

1 **Aromatic changes in home-made truffle products after heat treatments**

2 **Eva Tejedor-Calvo<sup>1</sup>, Diego Morales<sup>2</sup>, María Ángeles Sanz<sup>3</sup>, Sergio Sánchez<sup>1</sup>, Pedro**  
3 **Marco<sup>1</sup>, Sergi García-Barreda<sup>1</sup>**

4 <sup>1</sup>Department of Plant Science, Agrifood Research and Technology Centre of Aragón  
5 (CITA), Avda. Montañana 930, 50059, Zaragoza, Spain

6 <sup>2</sup>Nutrigenomics Research Group, Department of Biochemistry and Biotechnology,  
7 Universitat Rovira i Virgili, 43007 Tarragona, Spain

8 <sup>3</sup>Laboratories and Technological Assistance, Agrifood Research and Technology Centre  
9 of Aragón (CITA), Avda. Montañana, 50059, Zaragoza, Spain

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15 \*Corresponding author:

16 Eva Tejedor Calvo

17 [etejedorc@aragon.es](mailto:etejedorc@aragon.es)

18

19 **Abstract**

20 Truffles are highly valued by their aromatic properties and can aromatize food products.  
21 However, the truffle aroma could be reduced or lost with heat treatments (pasteurization  
22 and sterilization) necessary for products security and safety. In this study, sunflower oil  
23 and honey were aromatized with black truffle (lyophilized and fresh) using two different  
24 concentrations (5 and 10%) for 24 h and then heat treatments (pasteurization and  
25 sterilization) were carried out. Truffle organic volatile compounds from products were  
26 investigated by SPME-GC-MS and sensory analysis by trained panel. More than 80  
27 compounds were detected. Some of them were affected differently by heat process  
28 depending on the food matrix. Professional tasters scored higher key aromatic attributes  
29 such as sulphurous and olive oil in fresh truffle products, regardless the heat treatment  
30 applied.

## 31 1. Introduction

32 Truffles are gourmet mushrooms appreciated worldwide because of their unique aroma.  
33 Food industry has exploited the ability of truffles to transfer aroma to various food  
34 matrices for developing many truffle products and bring them an added value (Tejedor-  
35 Calvo et al., 2023). Among the product variety, fatty products such as oil, cheese, cream  
36 and butter are the most popular (Mustafa et al., 2020).

37 Food processing or preservation technologies reduce, modify or remove some of the key  
38 aromatic compounds of truffles (Campo, Marco, Oria, Blanco, & Venturini, 2017). To  
39 compensate this aroma loss, 2,4-dithiapentane (bis-(methylthio)-methane or BMTM) is  
40 commonly used as truffle flavoring (Pacioni, Cerretani, Procida, & Cichelli, 2014;  
41 Torregiani et al., 2017). This compound is characteristic of the white truffle (*Tuber*  
42 *magnatum*) aroma, so far has not been detected in other truffles species such as the black  
43 truffle (*Tuber melanosporum*). However, a mixture of DMS and 2-methyl-butanal (2-  
44 MB) is also used in black truffle products as a new formula to replicate black truffle  
45 aroma, as Tejedor-Calvo et al. (2023) studied. Since the aroma of these products is given  
46 by BMTM, the food industry tends to use cheaper truffle species instead of the valued *T.*  
47 *magnatum* or *T. melanosporum*. For this, they use species with morphological similarity  
48 such as *Tuber borchii* instead of *T. magnatum*, or *T. indicum* and *T. aestivum* instead of  
49 *T. melanosporum* (Oliach et al., 2021)

50 The use of synthetic flavours causes truffle price drops (Oliach et al., 2021) and increases  
51 consumer confusion (Tejedor-Calvo et al., 2023), among others. Therefore, a genuine  
52 truffle flavouring extract has a potential place in the truffle products market (Phong et al.,  
53 2022; Tejedor-Calvo et al., 2021).

54 The black truffle aroma is a complex mixture composed by numerous and different  
55 aromatic volatile compounds (VOCs). Among them, sulphur compounds such as

56 dimethyl disulphide (DMDS) and dimethyl sulphide (DMS) are the most relevant (Costa  
57 et al., 2015; Culleré et al., 2010; Culleré, Ferreira, Venturini, Marco, & Blanco, 2013;  
58 Tejedor-Calvo et al., 2021). To aromatize products, in most of them, the truffle and the  
59 food matrix are mixed to enhance the aromatization process. This has the drawback that  
60 a sanitizing treatment (*e.g.* with heat) is required for food safety reasons, especially if  
61 fresh truffles are used (Rivera, Venturini, Oria, et al., 2011). Heat treatment of truffles  
62 severely changes their aroma, resulting in a product that is barely reminiscent of the  
63 original, but this treatment is necessary in case fresh or freeze-dried truffle are included  
64 because some microorganism might be present (Campo et al., 2017) Our hypothesis is  
65 that black truffle VOCs surrounded by a food matrix might not be as affected by heat  
66 treatment as fresh truffle VOCs, thus enhancing that some of the relevant truffle VOCs  
67 remain in the heat-treated truffle products. So, the main aim of our study was to evaluate  
68 the effect of two different heat treatments (pasteurization and sterilization) on the VOCs  
69 transferred by black truffle to two of the most common truffle products (sunflower oil  
70 and honey). As a secondary aim, we assessed whether heat treatments affected differently  
71 the aroma of truffle products when fresh and lyophilized truffle were added in two  
72 different amounts.

73 To date, the existing international recommendations concerning the marketing and  
74 commercial quality control of truffles (Nations, 2017) do not regulate the  
75 commercialization of truffle products. Among the major truffle-producing countries,  
76 France and Italy have their own specific regulations (Tejedor-Calvo et al., 2023).  
77 However, Spain –which is the world leader in black truffle production– and other  
78 countries lack regulation (Oliach et al., 2020)). Our study addresses this gap by providing  
79 scientific evidence to support the regulation of truffle products elaboration and  
80 information to consumers.

