



22 O). The results showed that the bags heated in a conventional oven had a higher effect  
23 on the increment of odor impact compounds from the packaging than those heated in  
24 microwaves. Aldehydes and ketones were the major responsible for the odors detected  
25 in the olfactometry. Migration experiments to different food simulants (ethanol 10%,  
26 vegetable oil and ethanol 95%) and food samples (chicken) were also performed. In  
27 migration to food simulants, migrants were only detected in ethanol 10 %: 1-nonanol  
28 was detected below the specific migration limit established in the European Regulations  
29 and nonanal and decanal were below 10 ng/g. In migration to chicken a total of 27  
30 compounds, mostly aldehydes, were transferred to it under the worst case migration  
31 conditions.

32

33 **Keywords:** food contact materials; aroma; cooking bags; olfactometry; migration

34

## 35 **1. INTRODUCTION**

36 Lifestyle factors can influence the eating habits of consumers and their way of cooking.  
37 In-pack cooking has been increasingly used, since it is a fast and clean technique that  
38 requires minimum hand manipulation. Since plastic bags used for this purpose are in  
39 contact with food, it must be guarantee that there is no transference of compounds that  
40 could modify food sensory properties or consumers safety. It is well known that plastic  
41 packaging materials are not inert and different interactions between packaging and food  
42 can take place. One of the most important process is migration, defined as the  
43 transference of compounds from packaging to food (Castle, 2007). These compounds  
44 could modify the sensory food properties or cause damage in consumers' health. For

45 this reason, all packaging materials intended for food contact must fulfil the frame  
46 Regulation (EC) 1935/2004 (European-Commission, 2004), whose main principle is  
47 that “Any material or article intended to come into contact directly or indirectly with  
48 food must be sufficiently inert to preclude substances from being transferred to food, in  
49 quantities large enough to endanger human health, or to bring about an unacceptable  
50 change in the composition of the food or a deterioration in its organoleptic properties”.  
51 The most employed materials for the manufacturing of food packaging are plastic  
52 polymers, that must also fulfill Regulation EU/10/2011 for plastic materials intended for  
53 food contact (European-Commission, 2011). This Regulation establishes a positive list  
54 of substances that can be used in the materials manufacturing as well as the migration  
55 tests that must be done before the commercialization of the materials.

56 Some research studies have been focused on the identification and quantification of  
57 migrants that could have adverse effects on human health; (Ibarra, de Quiros, Losada, &  
58 Sendon, 2019). Other authors have focused their work on the study of the sensory  
59 impact of migrating compounds on the packaged food and the determination of the main  
60 odor-active compounds (Vera, Canellas, & Nerin, 2019). These studies have been  
61 performed mostly by gas chromatography-olfactometry.

62 Most of the studies have been performed in polyolefins (polyethylene and  
63 polypropylene), where carbonyl compounds, such as aldehydes and ketones, coming  
64 from oxidation processes, were the main responsible for off-flavors (Bravo, Hotchkiss,  
65 & Acree, 1992; Hopfer, Haar, Stockreiter, Sauer, & Leitner, 2012; Rebeyrolle &  
66 Etievant, 1992; Sanders et al., 2005; Wrona, Vera, Pezo, & Nerin, 2017). Bravo et al  
67 studied the odor-active compounds produced by thermal oxidation of polyethylene  
68 (Bravo et al., 1992), determining that the thermal processing in the presence of oxygen  
69 could lead to the formation of by-products with high aroma impact. Compounds

70 responsible for the wax-like flavor of polyolefins were mainly saturated and unsaturated  
71 aldehydes and ketones (C6-C9) such as hexanal, 1-hepten-3-one, octanal, 1-nonen-3-  
72 one, nonanal, E-2-nonenal and diacetyl. Subsequently, Sanders et al. determined that 8-  
73 nonenal was the main contributor to the “plastic” off-odor in polyethylene packaging  
74 (Sanders et al., 2005). According to the studies performed by several authors (Dzieciol,  
75 2012; Hopfer et al., 2012; Miskolczi, Bartha, & Deak, 2006), high temperature, the  
76 presence of oxygen during processing or the presence of antioxidants are key factors in  
77 the formation of odor-active oxidation species in plastic polymers. The irradiation of  
78 polypropylene has also shown to influence the formation of odor-active compounds  
79 (Tyapkova, Czerny, & Buettner, 2009). In the case of polyethylene terephthalate (PET),  
80 the studies have been focused on migration of odorants from PET bottles to water, being  
81 acetaldehyde the main responsible for off-flavours detected in bottled water (Bach,  
82 Dauchy, Chagnon, & Etienne, 2012). In addition to conventional polymers, odor-active  
83 compounds have also been studied in emerging packaging materials such as starch  
84 (Osorio, Aznar, & Nerin, 2019) or polylactic acid (PLA) (Ubeda, Aznar, & Nerin,  
85 2019).

86 The release of odor compounds from the packaging and its transference to food will be  
87 influenced by external factors such as the temperature, and in this way the new eating  
88 and cooking habits should be considered. Then, in-pack cooking heats not only the food  
89 but also the packaging materials, and the high temperatures could cause modifications  
90 in the composition of the packaging that will affect both, the safety and quality of the  
91 packaged food. The new cooking methods are increasingly used nowadays and this fact  
92 makes necessary to guarantee the consumers health and food sensory properties when  
93 they are used. One of this new methods is in-pack cooking, where PET is commonly  
94 used as packaging material due to the high temperatures that are reached during

95 cooking, especially in the oven. Previous works studied the effect of high temperatures  
96 in the migration from PET food packaging materials, most of them focused on the  
97 migration of PET oligomers (Alin & Hakkarainen, 2013; Lopez-Cervantes, Sanchez-  
98 Machado, Simal-Lozano, & Paseiro-Losada, 2003; Begley & Hollifield, 1990) or  
99 antimony (Haldimann et al., 2013). However, as far as the authors know, the effect on  
100 the release of volatile compounds, and specially odorants, had not been determined. In  
101 this work, the effect of two heating sources (oven and microwave) in the release of  
102 odorant compounds from two different PET cooking bags, has been studied by GC-  
103 MS-O The volatile migrants generated during this process have also been determined  
104 both in food simulant and in a real food sample.

105

## 106 **2. MATERIAL AND METHODS**

### 107 **2.1. Samples**

108 Two different kinds of plastic bags designed for in-pack cooking were studied (B1 and  
109 B2), they were purchased in two different supermarkets. These cooking bags were  
110 intended for cooking different types of meat, such as chicken or pork, as well as fish.  
111 They were made of polyethylene terephthalate.. For oven cooking, the packaging  
112 instructions recommended not to exceed a maximum temperature of 200 °C and cooking  
113 times from 45 to 90 min depending on the food weight. In the case of microwave  
114 cooking, 800 W and times from 25 to 35 min were recommended.

### 115 **2.2. Reagents and SPME fibers**

116 Methanol (LC-MS quality) from Scharlau Chemie (Sentmenat, Spain) and  
117 dichloromethane from Panreac (Barcelona, Spain) were used.

118 Hexanal, m-xylene, 1-octen-3-one, octanal, (E)-2-octenal, 1-nonanol, nonanal, sotolon,  
119 (E)-2-nonenal, estragol, decanal, undecanal, tridecanal, heptanal, 1-octanol, (E)-2-  
120 decenal and tridecane were bought to Sigma-Aldrich (Barcelona, Spain).

121 SPME fibers (PDMS 100  $\mu\text{m}$ , DVD/CAR/PDMS 50/30  $\mu\text{m}$  and CAR/PDMS 75  $\mu\text{m}$ )  
122 were provided by Supelco (Bellefonte, PA, USA).

123

## 124 **2.3 Analysis by GC-MS-Olfactometry of the odorants released from cooking bags**

### 125 **2.3.1. Protocol for the heating of cooking bags**

126 For this study, cut-offs of 2.5 x 2.5 cm of the cooking bags (B1 and B2) were  
127 introduced inside 20 mL glass vials and closed with screw caps in order to avoid  
128 volatiles losses during the heating process. These vials were kept at the most extreme  
129 cooking conditions described in the packaging: 190 °C (to assure that temperatures did  
130 not exceed 200 °C) during 90 min for oven heating and 800 W during 35 min for  
131 microwave heating.

### 132 **2.3.2. Selection of the methodology for the analysis of the main odorants**

133 Different protocols were tested over the bag samples, in order to select the best  
134 methodology for the determination of the main odorants generated during the samples  
135 heating: liquid extraction and direct analysis by HS-SPME. All the analyses were  
136 performed with the samples before and after oven heating (BH and AOH), following the  
137 protocol described in section 2.3.1., and the GC-MS chromatograms were compared.

