± 0.0571	3.38c ± 0.0991	4.94f ± 0.118	8.45e ± 0.0139	4.98f ± 0.234	4.51e ± 0.221	4.43d ± 0.108

Table 2. (continued)

	UB control	**UB Flour 1**	**UB Flour 2**	**UB Flour 3**	**FB control**	**FB Flour 1**	**FB Flour 2**	**FB Flour 3**
Benzaldehyde (27)	0.353a ± 0.000883	0.628b ± 0.0107	0.751c ± 0.00503	1.92d ± 0.0158	2.01f ± 0.0623	2.92g ± 0.0271	2.80f ± 0.0687	3.65h ± 0.0137
2-(E)-Nonenal (28)	0.208a ± 0.00694	0.185a ± 0.00266	0.193a ± 0.0187	0.259b ± 0.00476	0.584de ± 0.0210	0.379d ± 0.0277	0.323c ± 0.0153	0.478e ± 0.00278
5-Methyl-2-furaldehyde (29)	0.108a ± 0.00241	0.183b ± 0.000330	0.134a ± 0.00458	0.447e ± 0.0187	0.413a ± 0.000337	0.333c ± 0.00682	0.386e ± 0.00322	0.755f ± 0.0745
Butyrolactone (30)	0.0466a ± 0.00126	0.143ab ± 0.00435	0.257bc ± 0.00891	0.353c ± 0.00993	4.62f ± 0.0483	5.77b ± 0.137	7.28c ± 0.0207	7.98d ± 0.0226
2-Acetylpyrazine (31)	0.780a ± 0.00777	0.527a ± 0.0144	1.58ab ± 0.159	2.11b ± 0.0637	26.4d ± 1.469	8.32d ± 0.0628	7.22c ± 0.0500	10.2e ± 0.149
Butyric acid (32)	0.428ab ± 0.0109	0.407ab ± 0.0122	0.485b ± 0.0149	0.216a ± 0.0139	4.39e ± 0.0481	2.66c ± 0.144	4.37d ± 0.124	6.41e ± 0.264
Phenylacetaldehyde (33)	1.08b ± 0.0163	0.846a ± 0.0192	1.07b ± 0.00729	1.56c ± 0.0794	4.29d ± 0.0117	5.18f ± 0.0222	5.47g ± 0.0289	2.84d ± 0.0775
Furfuryl alcohol (34)	2.15a ± 0.140	1.18a ± 0.00488	1.21a ± 0.0124	1.03a ± 0.00545	58.1b ± 0.901	67.5c ± 1.07	73.9d ± 1.44	80.7e ± 0.0484
2-Methylbutanoic acid (35)	0.284ab ± 0.0165	0.460b ± 0.0260	0.162a ± 0.0172	0.351ab ± 0.0206	3.02d ± 0.162	2.54c ± 0.0738	3.77e ± 0.120	3.44f ± 0.137
3-Methylbutanoic acid (36)	0.439ab ± 0.00923	0.492b ± 0.00893	0.251a ± 0.00766	0.286a ± 0.0139	2.71d ± 0.165	2.59c ± 0.0992	3.97f ± 0.0612	3.63e ± 0.177
2,4-(E,E)-Decadienal (37)	0.982a ± 0.0317	0.213a ± 0.0165	0.780a ± 0.0207	1.00a ± 0.0180	120e ± 1.09	8.06b ± 0.138	103c ± 3.90	108d ± 2.76
Hexanoic acid (38)	0.161a ± 0.00711	0.207a ± 0.0118	0.429b ± 0.00881	0.529b ± 0.00984	1.81c ± 0.0110	2.49d ± 0.0844	3.63e ± 0.0463	3.64e ± 0.167
Benzyl alcohol (39)	0.351d ± 0.00281	0.322bc ± 0.00619	0.375e ± 0.0154	0.690f ± 0.00701	0.315ab ± 0.0147	0.301a ± 0.0161	0.391e ± 0.00659	0.343cd ± 0.0182
Phenylethyl alcohol (40)	0.449f ± 0.0145	0.420e ± 0.00746	0.520g ± 0.00537	0.724h ± 0.0163	0.351c ± 0.00728	0.292a ± 0.00543	0.367d ± 0.0101	0.318b ± 0.00956

Table 2. (continued)