81

## 82 2. Materials and methods

### 83 2.1 Truffle and food matrices

84 *Tuber melanosporum* (Vittad.) ascocarps were harvested in Moncayo forests (Zaragoza,  
85 Spain). Fresh truffles (20 fruiting bodies) were identified, selected, and processed  
86 according to (Rivera, Venturini, Marco, et al., 2011). Half of the truffles were lyophilized  
87 (LyoBeta 15 lyophilizer, Telstar, Madrid, condenser temperature was -80 °C), ground,  
88 mixed and sieved until a particle size lower than 0.5 mm was obtained. Powdered truffles  
89 were kept at -80 °C until further use. The other half was sliced before their use using a  
90 mandolin slicer. Refined sunflower oil and mixed-flower honey products were purchased  
91 from a local supermarket (Zaragoza, Spain).

### 92 2.2 Aromatization process

93 In glass jars (100 mL), 50 mL of food matrix (oil or honey) were poured, with fresh  
94 (sliced) or lyophilized (powder) black truffle being added at two proportions (5 and 10%).  
95 The samples were homogenized in a digital rotator 220 V (LW Scientific, Spain) during  
96 2 h. Afterwards, the samples were kept in refrigeration (4°C) for 48 h for aromatization.

### 97 2.3 Heat treatments and storage

98 Two of the most used heat treatments in the food industry were selected: pasteurization  
99 and sterilization. For that, the jars were closed hermetically. The sterilization treatment  
100 was carried out in an autoclave (Micromar Mini autoclave, Marrodán, Lodosa, Spain) at  
101 121 °C for 30 min. The pasteurization process was carried out in a thermostatic water bath  
102 (Lab Systems, Barcelona, Spain) at 75 °C during 5 min. Afterwards, the samples were  
103 kept at 4 °C until their sensory analyses.

104 Two samples were prepared for each combination of food matrix (oil, honey), truffle  
105 preservation method (fresh, freeze dried), truffle content in the product (5%, 10%) and

106 heat treatment of the product (no treatment, pasteurization and sterilization. Additionally,  
107 samples of the food matrices without truffle were prepared in duplicate for all the heat  
108 treatments. The VOC profile of the fresh and lyophilized truffles before use in food  
109 products were also analyzed, in order to check the differences between them and to  
110 compare these profiles with those of the truffle products.

111

## 112 2.4 VOCs analysis

### 113 2.4.1 VOCs extraction by SPME

114 The methodological approach was based on works carried out by (Culleré, Ferreira,  
115 Venturini, Marco, & Blanco, 2012)(Culleré, Ferreira, Venturini, Marco, & Blanco, 2012)  
116 with some modifications. A solid phase microextraction (SPME) was used to extract the  
117 aromatic compounds. For that, a fused silica fiber coated with a 50/30 mm layer of  
118 divinylbenzene/carboxen/polydimethylsiloxane from Supelco (Barcelona, Spain) was  
119 chosen. The samples (2 g of truffled product) were placed in a 20 mL glass vial closed  
120 with a septum. After the vial was conditioned at 50 °C for 10 min. The fiber was then  
121 exposed to the headspace of the vial for 20 min.

### 122 2.4.2 GC-MS analysis

123 The VOCs profile of the different samples was analysed by static GC-MS using a gas  
124 chromatograph Agilent 6890N (Termostest, Milan, Italy) coupled with a mass  
125 spectrometer detector. This instrument was equipped with a capillary column HP-5MS  
126 (Agilent Technologies, California, USA) of 30 m, 0.32 mm i.d., 0.25 µm film thickness  
127 and a flow of 1 mL/min with helium as a carrier gas. The oven temperature was 45 °C  
128 held for 2 min, 45–200 °C at a rate of 4 °C/min, and finally to 225 °C at 10 °C/min, and  
129 held for 5 min (Gómez et al., 2022). The MS used the electron impact mode with an

130 ionization potential of 70 eV and an ion source temperature of 200 °C. The interface  
131 temperature was 220 °C. The MS scanning was recorded in full scan mode (35–250 m/z).  
132 A TurboMass software was used for controlling the GC-MS system.

### 133 2.4.3 Data analysis

134 Peak identification of the VOCs was achieved by comparison of the mass spectra with  
135 mass spectral data from the NIST MS Search Program 2.0 library, and by comparison of  
136 previously reported Retention Indexes (RI) with those calculated using an n-alkane series  
137 (C6–C20) under the same analysis conditions. The n-alkane series and standards for MS  
138 identification (all standards of purity higher than 95%) were purchased from Sigma-  
139 Aldrich (Madrid, Spain). Semiquantification was done by integrating the area of one ion  
140 characteristic of each compound and normalization by calculating the relative percentage.  
141 This allowed comparison of each eluted compound between samples.

### 142 2.5 Sensory analysis

143 A panel of eight trained tasters (26-50 years old; 4 male and 4 female) evaluated the aroma  
144 of the samples before and after heat treatments. Tasters were previously trained for three  
145 sessions of 45 min. The analyses were conducted according to the ISO 11035:1994 and  
146 the following aromatic parameters were selected according to previous studies (Campo  
147 et al, 2017; Tejedor-Calvo et al., 2023): sulphur, mushroom, earthy, black olives, leather-  
148 animal, nuts and alcohol. Each parameter was assessed with a 9-point rating scale (1-  
149 minimum and 9 maximum aroma/taste). Samples were presented for each product in three  
150 sessions for three days (6 sessions in total). In total 30 samples were evaluated for each  
151 product (honey and sunflower oil), 5 products in each session. The experiment was carried  
152 out in one week for each product. The evaluation was carried out by olfactive and tasting  
153 phase, and between samples bread and apple was given to clean. The values presented for  
154 each truffle type (fresh or lyophilized), amount added, and heat treatment by duplicate.