138 In liquid extraction, the samples were cut in small pieces and 3 consecutive extractions  
139 were made with 3, 2 and 2 mL of the extraction solvent for 1 hour in an ultrasounds

140 bath. The obtained extracts were mixed and evaporated under a gentle nitrogen current  
141 up to 1 g under gravimetric control. Two extraction solvents were tested,  
142 dichloromethane and methanol. Finally 1  $\mu\text{L}$  of the extract was analyzed by GC-MS. In  
143 direct analysis by HS-SPME the samples were directly analyzed in the same 20 mL  
144 vials where they were oven heated by HS-SPME. Three kinds of fibers with different  
145 polarities were tested: PDMS, DVD/CAR/PDMS and CAR/PDMS.. All the experiments  
146 were performed in triplicate.

### 147 **2.3.3. Analysis of the odorants released from cooking bags by SPME-GC-MS-** 148 **Olfactometry**

149 According to the results obtained in the former experiment, the analysis of the odorants  
150 from cooking bags was performed by HS-SPME-GC-MS-O using a DVD/CAR/PDMS  
151 SPME fiber.

152 Materials 1 and 2 were analyzed before heating (BH) and after being oven heated  
153 (AOH). Material 1 was also analyzed after being heated in the microwave (AMH). The  
154 samples were directly analyzed in the same 20 mL vials where they were heated,  
155 without opening them, in order to avoid losses of volatiles. Blanks of the vials, with and  
156 without heating, were also analyzed.

157 For the SPME extraction, the samples were first equilibrated at 80 °C during 15 min  
158 and then, extracted at 80 °C during 20 min. The samples were heated in the heater  
159 module of a Combipal autosampler from Agilent. SPME fiber was desorbed at 250 °C  
160 for 2 min in splitless mode.

161 For the analysis, a gas chromatograph 7820A GC system coupled in parallel to a mass  
162 spectrometer 5977B MSD from Agilent Technologies (Santa Clara, CA, USA) and an

163 olfactory detection port from Phasor GL Sciences (Germany) were used. The column  
164 was a HP-5MS (30m x 25mm x 0.25 $\mu$ m film thickness) from Agilent. The oven  
165 temperature ramp was as follows: initially 40 °C for 5 min, 10 °C min<sup>-1</sup> to 300 °C and  
166 held at 300 °C for 10 min. MS analysis was performed in SCAN mode from m/z 50 to  
167 450. For the olfactometry, the transfer line was heated at 200 °C and the sniffing port  
168 was purged with humidified air. Olfactometries were performed by 5 trained panelists  
169 that described the aroma perceived and its intensity in a scale from 1 (low intensity) to 3  
170 (high intensity), middle values were also allowed. All the panelists performed the  
171 analysis of all samples. They were previously trained using the same methodology as  
172 Osorio et al. (Osorio et al., 2019). In order to compare the aroma impact of the  
173 compounds perceived, the modified frequency percentage (MF %) was calculated  
174 according to the following equation:

$$175 \quad MF (\%) = \sqrt{F(\%) \times I (\%)} \quad \text{Equation 1}$$

176 Where F is the frequency of perception and I the average intensity, both expressed as  
177 percentage. Compounds with MF% values above 50% were considered relevant for the  
178 global aroma of the material (Osorio et al., 2019; Wrona et al., 2017).

#### 179 **2.3.4. Identification of odorant compounds**

180 The identification of a detected compound was initially performed by comparison of its  
181 mass spectrum with those reported in NIST v2.2 library. A candidate was *confirmed by*  
182 *NIST*, when the match value between the mass spectrum of the compound and the  
183 proposed candidate (matching values from 0 to 1000) was above 800. The retention  
184 index (RI) of the detected compounds was also calculated. For this purpose, a solution  
185 of alkanes from C<sub>7</sub> to C<sub>40</sub> was injected under the same conditions as the sample and the  
186 RI was calculated. A candidate was *confirmed by RI* when the relative difference



187 between its calculated RI value and RI value from the bibliography was less than 5%.  
188 Bibliography databases consulted were [www.flavornet.org] or  
189 [www.thegoodscentcompany.com]. These databases were also consulted in order to  
190 know the aroma description of the candidates previously reported in the literature. Only  
191 compounds with similar descriptions to those detected by the panelists were taken into  
192 account for the identification.

193 Finally, when the standard of the compound was available, it was injected in the same  
194 conditions as the sample. When there was a good match of retention time and mass  
195 spectra between the compound and the standard, the candidate was considered  
196 *confirmed by standard*.

## 197 **2.4. Analysis of migration from cooking bags**

### 198 **2.4.1. Migration assays**

199 Migration assays were performed in food simulants as well as in real food. The  
200 following food simulants were used: ethanol 10% (simulant A), vegetable oil  
201 (sunflower oil as simulant D2) and ethanol 95% as simulant D2 substitute. The selection  
202 was based on the Regulation EU/10/2011 and according to the intended uses of these  
203 cooking bags, such as chicken or fish. First, a cut-off of 1 x 5 cm of the sample  
204 materials (1 and 2) was immersed in a 20 mL vial containing 9 g of the food simulant.  
205 Then, the vial was closed with a screw cap and introduced in the oven for carrying out  
206 the migration test. Since according to the results obtained in the analysis of the  
207 odorants, oven cooking provided a higher release of volatiles, this cooking method was  
208 chosen for migration experiments. Migration test with vegetable oil simulant was  
209 performed at the worst case conditions, 190 °C during 90 min. Migration tests with  
210 ethanol 10 % were performed at 100 °C during 90 min. These conditions were selected

211 according to Regulation EU/10/2011 that establishes that when migration temperatures  
212 exceed 100 ° C, contact temperature for ethanol 10% must be replaced by a test at 100 °  
213 C. In the case of ethanol 95%, substitute of simulant D2, the migration conditions of 6  
214 hours at 60 °C were selected according to the Guidelines on testing conditions for  
215 articles in contact with foodstuffs (European-Comission, 2009). A blank of the  
216 simulants was also simultaneously submitted to the same time-temperature conditions.

217 For the migration assays with food, chicken breasts were purchased in a retail store. In  
218 order to have a homogeneous sample, a chicken breast was cut in 3 similar size pieces;  
219 one of them was cooked in the oven without the plastic bags (blank), and the others  
220 wrapped in the cooking bags 1 and 2. The cooking conditions were selected according  
221 to the meat weight (190 °C, 20 min).

#### 222 **2.4.2. Analysis of migration samples by HS-SPME-GC-MS**

223 In migration to food simulants experiments, the migration vials were withdrawn from  
224 the oven, cooled at room temperature and opened. Then, the cooking bag samples were  
225 removed from the migration solution. The vials were immediately closed and migration  
226 solutions were analyzed by HS-SPME-GC-MS. In the case of ethanol 95% food  
227 simulant, solutions were previously diluted 10 fold with water and an aliquot of 9 g of  
228 solution was introduced in the 20 mL vials, for its analysis. In migration to chicken  
229 samples, 2.50 g of chicken (migration samples or blanks) were cut in small pieces and  
230 introduced in 20 mL glass vials. Then, the vials were closed with screw caps for its  
231 analysis by HS-SPME-GC-MS.

232 The analysis of food simulants and chicken was performed under the same conditions as  
233 those used for the analysis of cooking bags described in section 2.3.2. All the analyses  
234 were done in triplicate.

### 235 **2.4.3. Determination of migrants**

236 For determining the migrants present in the different simulants two strategies were  
237 followed. First, in order to check either the presence of new peaks or a significant  
238 increment in the peaks already present in the blank, chromatograms of migration  
239 solutions and migration blanks were overlaid and visually compared. Then, due to the  
240 complexity of some matrices, such as vegetable oil and chicken, a targeted analysis was  
241 performed. This analysis was focused on the volatiles released from cooking bags after  
242 being heated. For this purpose, chromatograms of the cooking bags obtained by HS-  
243 SPME-GC-MS before and after being oven heated were overlaid and visually  
244 compared. A list of the compounds released during heating was created (Supplementary  
245 Material 1). This list includes 72 compounds, their retention time, their identification  
246 based on NIST library match value, and the masses used for its confirmation. The listed  
247 compounds were searched in both vegetable oil, chicken migration samples and their  
248 blanks, and the areas of the peaks were measured. A t-student test was performed in  
249 order to know if there were significant differences between blanks and migration  
250 samples.