	UB control	**UB Flour 1**	**UB Flour 2**	**UB Flour 3**	**FB control**	**FB Flour 1**	**FB Flour 2**	**FB Flour 3**
2-Acetylpyrrol (41)	0.147b ± 0.00956	0.0603a ± 0.00134	0.0877ab ± 0.00261	0.0983ab± 0.000406	0.840c ± 0.00791	1.29d ± 0.0110	1.90f ± 0.0784	1.62e ± 0.0510
4-Hydroxy-2,5-dimethyl-3(2H)-furanone (42)	0.0504b ± 0.00168	0.0192a ± 0.000391	0.0431b ± 0.000222	0.0339ab±0.000943	0.504d ± 0.0145	0.441c ± 0.00686	0.746f ± 0.0131	0.506d ± 0.0125
4-Vinylguaiacol (43)	0.0932b ± 0.000454	0.0727a ± 0.00118	0.0945d ± 0.00230	0.150c ± 0.00290	0.262d ± 0.00502	0.630f ± 0.00709	0.581e ± 0.0121	0.709g ± 0.0237

Values ± standard deviation followed by the same letters for each volatile compound indicate no significant differences ($p \leq 0.05$). The numbers between brackets after the names of the volatile compounds indicate the numeration followed in the PCA of the Fig. 2.

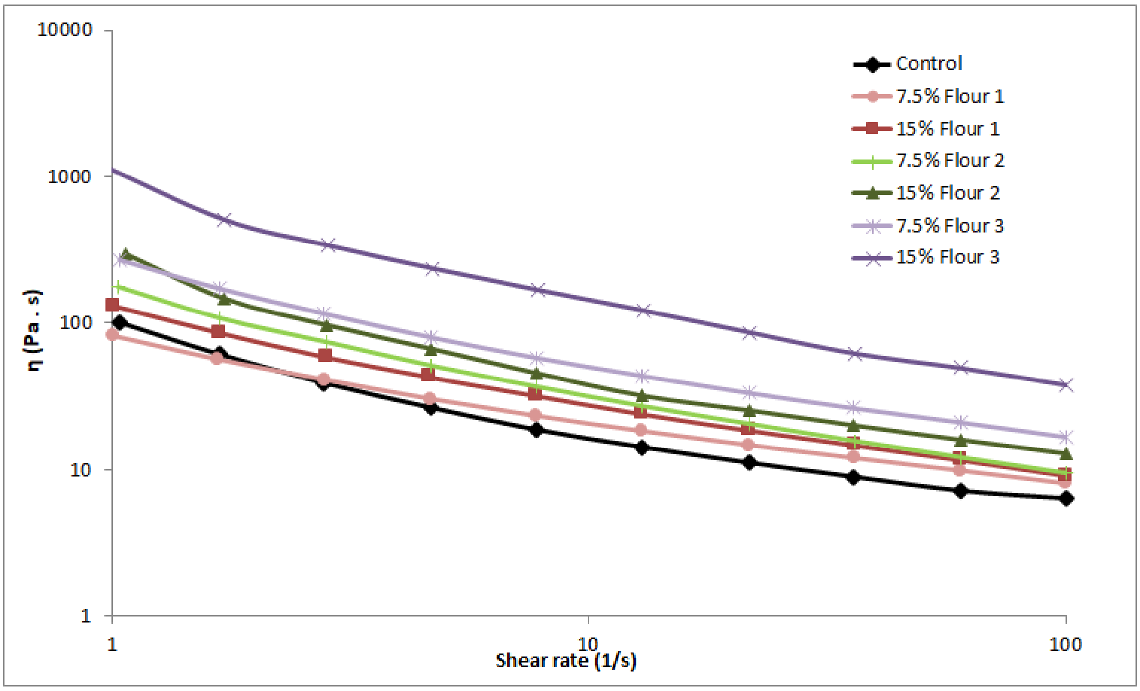
Table 3. Effect of extruded flours on sensorial properties of battered nuggets

Sample	Appearance	Odour	Texture	Taste	Overall acceptability
Control	5.6a ± 1.5	6.4a ± 1.4	6.6ab ± 1.5	6.7a ± 1.4	6.5a ± 1.3
Flour 1 (15%)	6.0ab ± 1.5	6.4a ± 1.4	6.8ab ± 1.5	6.9a ± 1.3	6.7ab ± 1.2
Flour 2 (15%)	6.7c ± 1.4	6.5a ± 1.4	6.9b ± 1.4	7.0a ± 1.4	7.0b ± 1.3
Flour 3 (15%)	6.4bc ± 1.7	6.5a ± 1.2	6.4a ± 1.5	6.7a ± 1.4	6.6a ± 1.3

Values ± standard deviation followed by the same letters within each parameter indicate no significant differences ($p \leq 0.05$). 15% indicates the level of extruded flour replacement in wheat flour based batters.

Supplementary material

Supplementary material 1. Flow curves at 20 ºC of the different extruded flour containing batters.



Supplementary material 2. External structure of fried coatings. From left to right, Control; Flour 1 (15%); Flour 2 (15%); Flour 3 (15%). Control batter was elaborated with native wheat flour, while the rest of batters were prepared with 15% of extruded flours of different extrusion levels (Flour 1= mild, Flour 2 = intermediate, Flour 3 = high).