## 155 2.6 Statistical analysis

156 The sensory analysis was analyzed with one-way analysis of variance (ANOVA)  
157 followed by Tukey's multiple comparison test. Differences were evaluated at 95%  
158 confidence level ( $P \leq 0.05$ ). Statistical analysis was performed using GraphPad Prism  
159 version 9.3 (GraphPad Software, San Diego, CA).

160 The VOCs were analyzed with principal component analysis (PCA), performed with the  
161 statistical software R (R Core Team, 2022) and visualized using XLStat 2009 (Addinsoft,  
162 Paris, France) and the R package factoextra (Kassambara and Mundt, 2020).

163

## 164 3. Results and discussion

### 165 3.1 VOCs in fresh and lyophilized truffle

166 In the fresh truffle samples, a total of 32 VOCs were detected, with 2-propanone, DMS,  
167 2-methyl-propanol, 3-methyl-1-butanol, 2-methyl-butanal, 2-methyl-butanol, octanal, 3-  
168 methylanisol and 3-methyl-1-butanol being amongst those with higher presence. In the  
169 lyophilized samples, a total of 41 VOCs were detected, with ethyl acetate, 1,3-butanediol,  
170 ethyl-2-methylbutanoate, anisol, isobutyl isobutyrate, 3-methyl-1-butanol and 3-methyl-  
171 1-butanol being amongst those with higher presence (Table 1). Twenty VOCs were found  
172 in both fresh and lyophilized samples, whereas 12 were found only in fresh truffle and 21  
173 only in lyophilized samples. Truffle aroma can change dramatically depending on the  
174 postharvest treatment applied. Among them, lyophilization has been reported as the best  
175 one to preserve the main truffle odor compounds (Campo et al., 2017). When truffles are  
176 lyophilized, water is totally removed and moisture is reduced, so more compounds might  
177 be concentrated and therefore can be detected by gas chromatography in comparison to



178 fresh truffles, for instance, 2-methylpropanal, 2-methylbutanal and 3-methylbutanal  
179 (Campo et al., 2017; Palacios et al., 2014).

180 Sulphur compounds as DMS and DMDS are the main components of truffle aroma. DMS  
181 and DMDS have been reported in most truffle species and are thought to derive from the  
182 catabolism of L-methionine through the Ehrlich pathway (Liu et al., 2013; Splivallo et  
183 al., 2011). According to other studies, the freeze-drying process was able to retain DMS,  
184 but not DMDS (Palacios et al., 2014), as it is observed in our results (Table 1). This study  
185 reported many VOCs in the lyophilized truffle, mainly aldehydes like hexanal and  
186 methional. Both compounds were detected in higher value in lyophilized samples  
187 compared with fresh truffles (Table 1). Other volatiles were also very intense in the  
188 lyophilized truffle, mainly aldehydes such as 2-methylbutanal, hexanal, Z-4-heptanal  
189 (Campo et al., 2017). The remarkable differences in the VOC profile between fresh and  
190 lyophilized truffles are expected to be reflected in the truffle products using one or the  
191 other.

### 192 3.2 VOCs in truffle products and effect of heat treatment

193 Both sunflower oil and honey were characterized by ethanol, methylene chloride, acetic  
194 acid and hexane, in the latter case together with 2-propanone, 2-butanone-3-hydroxy and  
195 linalool oxide cis (Fig. 1, Fig. S1). As regards the truffle product samples, the VOC  
196 profiles showed similar patterns for the two food matrices and the two truffle proportions,  
197 but different patterns for fresh and lyophilized truffles (Fig. 1). Some key truffle VOCs  
198 such as 2-propanone, 3-methyl-1-butanol, 2-methyl-1-butanol, 2-methyl-butanal were  
199 detected in both matrices. However, the VOCs amount might vary depending on the  
200 matrix, as hexanal in sunflower oil and furfural for honey.

201 It is believed that the aromatic compounds involved in a food matrix are more protected  
202 due to some links or connections such as sulphur bonds (Seuvre et al., 2000), although  
203 some compounds could have higher affinity depending on the matrix. However,  
204 preservation processes as heat treatment could break the links and therefore volatilize  
205 them.

206 According to a previous study (Seuvre et al., 2000) 2-nonanone and isoamyl acetate  
207 present opposite behaviors in two media with same composition, one emulsified, and the  
208 other not. The volatility of isoamyl acetate was not affected by the change of the medium  
209 structure whereas that of 2-nonanone increased. The decrease of retention of 2-nonanone  
210 in an emulsified system may be due to a modification of the fixation site for this  
211 compound on the protein or to a competition between the lipid and the aroma compound  
212 while the protein is adsorbed at the lipid-water interface (Seuvre et al., 2000). In our  
213 results, 2-nonanone was only present in honey, the only product with proteins. In the same  
214 way, some molecules such as 2-methyl-butanal might be preferable retained by oil matrix  
215 due to their hydrophobicity.

216 In addition, simple matrices (oil, protein, polysaccharides) as well as combinations  
217 (emulsions) can retain certain molecules (Guichard, 2002, 2006; Mao et al., 2017).  
218 However, in some systems the impact of oil was so dominant that binding effects of other  
219 food ingredients (e.g., proteins) to volatile compounds were insignificant (Roberts et al.,  
220 2003; Seuvre et al., 2000). As an example, grapeseed oil helped to trap key truffle  
221 aromatic compounds using supercritical fluids as extraction method (Tejedor-Calvo et al.,  
222 2021).