251 In food simulants, significant differences between samples and blanks were only found  
252 in ethanol 10%. The quantification in this simulant was performed by external  
253 calibration. Calibration curves were prepared in ethanol 10% and analyzed by HS-  
254 SPME-GC-MS following the procedure described in section 2.3.3. Table 2 shows the  
255 analytical parameters. All the analyses were done in triplicate.

256 For the analysis of chicken migration, a calibration curved was built spiking chicken  
257 blank samples (chicken cooked in the oven without cooking bag at 190°C – 20 min) at  
258 different concentration levels. For this purpose, aliquots of 2.5 g of chicken were cut in

259 small pieces, introduced in 20 mL vials and spiked with 20  $\mu$ L of the spiking solutions.  
260 The vials were closed with screw caps and analyzed by HS-SPME-GC-MS. Table 3  
261 shows the analytical parameters found. All the analyses were done in triplicate.

## 262 **2.5. Sensory analysis**

263 The panelists were 4 men and 11 women with ages between 22 and 65 years old and  
264 with experience in sensory tests. Sensory tests were only performed on odor  
265 (orthonasally) in all cases. The tests were performed in a room with no odor  
266 interferences.

267 First, panelists performed a triangle test where the differences in the aroma perception  
268 of cooking bags before and after the oven heating process were evaluated. For this test,  
269 cut-offs of the plastic bags (2.5 x 2.5 cm) were introduced in glass vials (20 mL) that  
270 were closed with screw caps. Half of them were introduced in the oven at the previously  
271 described conditions, 190  $^{\circ}$ C during 90 min (AOH) and the other half was not heated  
272 (BH). Afterwards, samples were codified with a 3 digits code and presented to the  
273 panelists for the triangle test. Each panelist performed 2 triangle tests (2 AOH + 1 BH, 1  
274 AOH + 2 BH). Finally, the heated samples were presented to the panelists for a free  
275 description of the main aroma notes perceived.

276

## 277 **3. RESULTS AND DISCUSSION**

### 278 **3.1. Identification of the odorants released from the cooking bags after heating**

279

280

281 **3.1.1. Methodology selected for the analysis of the main odorants**

282 The results obtained by the tested methodologies showed that there were clear  
283 differences in the volatile compounds composition of cooking bags before and after  
284 heating. These differences were detected in a higher extent in direct analysis by HS-  
285 SPME rather than with liquid extraction. Among the 3 different fibers used,  
286 DVD/CAR/PDMS provided the most intense peaks. Probably, because this fiber  
287 includes 3 different adsorbents and allows the extraction of compounds with different  
288 polarities. For this reason, this fiber was selected for further experiments. Figure 1  
289 shows the chromatogram obtained by HS-SPME-GC-MS of a cooking bag before and  
290 after being heated in the oven.

291 **3.1.2. Identification of the odorants released from the cooking bags during oven**  
292 **cooking**

293 The triangle sensory test allows knowing if there are perceivable differences between 2  
294 samples. A small probability value ( $p$ -value  $< 0.05$ ) evidences the existence of  
295 significant differences between them. The results obtained from the cooking bags  
296 (before and after oven heating) showed significant differences among them. 28 out of  
297 30 of the answers provided by the panelists were correct providing a  $p$ -value  $< 0.001$   
298 (Meilgaard, Civille, & Carr, 1999). These differences were described by the tasters with  
299 the notes: plastic, burnt, rubber, burnt oil, closed and old.

300 In order to know the volatile compounds responsible for these aroma notes, the samples  
301 were analyzed by HS-SPME-GC-MS-O. Table 1 shows the compounds detected in the  
302 olfactometries of both cooking bags (B1 and B2) after being submitted to the oven  
303 heating (AOH). Only odorous compounds with %MF values above 30% in at least, one  
304 of the samples are shown in the table. Regarding the materials before being submitted to

305 high temperatures, olfactometries did not show odor regions with MF% over 30% and  
306 therefore no data have been included in the table. Very similar results were found for  
307 both materials, B1 and B2. The results showed a total of 28 different odor regions where  
308 the largest proportion corresponded to aldehydes and ketones.

309 Compounds with the highest %MF values (above 80%) and therefore, the maximum  
310 responsible for the notes detected after the heating process were: 1-octen-3-one  
311 (*mushroom*), octanal (*lemon, green*), 3,5-octadien-one (*mushroom*), nonanal (*cucumber,*  
312 *green*), sotolon (*curry*), (E)-2-nonenal (*chemical, lipstick*), decanal (*cucumber, green*)  
313 and (E,Z)-3,6-nonadienol (*unpleasant, metallic*). Ketones and aldehydes have been  
314 described in previous works as compounds coming from polyolefin oxidation (Hopfer et  
315 al., 2012) (Bravo et al., 1992; Sanders et al., 2005) and 1-octen-3-one was also found in  
316 irradiated polypropylene (Tyapkova et al., 2009). Some of these compounds have been  
317 detected also in non-polyolefin polymers. (E)-2-nonenal has been linked to the  
318 autooxidation of fatty acids present in PVC lubricants (Wiedmer et al., 2017) and  
319 sotolon was described by Osorio as one of the main responsible for aroma of starch-  
320 base (Osorio et al., 2019) and PLA based films (Ubeda et al., 2019). Compounds with  
321 %MF values above 70%, and therefore with also a considerable impact in the final  
322 aroma, were: isoborneol, estragol,  $\gamma$ -octalactone, undecanal, (E)-2-undecenal, 4,5-  
323 epoxydec-2-enal and dodecanal.  $\gamma$ -Octalactone had been previously defined as a product  
324 of polyolefin oxidation (Hopfer et al., 2012) and 4,5-epoxydec-2-enal had been detected  
325 in packaging labels (Landy, Nicklaus, Semon, Mielle, & Guichard, 2004) and  
326 polypropylene (Tyapkova et al., 2009) .

327 Figure 1 shows a chromatogram obtained by HS-SPME-GC-MS of cooking bag 1  
328 before and after being submitted to oven heating. As it can be observed, the number of

329 peaks detected significantly increased after submitting the material to high  
330 temperatures, which was expected, due to the possible degradation processes linked to  
331 high temperatures (Bravo et al., 1992; Dzieciol, 2012; Hopfer et al., 2012; Miskolczi et  
332 al., 2006).

333 A total of 72 volatile compounds were identified in cooking bags after heating, they are  
334 displayed in Supplementary Material 1. Aldehydes were the major compounds, even  
335 though ketones, alcohols and alkenes also increased due to high temperatures.

### 336 **3.1.3. Identification of the odorants released from the cooking bags during** 337 **microwave cooking**

338 Since cooking bags were also intended to microwave cooking, the odorants released  
339 during this process were studied. Table 1 shows MF% values when the cooking bag 1  
340 was submitted to microwave heating (B1\_AMH). The results showed a lower intensity  
341 of the odorous compounds compared to those released after oven heating. These results  
342 were expected, since the temperature reached in MW was not as high as in the  
343 conventional oven. The same influence of the heating source was previously observed  
344 in the migration of different photoinitiators to Tenax® (Ji et al., 2019). Only 5  
345 compounds obtained MF% values above 50%: 1-octen-3-one, nonanal, isoborneol, (E)-  
346 2-decenal and (E)-2-undecenal; and only 2 out of them obtained MF% values above  
347 80%, 1-octen-3-one and isoborneol.

348 If the sum of the MF% is considered, the value obtained for the samples submitted to  
349 the oven temperatures, 1793 for B1 and 1872 for B2, is more than twice the value of  
350 samples submitted to the microwave heating, 704 for B1. Therefore, the release of off-  
351 flavors from the plastic bag will be much higher when bags will be used for cooking in  
352 a conventional oven..

#### 353 **3.1.4. Changes in the sensory notes of cooking bags during cooking**

354 The compounds detected in the olfactometries were classified in 6 categories according  
355 to their aroma: chemical-solvent (1); green-aldehyde-lemon (2); roasted-burnt (3);  
356 mushroom (4); spicy-liquorice (5) and pleasant-sweet-flowery (6). The sum of MF%  
357 values was calculated for each category in order to evaluate the differences in the  
358 different aromas among samples. The results are shown in figure 2. The three samples  
359 showed a similar profile, although the bags submitted to microwave heating obtained a  
360 lower intensity in the descriptors. In all cases, the notes green-aldehyde-lemon and  
361 chemical-solvent had a predominant role in the aromas produced due to high  
362 temperatures. It has to be taken into account that other compounds with similar aromatic  
363 notes with low MF% values, could enhance the perception of these descriptors. This is  
364 the case of other aldehydes produced during heating. Figure 3 shows the areas of the  
365 aldehydes detected by HS-SPME-GC-MS in bag 1 before and after oven heating. As it  
366 can be seen, there was a significant increment in all aldehydes detected, and a similar  
367 profile was observed in bag 2. All these aldehydes will also probably contribute to the  
368 green-aldehyde-lemon global notes.