223 After the heat treatments, several changes in the aromatic compounds were noticed  
224 regardless of the matrix used. Even oil and honey VOCs in control samples (without  
225 truffle) were modified. Some molecules appeared with the pasteurization (2-propanone-

226 1-hydroxy, 2,3-butanodione, acetaldehyde, pentanal) or the sterilization (2-propanone,  
227 2,3-pentanodione, 2-pentanol, 3-methyl-butanalm 3-methyl-butanol). This indicates that  
228 some changes in the VOC profile of the truffled samples were related to the effect of heat  
229 on the matrix.

230 The amount of hexanal in sunflower oil and furfural in honey were higher in all the  
231 sterilized samples than in non-treated or pasteurized samples. These two compounds have  
232 grass and almond odorant properties (Tejedor-Calvo et al. 2023), therefore these aromatic  
233 notes might be clearly detected in an olfactometry. However, depending on the  
234 concentration and the perception threshold of these molecules the aroma detected by  
235 humans might be different. In general, pasteurization decreased methylene chloride, 3-  
236 methyl-1-butanol and 2-butanone; and increased 2-methyl-butanal and 1-propanol.  
237 Among them, 3-methyl-1-butanol molecule is described as malt and whiskey odorant  
238 (Tejedor-Calvo et al. 2023). These samples might have the absence of those aromatic  
239 properties. Conversely, sterilization increased primarily acetic acid, 2-methyl-butanal,  
240 hexanal and furfural. A previous study reported that canning preserved mainly ethyl esters  
241 such as ethyl-2- and ethyl-3-methyl butyrate (Campo et al., 2017), but in our treated  
242 samples slight amounts of them were detected. All these changes due to heat treatments,  
243 might change dramatically the aromatic properties (Campo et al., 2017).

244 Among key truffle aromatic compounds, DMS presence was reduced by sterilization in  
245 both matrices, but in honey the compound was in higher amount when lyophilized truffle  
246 was used. One of the reasons might be because DMS is more soluble or trapped by water-  
247 based products than lipidic ones. The presence of 2-butanone in oil was reduced with heat  
248 treatments, whereas it increased with pasteurization in honey with fresh truffle. Finally,  
249 3-methyl-1-butanol was only affected by pasteurization in oil with fresh truffle, but not  
250 in the rest of samples, probably because the same reason as DMS.

251 3.3 Effect of heat treatment in sensory attributes

252 The sensory analysis carried out by the trained testers panel revealed significant effects  
253 of the food matrix, the addition of truffle to the matrices, and the heat treatment on several  
254 sensory attributes (Table 2).

255 Regarding the matrix effect, sunflower oil showed higher *per se* scores for “butter” and  
256 “black olives” (Table 2). This fact might be due to the presence of compounds that are  
257 close to margarine or butter VOCs and, moreover, specific phytochemicals that are shared  
258 between olives and sunflower oil such as, for instance, saturated and unsaturated  
259 aldehydes (Ontanón et al., 2013).

260 Truffle addition led to significant changes in specific attributes, although others that  
261 might be expected to increase, such as “mushrooms”, remained invariable, mainly in  
262 honey matrix. The case of “sulphur” is remarkable, since its scores were significantly  
263 increased after truffle addition, regardless of whether the aromatization was carried out  
264 with fresh or lyophilized truffles and being effective at both 5 and 10% concentrations  
265 (Table 2). This attribute score was increased in both matrices. This agrees with previous  
266 reports and is related to the richness of sulphured compounds in black truffle (Li et al.,  
267 2012; Liu et al., 2013). Another attribute that significantly increased its score when  
268 compared to the control was “leather-animal” when 10% lyophilized truffle was used to  
269 aromatize oil. This specific smell has been traditionally described for *T. melanosporum*,  
270 and sunflower oil seemed to be a more receptive environment than honey to trap and  
271 retain the associated VOCs (Allen & Bennett, 2021). This could be due to the lyophilic  
272 character of sunflower oil enhancing the trapping of a larger diversity of aromatic  
273 molecules than a high sugar content material as honey (Torregiani et al. 2017).

274 Regarding the effect of heat treatments to the potential loss of attributes, sulphur scores  
275 were not reduced for honey after any treatment. However, canning decreased sulphur  
276 scores in oil when 5% of fresh or lyophilized truffle was used, although not in 10%  
277 samples, suggesting that the highest concentration is required not to lose this feature  
278 (Table 2). Previous works have already described the detrimental effect of heat treatments  
279 on the typical aroma (mainly volatile sulphur compounds) of black truffle when applied  
280 directly to the fruitbody (Campo et al., 2017).

281 Interesting results were obtained for the “black olives” attribute, another of the typical  
282 ones in black truffle (Campo et al., 2017; Culleré et al., 2013): pasteurization reduced the  
283 score in the case of olive oil with fresh truffles (Table 2). However, no changes were  
284 observed in honey samples and, besides, the control sunflower oil sample (with no truffle  
285 aromatization) suffered a significant decrease in this attribute after canning. These facts  
286 suggest that the VOCs that are being affected in this case are molecules related to the oil  
287 and not the truffle. On the other hand, the other attribute that was linked to oil, “butter”,  
288 did not suffer any variation after heat treatments, as well as the other evaluated scores  
289 (mushroom, earthy, leather-animal, blue cheese, nuts, alcohol).

290 In short, sulphur compounds seemed to be the more sensitive to canning, particularly  
291 when a low concentration of truffle was used and specifically for oil matrix. This decrease  
292 could be avoided by using pasteurization instead or by adding at least a 10% of truffle in  
293 the aromatization step.

### 294 *3.4 Multivariable analysis of truffle honey and oil*

#### 295 *3.4.1 VOC profile*

296 The possible correlations of heat treatments, truffle type and amount with the relative  
297 abundance of the VOCs detected by SPME-GC-MS was explored with PCA (Figs. 2, 3).

298 The two food matrices were investigated individually due to the differences described  
299 above (section 3.2). In both cases the PCA explained with the first two components only  
300 a 35-42% of the variability, thus indicating the complexity of the relationships between  
301 VOCs, type of truffle and heat treatment applied (Figs. 2, 3).

302 In the PCA performed for the oil, the first component (explaining 24.8% of the variability)  
303 was mainly correlated to the truffle type used. Samples with freeze-fried truffle grouped  
304 with non-truffled samples, whereas samples with fresh truffle were clearly separated by  
305 the PCA (Fig. 2a). The second component (explaining an additional 17.2% of data  
306 variability) only separated the sterilized sample with 10% lyophilized truffles, although  
307 the 5% sample seemed to point in the same direction (Fig. 2a). Among the VOCs that  
308 showed more positive loadings with the first PCA component (*i.e.* those correlated to  
309 aromatization with fresh truffle), there were several VOCs with high presence in truffles  
310 (Table 1): 2-methyl-1-butanol, 2-methyl-1-propanol, 2-methyl-propanal, 3-methyl-  
311 butanal, 2-methyl-butanal, ethanol, 3-methyl-1-butanol, and 3-methyl-2-butanone (Fig.  
312 2b). Among the VOCs that showed more negative loadings with the first PCA component  
313 (*i.e.* those correlated to non-truffled samples or to aromatization with lyophilized truffle),  
314 there were several already present in lyophilized truffles such as 1,3-butanediol (Fig. 2b).  
315 Among the VOCs that showed more positive loadings with the second PCA component  
316 (*i.e.* those correlated to the sterilized samples with lyophilized truffle), there were many  
317 VOCs that were not present or very rare in the remaining oil samples, such as heptanal,  
318 2-methyl-butyl-acetate or 3-methylanisol (Fig. 2b).

319 In the PCA performed for honey, the first PCA component (explaining 19.2 % of the  
320 variability) was correlated mainly with the truffle type used. Samples with freeze-fried  
321 truffle grouped with non-truffled samples, whereas samples with fresh truffle were clearly  
322 separated (Fig. 3a). To a lesser degree, this component also separated samples with 5%

323 fresh truffle from samples with 10% fresh truffle. The second component (explaining an  
324 additional 16.3 % of the variability) was slightly correlated with the sterilization treatment  
325 (positive loadings), although only the sterilized sample with 10% lyophilized truffles was  
326 clearly separated from the rest (Figure 3a). Among the VOCs that showed more positive  
327 loadings with the first PCA component (*i.e.* those correlated to aromatization with fresh  
328 truffle), there were several VOCs with high presence in truffles (Table 1): 2-methyl-1-  
329 butanol, propanal-2-methyl, 3-methyl-1-butanol, and 2-methyl-1-propanol (Fig. 3b).  
330 According to previous studies, these compounds among others were reported as key  
331 molecules in fresh truffle aroma (Tejedor-Calvo et al., 2021). Among the VOCs that  
332 showed more negative loadings with the first PCA component (*i.e.* those correlated to  
333 non-truffled samples or to aromatization with lyophilized truffle), there were several  
334 VOCs linked to the VOC profile of honey, such as 3-hidroxy-2-butanone, methylene  
335 chloride, hexane or acetic acid. There was also the DMS, present in truffles but also  
336 detected in non-truffled honey after being heated (Fig. 1, 3b). Finally, within the VOCs  
337 that showed more positive loadings with the second PCA component (*i.e.* those correlated  
338 to the sterilized sample with 10% lyophilized truffle), there were VOCs already present  
339 in truffles, such as butanal-2-methyl, 3-methylanisole, anisole or isobutyl isobutyrate  
340 (Fig. 3b).

#### 341 *3.4.1 Aromatic profile*

342 PCA was also used to explore the correlations of the treatments with the results of the  
343 sensory analysis (Fig. 4). The first two principal components explained 81.1% of the total  
344 variability. All the sensory attributes showed strong positive loadings corresponding to  
345 the first component (63.7% of variability), whereas butter and leather animal showed  
346 correspondingly the more positive and the more negative loadings with the second  
347 component (17.4% of variability). The PCA allowed to clearly separate the samples into

348 four clusters. The west group (grey) grouped honey and oil samples without truffle. This  
349 cluster was clearly associated with lower values for all the sensory attributes, further  
350 suggesting that heat treatments in control samples did not affect the sensory profile. The  
351 middle-down group (orange) included all the honey samples. In this group, samples with  
352 lyophilized truffle tended to concentrate at the bottom, correlating with leather and earthy  
353 attributes which had been linked to lyophilized truffles in previous studies (Campo et al.,  
354 2017; Tejedor-Calvo et al., 2021). All the oil samples were placed in the right-side of the  
355 PCA. Those with fresh truffle grouped in the middle-left cluster, whereas those with  
356 lyophilized truffle were divided in two clusters (both yellow): the up cluster (low truffle  
357 concentration) and down cluster (high truffle concentration). The latter was related to  
358 alcohol and earthy attributes, whereas the other two oil clusters were associated with  
359 butter, black olives, sulphur, blue cheese and mushroom attributes, particularly samples  
360 with no treatment.

361

#### 362 **4. Conclusions**

363 An aromatization step was included in sunflower oil and honey products with fresh and  
364 lyophilized truffle. However, fresh truffle is a better suitable material since key truffle  
365 aromatic compounds were detected in the products. Pasteurization treatment is  
366 recommended to preserve sulphur compounds after aromatization step in truffle products.  
367 On the contrary, strong heat treatments can reduce or lose some key truffle volatiles. For  
368 the first time aromatic profiles of truffled products, and their changes in conservation  
369 processes have been investigated. In order to create knowledge about truffle use and  
370 truffle products processing further studies testing other food matrices and food  
371 postharvest treatments are needed.