#### 369 **3.2. Migration from cooking bags to food simulants**

370 The study of migration from the cooking bags to different food simulants was focused  
371 not only on the odorant compounds but also on the determination of other volatile  
372 migrants, since, depending on their toxicity and concentration, their presence in food  
373 could be a risk for human's health. As it was reported in section 2.4.3, the presence of  
374 all the volatile compounds released from cooking bags was checked in vegetable oil and  
375 chicken. Supplementary material 1 shows the list of the 72 targeted compounds, their  
376 retention time, NIST match value, confirmation and quantification ions.



377 In simulant A (ethanol 10%), 3 compounds were found in migration, nonanal, 1-  
378 nonanol and decanal. The results from migration are shown in table 2. Nonanal and  
379 decanal are not listed in Regulation EU/10/2011, therefore they should not migrate at  
380 detectable quantities, what means at quantities above 10 ng/g. In both cases, the values  
381 found were below 2 ng/g. 1-nonanol is listed in the Regulation with no SML, and it was  
382 found in both cases below 5 ng/g. According to the literature, the detection threshold of  
383 these compounds in water solutions is: 2.8 ng/g for nonanal (Czerny et al., 2008), 5 ng/g  
384 for decanal (Rychlik, Schieberle, & Grosch, 1998) and 0.1 µg/g for 1-nonanol (Sheftel,  
385 2000). Since the concentration values found in migration were in all cases below these  
386 values, no changes in the food aroma are expected. In ethanol 95%, no significant  
387 differences between migration samples and migration blanks were observed. Since  
388 ethanol 95% migration samples were 10 fold water diluted previous to its analyses, it  
389 can be stated that the concentration of nonanal, 1-nonanol and decanal in migration was  
390 below 10 times the limit of detection calculated for ethanol 10% solutions (nonanal <  
391 0.50 ng/g; 1-nonanol < 10.0 ng/g and decanal LOD < 0.10 ng/g).

392 In simulant D2 (vegetable oil) when chromatograms of migration samples and migration  
393 blank were overlapped, no visual differences were observed (Supplementary material  
394 2). The main compounds detected in the chromatogram were aldehydes, such as  
395 hexanal, (E)-2-heptenal, (E)-2-octenal, (E)-2-decenal or 2,4-decadienal. Due to the  
396 complexity of the matrix a target analysis of the 72 compounds detected in the cooking  
397 bags was performed. This analysis showed the presence of 24 compounds  
398 (Supplementary material 1), most of them also aldehydes. A bar chart of the average  
399 areas of these compounds in migration samples (cooking bags 1 and 2) and blank is  
400 shown in Supplementary Material 3. A t-student test was performed in the area values in  
401 order to detect significant differences between samples and blank. The results from the

402 statistical test did not reflect significant differences ( $p$ -value  $> 0.05$ ), and consequently,  
403 the contact with the cooking bag was not the origin of these compounds in the vegetable  
404 oil. In the case of aldehydes, it has to be taken into account the high aldehydes content in  
405 vegetable oil (Cao, Ruan, Chen, Hong, & Cai, 2017), especially if it is submitted to high  
406 temperatures (Katragadda, Fullana, Sidhu, & Carbonell-Barrachina, 2010). This fact  
407 was observed for aldehydes such as heptanal, (E)-2-nonenal or (E)-2-undecenal..

### 408 **3.3. Migration from cooking bags to chicken**

409 A chromatogram of migration to chicken samples is shown in Supplementary material  
410 4. Aldehydes were also the main compounds detected in this sample. In this case,  
411 nonanal was the aldehyde with the highest intensity, followed by hexanal and 1-octen-3-  
412 ol. In the targeted analysis, 28 compounds were detected, 27 out of them showed  
413 significant differences ( $p$ -value  $< 0.01$ ) between the samples and the blank, a bar chart  
414 of the average areas of these compounds is shown in Supplementary Material 5. The  
415 migrants were quantified and the concentration values are shown in table 4. Migration  
416 values were recalculated according to the EU/10/2011 Regulation rate,  $6 \text{ dm}^2 / 1 \text{ Kg}$   
417 food. None of the migrants, except for benzaldehyde, 1-octanol and 1-nonanol, were in  
418 the positive list of the Regulation and therefore they should not be detectable, it means  
419 that concentrations should be below  $10 \text{ ng/g}$ , what is not fulfilled in most cases. It has to  
420 be taken into account that migration experiments were performed in the most adverse  
421 conditions and that the contact between chicken and cooking bags was quite high  
422 compared to a real in-pack chicken cooking where bigger pieces are cooked.

423

## 424 **4. Conclusions**

425 The high temperatures used during oven cooking lead to the formation of high impact  
426 aroma compounds such as aldehydes and ketones in the cooking bag. In the case of  
427 microwave cooking, the formation of high impact aroma compounds was considerably  
428 less. Its sensory description was related to green-aldehyde-lemon and chemical-solvent  
429 notes. The presence of these compounds could modify the initial perception of the  
430 consumers once the cooking bag is opened, and produce a disgusting effect to them..  
431 When migration was evaluated, the results found in food simulants did not show risks  
432 for consumers' health. In the case of migration to chicken, the results reveal that  
433 compounds generated from cooking bags due to the extreme temperatures reached in the  
434 oven could be transferred to food and therefore a control of these materials is needed.

435

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440

441 Potential conflicts of interest do not exist.

442

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576

577 **Figure captions**

578 **Figure 1.** Chromatogram obtained by HS-SPME-GC-MS of cooking bag 1 before and  
579 after being submitted to oven heating. Compounds: (1) hexanal; (2) heptanal; (3) E-2-  
580 heptenal; (4) 2-pentyl furan; (5) 2-ethyl-1-hexanol; (6) E-2-octenal; (7) 1-octanol; (8) 2-  
581 nonanone; (9) nonanal; (10) 1-nonanol; (11), 1-methyl-cyclododecene; (12) 2-decanone;  
582 (13) dodecane; (14) decanal; (15) benzothiazole; (16) E-2-decenal; (17) undecanal; (18)  
583 E-2-undecenal; (19) tetradecane; (20) dodecanal; (21) non identified; (22) dodecanol;  
584 (23) tridecanal; (24) nonylcyclohexane; (25) cetene; (26) tetradecanal; (27) 2-  
585 pentadecanone; (28) pentadecanal; (29) 1-octadecene; (30) hexadecanal; \*siloxanes.

586 **Figure 2.** Spider graph of the main aroma categories perceived during GC-O in cooking  
587 bags after being submitted to oven heating (B1\_AOH, B2\_AOH) or microwave heating  
588 (B1\_AMH).

589 **Figure 3:** Intensity of aldehydes in cooking bag 1 before and after being submitted to  
590 oven heating (B1\_BH and B1\_AOH).