372



373 **Declaration of competing interest**

374 None

375

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383 **CRediT authorship contribution statement**

384 **Eva Tejedor-Calvo:** Conceptualization, Investigation, Methodology, Writing - original  
385 draft. **Diego Morales:** Formal analysis, Data curation, Methodology, Writing - original  
386 draft. **María Ángeles Sanz:** Formal analysis. **Sergio Sánchez:** Funding acquisition,  
387 Supervision. **Pedro Marco:** Supervision, Validation, Visualization, Writing - review &  
388 editing. **Sergi García-Barreda:** Data curation, Software, Visualization, Writing - review  
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499 **Tables**

500 **Table 1.** List of volatile organic compounds identified by SMPE-GC-MS in truffles  
 501 samples, sunflower oil and honey. Relative percentage of area values (%) obtained in the  
 502 GC-MS for the fresh truffles (F) and the lyophilized truffles (L) used to aromatize the  
 503 samples.

N°	Name	CAS n°	RT	RI <sub>exp</sub>	RI <sub>lit</sub>	Mass ( <i>m/z</i> )			F	L
1	Ethanol	64-17-5	1.33	<500	427	45	46	43	3.83	2.11
2	2-propanone	67-64-1	1.378	<500	500	43	58	42	2.80	0.51
3	Dimethyl sulphide	75-18-3	1.474	521	521	62	61	47	3.76	1.57
4	Methylene chloride	75-09-2	1.503	532	531	49	86	84	-	-
5	1-propanol	71-23-8	1.568	555	548	31	29	42	-	-
6	Propanal-2-methyl	78-84-2	1.575	558	560	43	72	41	2.69	2.99
7	2,3-butanedione	431-03-8	1.626	577	587	43	87	86	-	2.13
8	Isopropyl formate	625-55-8	1.64	582	-	45	73	42	-	-
9	Butanal	123-72-8	1.676	595	598	44	72	57	-	-
10	3-methyl-2-butanone	563-80-4	1.683	597	-	43	86	41	1.59	3.46
11	Acetic acid	64-19-7	1.69	600	602	43	60	45	-	-
12	2-butanone	78-93-3	1.719	603	602	43	72	57	2.05	-
13	Hexane	110-54-3	1.726	604	-	57	86	56	-	-
14	Ethyl acetate	141-78-6	1.77	609	607	43	80	70	-	9.42
15	2-methyl-1-propanol	78-83-1	1.878	622	626	43	42	41	7.91	0.77
16	Acetaldehyde	75-07-0	1.899	624	-	44	43	42	-	-
17	3-methyl-butanal	590-86-3	2.08	645	646	44	71	58	10.70	6.68
18	Butanol	71-36-3	2.101	648	656	56	55	43	-	-
19	2-methyl-butanal	96-17-3	2.123	653	653	57	86	58	7.23	2.64
20	2-propanone-1-hydroxy	116-09-6	2.224	662	-	43	74	42	-	0.34
21	1-penten-3ol	616-25-1	2.23	663	680	57	58	55	-	-
22	Metylpropylformate	589-40-2	2.31	672	-	45	73	59	0.97	0.31
23	2-pentanone	107-87-9	2.368	679	687	43	86	71	0.61	-
24	2,3-pentadione	600-14-6	2.454	689	696	43	100	57	-	-
25	Pentanal	110-62-3	2.469	691	704	44	58	57	-	-
26	2-pentanol	6032-29-7	2.483	692	700	45	73	55	-	-
27	2-butanone,3-hidroxy	513-86-0	2.613	704	707	45	88	55	-	0.65
28	3-methyl-1-butanol	123-51-3	2.966	723	737	55	70	57	28.32	7.00
29	2-methyl-1-butanol	137-32-6	3.045	728	743	57	70	56	4.07	4.19
30	Dimethyl-disulphide	624-92-0	3.103	731	733	94	79	61	0.51	-
31	2-methyl-pentanal	123-15-9	3.18	735	-	43	58	41	-	-
32	Propanoic-ac-2methyl- ester	97-62-1	3.434	775	760	43	29	71	-	1.30
33	Isobutylacetate	110-19-0	3.737	783	770	43	56	41	-	0.26
34	Propanoic-ac-2methyl	79-31-2	3.989	735	753	43	88	73	-	0.41
35	1,3-butanediol	513-85-9	4.3	785	803	45	27	43	-	6.18
36	Octane	111-65-9	4.342	800	800	43	85	71	-	3.25
37	Hexanal	66-25-1	4.378	802	801	44	57	56	1.64	3.25
38	ethylbutanoate	105-54-4	4.443	803	803	71	43	29	-	-
39	Furfural	98-01-1	5.394	830	830	96	95	67	-	0.62
40	2-methylthio-ethanol	5271-38-5	5.639	873	-	61	92	47	0.08	-
41	ethyl-2-methylbutanoate	7452-79-1	6.086	849	853	102	85	74	0.69	5.19
42	ethyl-3-methylbutanoate	108-64-5	6.208	853	851	88	85	60	-	0.87
43	4-pentenal	2100-17-6	6.392	858	-	55	29	41	-	-
44	Hexanol	111-27-3	6.75	868	867	56	69	55	0.24	-
45	2-methyl-butyl-acetate	624-41-9	7.167	880	880	43	70	55	-	-