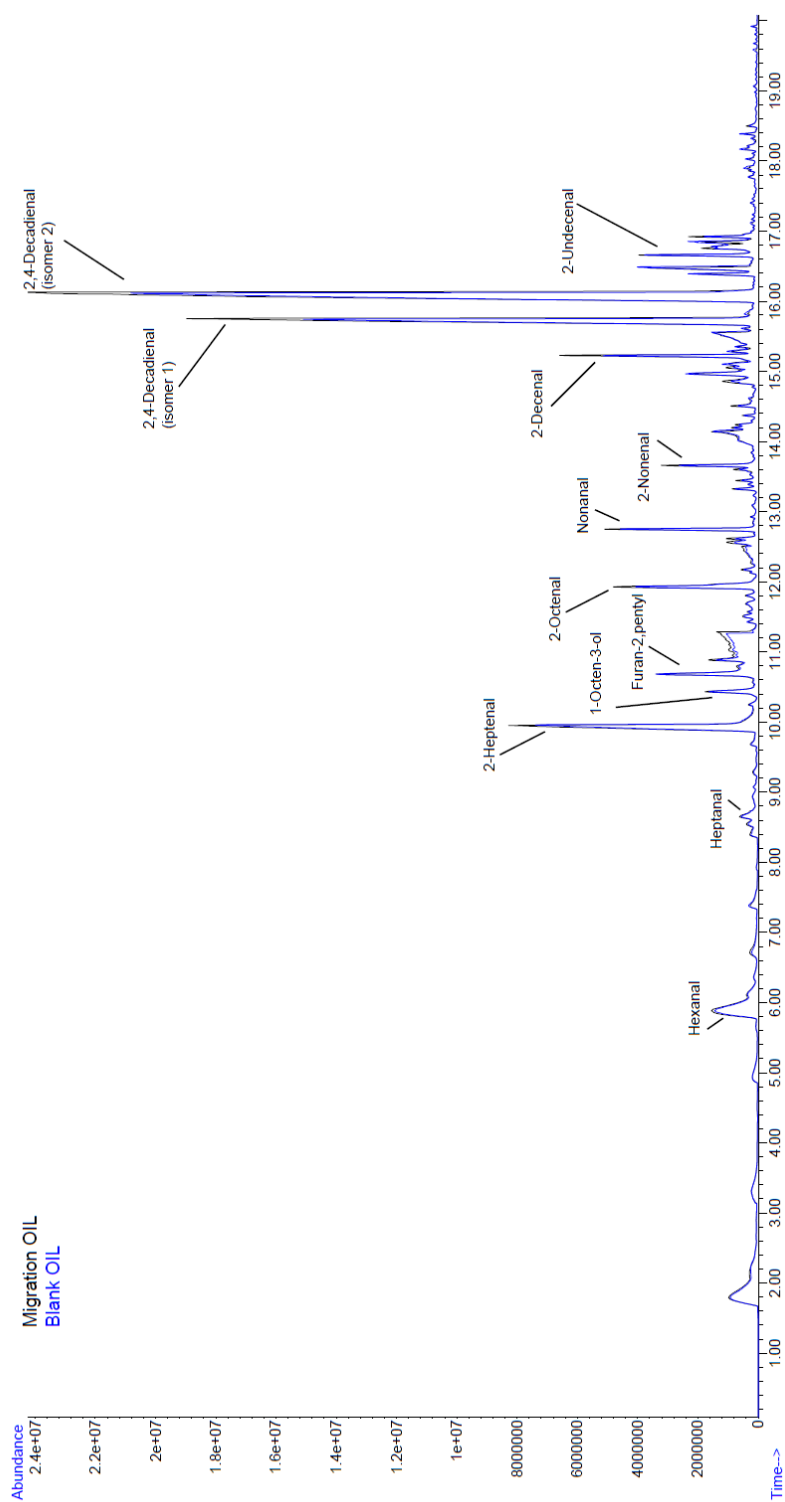
**Supplementary material 1.** Compounds whose signal in HS-SPME-GC-MS analysis increased when cooking bags were oven heated, retention time (RT), NIST match value, quantification ion (QI) and confirmation ions (CI1 and CI2) used for its determination and its presence in migration to vegetable oil (simulant D2) and chicken.

	RT	Candidate	NIST Match	QI	CI 1	CI2	Vegetal Oil	Chicken
C1	5.88	Hexanal	839	56.1	72.1	82.1	X	X
C2	8.65	Heptanal	913	70.1	55.1	81.1	X	X
C3	8.86	Oxime-, methoxy-phenyl-	879	133.0	151.0	86.0		
C4	9.90	(E)-2-Heptenal	952	83.1	70.1	97.0	X	X
C5	9.97	Benzaldehyde	855	105.0	77.0	51.0	X	X
C6	10.12	2H-Pyranmethanol, tetrahydro-2	778	113.1	95.1	59.1		
C7	10.24	Formic acid, heptyl ester	770	70.1	56.1	83.1	X	X
C8	10.42	1-Octen-3-ol	828	57.1	99.1	85.1	X	X
C9	10.68	Furan, 2-pentyl-	802	81.1	138.1	57.1	X	X
C10	11.41	1-Hexanol, 2-ethyl-	816	57.1	83.1	98.1		
C11	11.78	2(3H)-Furanone, 5-heptyldihydro-	942	85.0	57.1	100.0	X	
C12	11.92	(E)-2-Octenal	893	70.1	83.1	97.1		X
C13	12.06	Acetophenone	951	105.1	77.1	120.1		
C14	12.16	1-Octanol	884	56.1	69.1	84.1	X	X
C15	12.53	2-Nonanone	821	58.1	71.1	142.1		
C16	12.74	Nonanal	942	57.1	98.1	114.1	X	X
C17	13.29	2-Oxepanone	824	55.1	84.1	114.1		
C18	13.65	(E)-2-Nonenal,	924	55.1	70.1	96.1	X	X
C19	13.72	$\alpha$ -Campholenal	743	108.1	95.0	85.1		
C20	13.83	1-Nonanol	923	56.1	70.1	97.1	X	X
C21	13.94	1-methyl-cyclododecene	746	97.1	68.1	180.2		
C22	14.11	Ethanol, 2-(2-butoxyethoxy)-	782	57.1	128.1	75.0		
C23	14.17	2-Decanone	770	58.1	71.1	156.1	X	X
C24	14.29	Dodecane	929	57.1	71.1	170.2	X	X
C25	14.37	Decanal	936	57.1	70.1	128.1	X	X
C26	14.71	Benzothiazole	792	135.0	108.0	69.0		
C27	14.93	NI (RI: 1239)		85.1	55.1	147.1	X	
C28	15.14	$\gamma$ -Dodecalactone	800	85.0	57.1	69.1		
C29	15.20	(E)-2-Decenal	935	70.1	55.1	136.1	X	X
C30	15.27	Octan-2-one, 3,6-dimethyl-	776	55.1	83.1	154.1		
C31	15.34	1-Decanol	884	70.1	83.1	97.1		
C32	15.67	(E,Z)-2,4-Decadienal	836	81.0	95.1	180.2		X
C33	15.76	Tridecane	886	57.1	71.1	85.1		X
C34	15.81	Formamide, N,N-dibutyl-	876	72.1	114.1	157.1		
C35	15.85	Undecanal	966	57.1	82.1	126.1	X	X