46	2-heptanone	110-43-0	7.57	890	889	43	71	59	-	1.59
47	Heptanal	111-71-7	7.945	902	894	70	57	55	0.26	-
48	Methional	3268-49-3	8.111	906	908	48	104	47	-	0.39
49	Anisole	100-66-3	8.485	916	918	108	93	78	2.51	7.30
50	isobutyl isobutyrate	97-85-8	8.55	917	914	71	89	57	-	7.47
51	dimethyl-sulfone	67-71-0	8.795	924	924	79	15	94	-	-
52	isobutyl butyrate	539-90-2	10.085	957	961	71	56	43	-	-
53	Benzaldehyde	100-52-7	10.102	957	961	77	106	105	0.23	1.85
54	1-heptanol	111-70-6	10.617	971	967	70	69	56	-	-
55	1-octen-3-ol	3391-86-4	10.97	980	978	57	72	55	2.06	-
56	3-octanone	106-68-3	11.245	987	988	43	57	29	0.35	-
57	2-octen-4-ona	4643-27-0	11.49	993	-	69	41	84	0.36	0.66
58	hexanoic acid, ethyl ester	123-66-0	11.757	1000	998	88	29	27	-	0.33
59	Butyric acid	2445-67-2	11.8	1002	1002	57	85	103	-	0.31
60	Octanal	124-13-0	11.822	1002	1003	43	84	56	2.90	4.20
61	3-methyl-acid butanoic	589-59-3	12.016	1003	1004	85	57	41	0.62	0.51
62	3-methylanisole	100-84-5	12.384	1018	1028	122	107	92	4.96	1.25
63	Benzeneacetaldehyde	122-78-1	13.277	1043	1047	91	120	92	0.62	-
64	E-2-octenal	2548-87-0	13.803	1058	1059	41	83	70	-	1.24
65	1-octanol	111-87-5	14.286	1071	1067	56	84	70	-	0.65
66	linalool oxide cis	60047-17-8	14.336	1073	1074	59	94	93	-	-
67	3-methyl-phenol	108-39-4	14.502	1088	1083	118	107	79	-	-
68	linalool oxide trans	34995-77-2	14.89	1088	1090	59	94	55	-	-
69	2-nonanone	821-55-6	15.028	1091	1090	43	58	41	-	-
70	isoamyl-2methylbutyrate	27625-35-0	15.331	1101	1103	70	103	85	-	0.37
71	Nonanal	124-19-6	15.424	1103	1106	57	98	70	1.72	-
72	2-methyl-butanoic acid	2445-78-5	15.453	1104	1105	70	57	85	-	1.37
73	Benzeneethanol	60-12-8	15.72	1113	1113	91	122	65	0.33	0.63
74	Benzene, 1,2-dimethoxy-	91-16-7	16.88	1147	-	138	95	77	0.65	-
75	Benzene, 1,3-dimethoxy-	151-10-0	17.507	1167	-	138	109	95	-	-
76	ethyl caprylate	106-32-1	18.516	1198	1196	88	101	57	0.32	-
77	2,4-nonadienal	5910-87-2	18.997	1213	1214	81	41	67	-	0.30
78	2-undecanone	112-12-9	21.412	1293	1296	68	43	59	-	-

504 RT= retention time

505  $RI_{\text{exp}}$  = Retention Index experimental.

506  $RI_{\text{lit}}$  = Retention Index Literature database NIST

507 **Table 2.** Scores and standard deviation obtained in the sensory analysis. For each sensory attribute  
508 (column) and food matrix (honey or oil), letters denote significant differences ( $P \leq 0.05$ ): capital  
509 letters (A-D) denote differences among aromatization processes and/or truffle concentration (for  
510 samples with the same heat treatment), whereas lowercase letters (a-d) denote differences among  
511 heat treatments (for samples with the same aromatization process and truffle concentration).