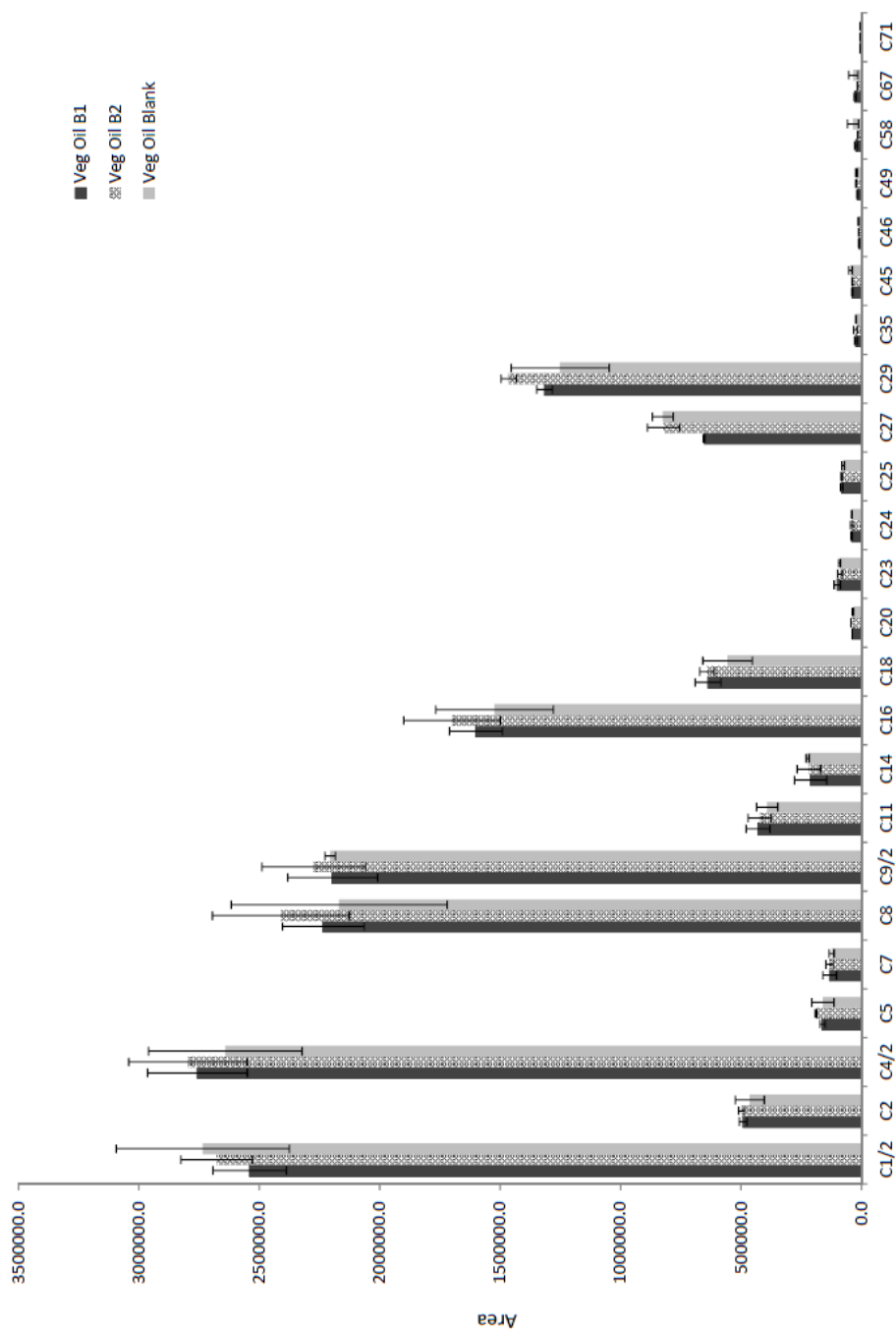
	RT	Candidate	NIST Match	QI	CI 1	CI2	Vegetal Oil	Chicken
C36	16.00	(E,E)-2,4-Decadienal	910	81.1	152.1	95.1		<b>X</b>
C37	16.28	NI (RI: 1338)		57.1	71.1	182.2		
C38	16.35	Trimethyl-cyclohex-2-en-1-ol	740	84.0	125.1	97.1		
C39	16.40	Heptylcyclohexane	834	83.1	55.1	182.2		
C40	16.63	(E)-2-Undecenal	813	70.1	83.1	121.1		<b>X</b>
C41	16.74	1-Octanol, 2-butyl-	898	57.1	83.1	169.2		
C42	16.82	Alcane (RI: 1368)		71.1	97.1	127.1		
C43	17.01	NI (RI: 1392)	892	55.1	83.1	111.1		
C44	17.04	2-Dodecanone	864	58.1	71.1	184.2		
C45	17.11	Tetradecane	956	57.1	85.1	198.2	X	<b>X</b>
C46	17.23	Dodecanal	971	57.1	82.1	140.2	X	<b>X</b>
C47	17.40	Epiglobulol	827	161.1	189.1	204.1		
C48	17.61	Alcane (RI: 1438)		57.1	85.1	183.2		
C49	17.78	NI (RI 1451)	885	83.1	69.1	183.2	X	
C50	18.05	Dodecanol	931	55.1	71.1	140.1		
C51	18.29	1-Pentadecene	944	83.1	97.1	111.1		<b>X</b>
C52	18.33	2-Tridecanone	849	58.1	71.1	85.1		
C53	18.51	Tridecanal	927	57.1	82.1	154.2		<b>X</b>
C54	19.02	NI (RI: 1550)		85.1	197.2	71.1		
C55	19.07	n-Nonylcyclohexane	873	83.1	55.1	210.2		
C56	19.49	Cetene	920	83.1	97.1	224.2		
C57	19.56	NI (RI: 1595)		58.1	149.0	177.1		
C58	19.59	Hexadecane	930	57.1	71.1	226.2	X	
C59	19.74	Tetradecanal	940	57.1	82.1	168.2		<b>X</b>
C60	20.26	Cyclopentane, undecyl-	822	69.1	83.1	224.3		
C61	20.46	1-Tetradecanol	933	55.1	83.1	168.1		
C62	20.64	NI (RI: 1689)		71.1	197.1	212.1		
C63	20.71	2-Pentadecanone	887	58.1	71.1	226.2		
C64	20.89	Pentadecanal	969	57.1	82.1	182.2		<b>X</b>
C65	21.44	NI (RI: 1765)		83.1	55.1	185.1		
C66	21.73	1-Octadecene	917	83.1	97.1	252.4		
C67	21.81	Octadecane	850	57.1	71.1	97.1	X	
C68	21.98	Hexadecanal	935	57.1	82.1	196.2		X
C69	22.85	2-Heptadecanone	892	58.1	71.1	254.2		
C70	23.04	Heptadecanal	900	57.1	82.1	96.1		
C71	24.75	Henicosane	800	57.1	85.1	296.1	X	
C72	25.65	Docosane	800	57.1	71.1	310.3		

**X** in bold represent those compounds that showed a significant increment in migration samples compared to migration blanks (t-student test,  $p < 0.01$ ); NI: non identified; RI: retention index.

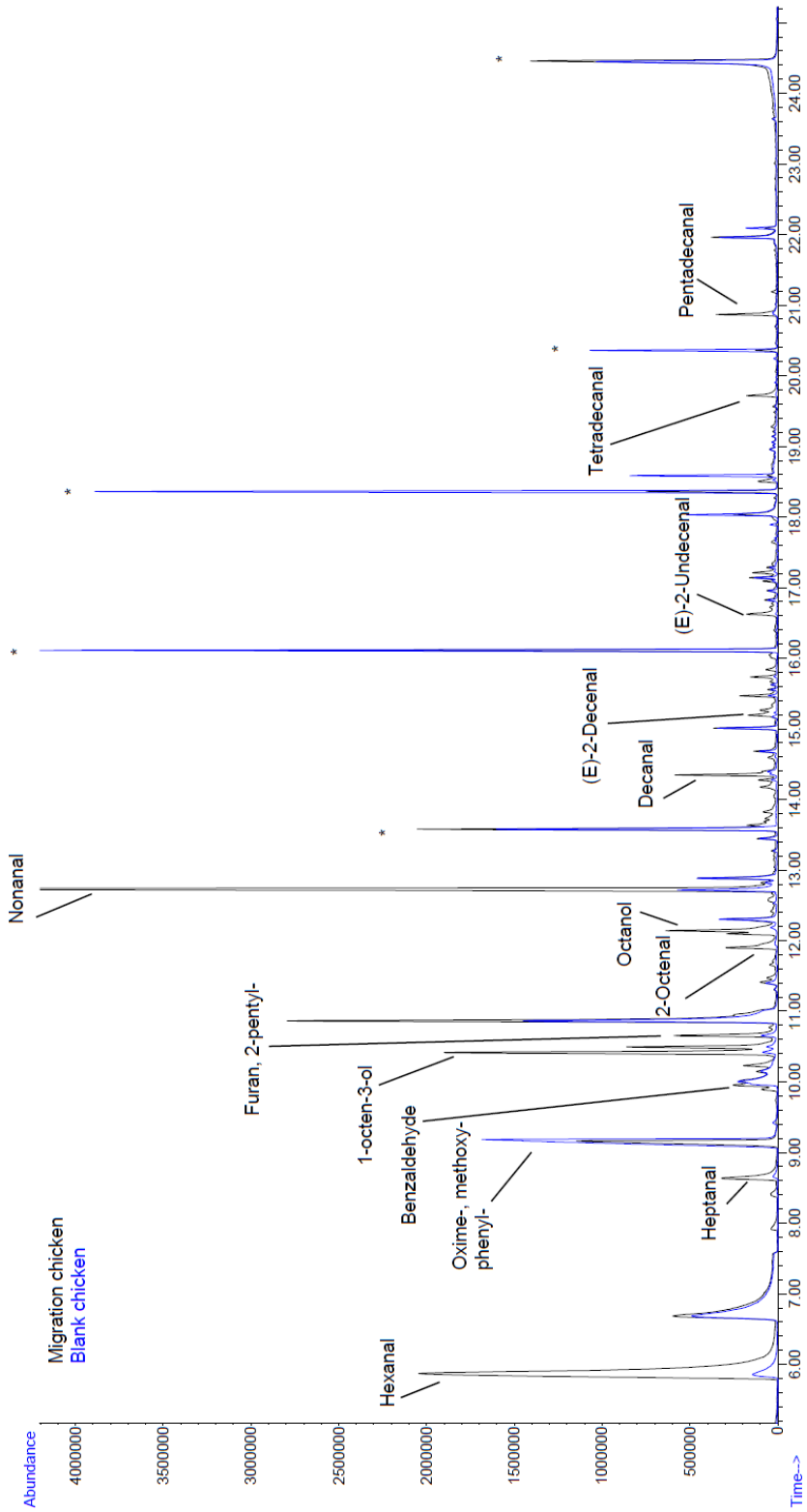
**Supplementary material 2:** Chromatogram obtained by HS-SPME-GC-MS of migration samples to vegetable oil (190°C, 90 min).



**Supplementary Material 3:** Bars diagram of the peak areas of the compounds detected in migration to vegetable oil from cooking bags (Veg Oil B1 and Veg Oil B2) and vegetable oil blank (Veg Oil Blank). Compound codes taken from Supplementary Material 1.



**Supplementary material 4:** Chromatogram obtained by HS-SPME-GC-MS of migration samples to chicken. (\* correspond to siloxanes)



**Supplementary Material 5:** Bars diagram of the peak areas of the compounds detected in migration to chicken from cooking bags (Chicken B1 and Chicken B2) and Chicken (n=3). Compound codes taken from Supplementary Material 1. (Areas of compounds C1, C8, C9 and C16 were divided by 2, 4 or 8 in order to scale them).

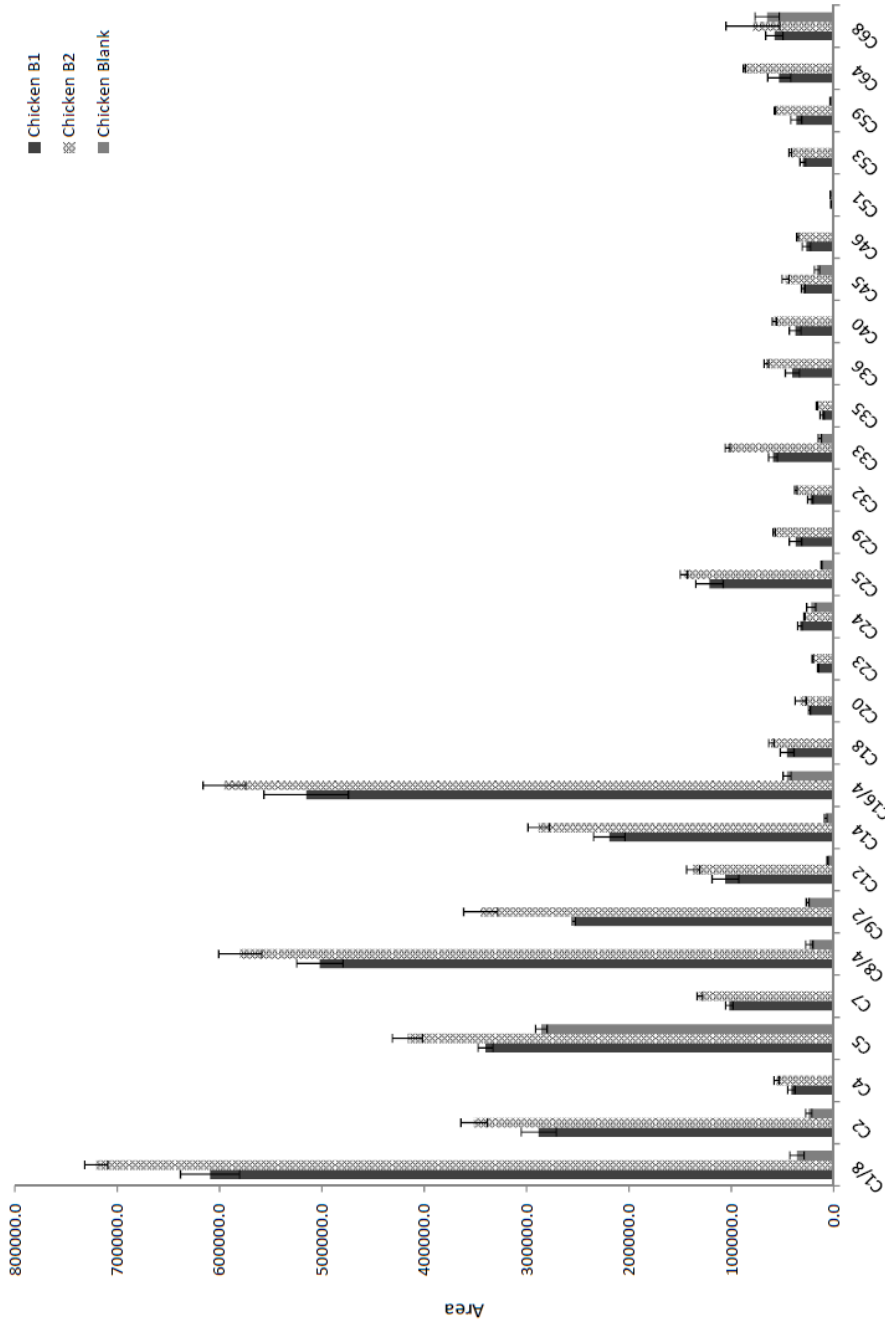


Table 1: Compounds detected by GC-O-MS , experimental and bibliographic retention index (RI<sub>exp</sub> and RI<sub>bib</sub>), aroma descriptors and aroma group (AG) (1: Chemical; 2: Green, aldehyde; 3: Roasted; 4: Mushroom; 5: spicy, liquorice; 6: pleasant, sweet); modified frequency (%MF) in cooking bags (B1 and B2) after oven (AOH) or microwave (AMH) heating.

	rt	Candidates	CAS	RI <sub>exp</sub>	RI <sub>bib</sub>	Aroma descriptors	AG	% MF		
								B1 AOH	B2 AOH	B1 AMH
1	6.55	3-methyl-2-pentanol	565-60-6	772	768	Gas, solvent, chemical	1	50.9	61.1	44.7
2	7.11	Hexanal <sup>✓*</sup>	66-25-1	795	801	Lemon, green	2	11.8	30.6	<10.0
3	8.25	m-Xylene*	108-38-3	843	866	Chemical, plastic, moisture	1	56.1	65.3	<10.0
4	8.87	Methyl furanthiol	28588-74-1	869	868	Roasted corn	3	56.9	11.5	16.3
5	9.11	2-4-Dimethyl thiazole	541-58-2	879	878	Chemical	1	62.3	61.1	<10.0
6	10.10	Methyl dihydrofuranthiol	26486-13-5	924	936	Roasted corn, food, bread	3	57.7	67.3	44.7
7	11.23	<b>1-octen-3-one<sup>✓*</sup></b>	4312-99-6	981	980	Mushroom	4	<b>88.2</b>	<b>89.0</b>	<b>81.6</b>
8	11.68	<b>Octanal<sup>✓*</sup></b>	124-13-0	1004	1006	Lemon, green	2	<b>71.4</b>	<b>89.0</b>	32.7
9	12.77	2-octenal <sup>✓*</sup>	2363-89-5	1068	1060	Chemical, green, sweet	2	58.9	53.0	18.3
10	13.03	<b>3,5-octandienone</b>	38284-27-4	1083	1095	Mushroom	4	<b>89.7</b>	<b>84.2</b>	49.0
11	13.32	<b>Nonanal<sup>✓*</sup></b>	124-19-6	1100	1104	Cucumber, green, pine	2	<b>94.3</b>	<b>89.0</b>	61.3
12	13.50	<b>Sotolon*</b>	28664-35-9	1112	1113	Curry, marple syrup	5	<b>94.3</b>	<b>89.0</b>	<10.0
13	14.13	<b>Isoborneol<sup>✓</sup></b>	124-76-5	1152	1158	Green, moisture	2	<b>71.4</b>	<b>73.6</b>	<b>83.7</b>
14	14.25	Unknown		1160		Unpleasant, chemical	1	39.1	61.2	<10.0
15	14.35	<b>(E)-2-nonenal<sup>✓*</sup></b>	60784-31-8	1167	1160	Chemical, lipstick, modelling clay	1	<b>77.6</b>	<b>86.6</b>	36.5
16	14.57	Unknown		1181		Cucumber, green	2	37.3	55.9	<10.0
17	14.85	<b>Estragol<sup>✓*</sup></b>	140-67-0	1199	1200	Liquorice, sweet	5	<b>76.4</b>	<b>76.4</b>	11.5
18	15.01	<b>Decanal<sup>✓*</sup></b>	112-31-2	1210	1209	Green, cucumber	2	<b>84.9</b>	<b>84.2</b>	<10.0
19	15.57	Unknown		1250	--	Sweet, pleasant	6	50.9	<b>70.7</b>	38.7
20	15.72	<b>(E)-2-decenal<sup>✓</sup></b>	730-46-1	1261	1261	Flowery, lipstick	6	61.0	28.9	56.6
21	15.83	<b>g-octalactone</b>	104-50-7	1269	1261	Coconut, sweet	6	<b>71.6</b>	<b>76.4</b>	<10.0
22	16.43	<b>Undecanal<sup>✓*</sup></b>	112-44-7	1291	1291	Flowery, lemon, aldehyde	2	64.5	<b>76.4</b>	<10.0
23	17.20	<b>(E)-2-Undecenal</b>	2463-77-6	1372	1366	Aldehyde, cucumber	2	<b>73.1</b>	<b>73.6</b>	54.2
24	17.32	<b>4,5-epoxydec-2-enal</b>	134454-31-2	1381	1380	Liquorice	5	59.3	<b>73.6</b>	34.6
25	17.42	<b>(E,Z)-3,6-nonadien-1-ol<sup>✓</sup></b>	28069-72-9	1389	1386	Unpleasant, metallic	1	<b>71.4</b>	<b>89.0</b>	<10.0

26	17.65	<b>Dodecanal</b> ✓*	112-54-9	1407	1420	Sweet, flowery	6	55.0	<b>79.1</b>	23.1
27	18.52	Unknown		1477		Unpleasant, old, powder	1	64.5	40.8	<10.0
28	19.00	Tridecanal✓*	10486-19-8	1516	1511	Coconut, sweet	6	43.0	35.4	16.3

✓Confirmed by NIST \*Confirmed by standard injection.



Table 2. Analytical parameters and migration values from cooking bags 1 and 2 (B1 and B2) to simulant A (Ethanol 10%) (n=3).

Migrant	Linear range (ng/g)	Correlation coefficient $R^2$	LOD (ng/g)	Ethanol 10% (ng/g)	
				B1	B2
Nonanal	0.15 – 57.0	0.998	0.05	1.4±0.18	0.58±0.02
1-Nonanol	3.0 – 95.0	0.997	1.0	3.8±0.33	<1.0
Decanal	0.03 – 59.0	0.998	0.01	0.49±0.01	0.31±0.04

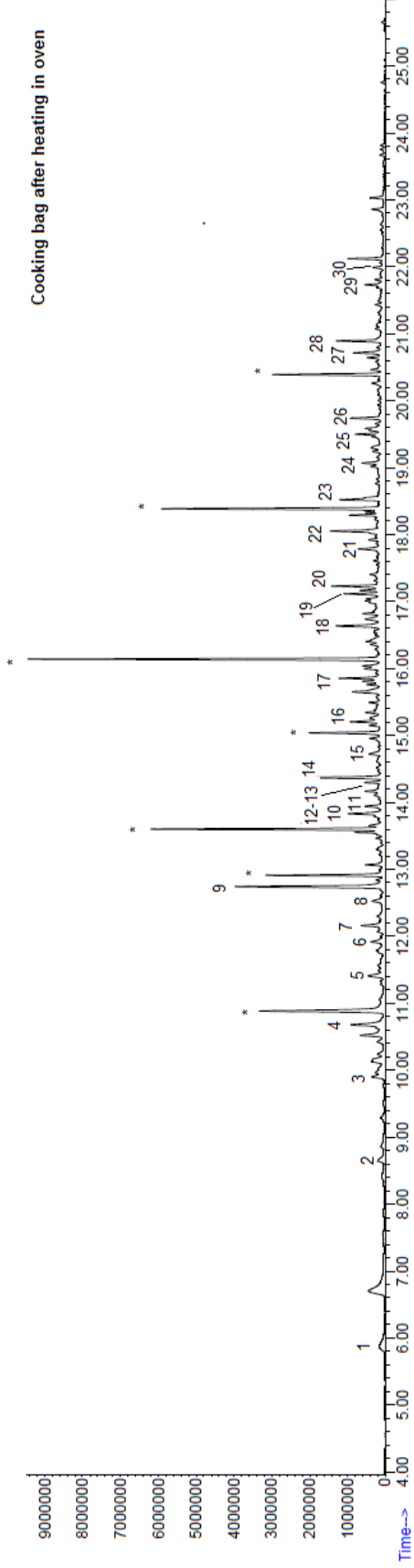
**Table 3.** Analytical parameters of the calibration curves performed in spiked chicken and analyzed by SPME-GC-MS (n = 3)

<b>Compound</b>	<b>Linear range (ng/g)</b>	<b>Correlation coefficient (R<sup>2</sup>)</b>	<b>LOD (ng/g)</b>
Hexanal	5.1 – 77	0.993	1.5
Heptanal	9.0 – 127	0.973	3.0
(E)-2-Octenal	30 - 141	0.990	10
1-Octanol	6.0 - 180	0.978	2.0
Nonanal	9.0 - 150	0.975	3.0
(E)-2-Nonenal	15 - 130	0.987	5.0
1-Nonanol	9.0 - 280	0.995	3.0
Decanal	10 - 150	0.978	3.5
(E)-2-Decenal	24 - 240	0.977	8.0
Tridecane	3.0 - 210	0.982	1.0
Undecanal	18 - 200	0.983	6.0

**Table 4.** Compounds detected in migration to chicken from cooking bags 1 and 2 (B1 and B2) ; their migration values; and the standards used for their quantification (QS) and their specific migration values (SML) on EU/10/2011 Regulation. Compounds are coded according to the table reported in Supplementary material 1. (n = 3)

	<b>Compound</b>	<b>CAS N°</b>	<b>QS</b>	<b>B1 (ng/g)</b>	<b>B2 (ng/g)</b>	<b>EU/10/2011</b>
C1	Hexanal	66-25-1	C1	48.4 ± 2.3	64.0 ± 1.0	--
C2	Heptanal	111-71-7	C2	12.4 ± 0.7	27.5 ± 1.0	--
C4	(E)-2-Heptenal,	18829-55-5	C12	16.6 ± 1.4	23.6 ± 1.0	--
C5	Benzaldehyde	100-52-7	C2	24.8 ± 0.5	43.1 ± 1.5	No SML
C7	Formic acid, heptyl ester	112-23-2	C2	17.3 ± 0.6	24.2 ± 0.5	--
C8	1-Octen-3-ol	3391-86-4	C14	257 ± 11.6	299 ± 10.8	--
C9	Furan, 2-pentyl-	3777-69-3	C2	64.9 ± 0.4	109 ± 5.2	--
C12	(E)-2-Octenal	2548-87-0	C12	22.8 ± 2.8	38.2 ± 1.8	--
C14	1-Octanol	111-87-5	C14	18.7 ± 1.3	27.9 ± 1.0	No SML
C16	Nonanal	124-19-6	C16	19.8 ± 1.6	37.3 ± 1.3	--
C18	(E)-2-Nonenal	18829-56-6	C18	16.5 ± 2.4	22.9 ± 0.9	--
C20	1-Nonanol	143-08-8	C20	5.10 ± 0.21	7.10 ± 1.27	No SML
C23	2-Decanone	693-54-9	C25	1.37 ± 0.04	1.84 ± 0.07	--
C24	Dodecane	112-40-3	C33	<1.0	<1.0	--
C25	Decanal	112-31-2	C25	12.0 ± 1.3	15.1 ± 0.4	--
C29	(E)-2-Decenal	3913-81-3	C29	16.2 ± 2.6	26.1 ± 0.4	--
C32	(E,Z)-2,4- Decadienal	2363-88-4	C29	9.47 ± 1.07	16.1 ± 0.5	--
C33	Tridecane	629-50-5	C33	<1.0	1.56 ± 0.04	--
C35	Undecanal	112-44-7	C35	3.57 ± 0.65	5.38 ± 0.20	--
C36	(E,E)-2,4- Decadienal	25152-84-5	C29	17.7 ± 3.0	29.8 ± 1.0	--
C40	2-Undecenal	2463-77-6	C29	16.4 ± 2.5	26.0 ± 0.9	--
C45	Tetradecane	629-59-4	C33	<1.0	<1.0	--
C46	Dodecanal	112-54-9	C35	9.01 ± 1.4	12.0 ± 0.3	--
C51	1-Pentadecene	13360-61-7	C29	<1.0	<1.0	--
C53	Tridecanal	10486-19-8	C35	10.3 ± 0.9	14.6 ± 0.5	--
C59	Tetradecanal	124-25-4	C35	12.5 ± 1.8	19.8 ± 0.2	--
C64	Pentadecanal	2765-11-9	C35	18.4 ± 3.9	30.4 ± 0.3	--

Cooking bag after heating in oven



Cooking Bag before heating in oven