Sensory attributes									
	Sulphur	Mushroom	Earthy	Butter	Black olives	Leather-animal	Blue cheese	Nuts	Alcohol
HONEY Non treatment									
Control	1.0 ± 0.0C,a	1.0 ± 0.0B,a	1.5 ± 1.1A,a	1.8 ± 1.2B,a	1.0 ± 0.0B,b	1.0 ± 0.0B,a	1.0 ± 0.0A,a	1.4 ± 0.7A,ab	1.3 ± 0.5A,a
F_5	3.1 ± 1.6BC,b	1.8 ± 1.0AB,b	1.8 ± 0.7A,a	2.0 ± 1.2B,b	2.3 ± 1.5B,ab	1.8 ± 1.5AB,a	1.4 ± 0.7A,a	1.8 ± 1.2A,a	1.4 ± 0.5A,a
F_10	3.0 ± 1.4BC,a	2.4 ± 1.4AB,a	2.8 ± 1.9A,a	1.9 ± 1.4B,a	2.1 ± 1.2B,b	2.5 ± 1.5AB,a	1.6 ± 0.9A,a	1.5 ± 0.9A,a	1.6 ± 0.8A,a
L_5	3.1 ± 1.1BC,b	2.1 ± 0.8AB,a	2.4 ± 0.9A,a	2.5 ± 1.3B,ab	2.6 ± 1.8B,ab	2.0 ± 0.8AB,a	2.1 ± 1.1A,a	1.5 ± 1.1A,ab	2.0 ± 1.1A,a
L_10	3.6 ± 1.1B,a	2.3 ± 1.4AB,a	2.9 ± 2.0A,a	2.1 ± 1.6B,a	2.8 ± 1.7B,ab	3.8 ± 2.3A,a	1.9 ± 1.6A,a	2.1 ± 1.8A,a	2.1 ± 1.6A,a
HONEY Heat treatment 1 (Pasteurization)									
Control	1.0 ± 0.0B,a	1.0 ± 0.0B,a	1.1 ± 0.3A,a	1.9 ± 1.3A,a	1.1 ± 0.3B,b	1.1 ± 0.3B,a	1.0 ± 0.0A,a	1.0 ± 0.0A,b	1.2 ± 0.7A,a
F_5	3.2 ± 1.7AB,ab	1.9 ± 0.9AB,b	1.3 ± 0.7A,a	2.1 ± 1.5A,b	1.4 ± 0.7B,b	1.8 ± 0.8AB,a	1.2 ± 0.7A,a	1.4 ± 0.9A,a	1.3 ± 0.7A,a
F_10	2.9 ± 1.3AB,a	2.1 ± 1.3AB,a	2.8 ± 2.3A,a	2.1 ± 2.1A,a	1.8 ± 1.4B,b	2.7 ± 1.9AB,a	1.3 ± 1.0A,a	1.8 ± 1.1A,a	1.6 ± 1.1A,a
L_5	4.2 ± 1.6AB,b	1.9 ± 1.6AB,a	2.3 ± 1.9A,a	2.3 ± 1.7A,b	1.6 ± 1.0B,b	2.4 ± 1.9AB,a	1.4 ± 1.0A,a	1.9 ± 1.5A,ab	1.6 ± 1.1A,a
L_10	3.2 ± 1.8AB,a	2.3 ± 1.8AB,a	3.3 ± 2.1A,a	2.2 ± 1.7A,a	2.1 ± 1.7B,b	3.0 ± 1.4AB,a	1.4 ± 1.0A,a	1.6 ± 1.3A,a	2.0 ± 1.5A,a
HONEY Heat treatment 2 (Canning)									
Control	1.3 ± 0.7B,a	1.3 ± 0.7B,a	1.3 ± 0.7B,a	1.8 ± 1.2A,a	1.0 ± 0.0B,b	1.1 ± 0.4B,a	1.1 ± 0.4A,a	1.4 ± 0.5A,ab	1.3 ± 0.7AB,a
F_5	3.1 ± 1.6AB,b	1.9 ± 1.0B,b	1.9 ± 0.6B,a	2.0 ± 1.3A,b	1.3 ± 0.7B,b	2.1 ± 0.8AB,a	1.1 ± 0.4A,a	1.4 ± 0.5A,a	1.3 ± 0.7AB,a
F_10	2.9 ± 0.8AB,a	2.0 ± 1.2B,a	2.8 ± 1.3AB,a	2.1 ± 1.0A,a	1.8 ± 1.0B,b	3.1 ± 2.0AB,a	1.3 ± 0.7A,a	1.8 ± 1.2A,a	1.6 ± 1.1AB,a
L_5	2.9 ± 1.1AB,b	2.0 ± 1.0B,a	2.8 ± 1.4AB,a	2.1 ± 1.4A,b	1.8 ± 1.1B,b	3.1 ± 1.6AB,a	1.3 ± 0.8A,a	1.8 ± 1.2A,ab	1.6 ± 0.7AB,a
L_10	4.0 ± 1.1A,a	2.0 ± 1.0B,a	3.5 ± 1.4AB,a	2.1 ± 1.4A,a	2.1 ± 1.1AB,b	3.5 ± 1.6AB,a	1.4 ± 0.8A,a	1.9 ± 1.2A,a	2.1 ± 0.7AB,a
OIL Non treatment									
Control	1.1 ± 0.4C,a	1.9 ± 1.9AB,a	1.6 ± 1.1A,a	3.7 ± 1.7AB,a	3.1 ± 2.4AB,a	1.3 ± 0.8B,a	1.0 ± 0.0A,a	1.9 ± 0.7A,a	1.3 ± 0.5A,a
F_5	4.9 ± 1.2AB,a	3.7 ± 1.4A,a	2.3 ± 1.0A,a	3.4 ± 1.3AB,ab	4.4 ± 2.4AB,a	2.0 ± 1.4AB,a	2.0 ± 1.3A,a	1.6 ± 0.5A,a	1.3 ± 0.5A,a
F_10	4.0 ± 2.0AB,a	2.7 ± 2.0AB,a	2.9 ± 2.0A,a	4.6 ± 1.4A,a	5.9 ± 1.3A,a	2.7 ± 2.2AB,a	1.9 ± 1.2A,a	2.0 ± 1.2A,a	2.7 ± 2.6A,a
L_5	6.4 ± 1.7A,a	3.6 ± 2.1A,a	2.6 ± 2.1A,a	4.6 ± 1.1A,a	4.6 ± 2.0AB,a	1.4 ± 0.5AB,a	3.0 ± 1.5A,a	3.3 ± 1.6A,a	2.0 ± 1.4A,a
L_10	4.4 ± 2.6AB,a	3.0 ± 1.3AB,a	3.7 ± 2.7A,a	4.4 ± 1.8AB,a	5.1 ± 2.3AB,a	3.9 ± 2.4A,a	2.9 ± 2.5A,a	2.4 ± 2.0A,a	3.3 ± 2.6A,a
OIL Heat treatment 1 (Pasteurization)									
Control	1.0 ± 0.0B,a	1.0 ± 0.0B,a	1.3 ± 0.8A,a	3.3 ± 1.3A,a	1.0 ± 0.0B,b	1.3 ± 0.8B,a	1.0 ± 0.0A,a	1.0 ± 0.0A,b	1.1 ± 0.4A,a
F_5	4.1 ± 1.9AB,ab	3.1 ± 1.5AB,ab	2.1 ± 1.1A,a	3.7 ± 1.3A,ab	3.3 ± 1.6AB,ab	1.3 ± 0.5B,a	1.6 ± 1.0A,a	1.3 ± 0.8A,a	1.4 ± 0.8A,a
F_10	3.4 ± 1.6AB,a	2.0 ± 1.4AB,a	2.4 ± 1.1A,a	3.7 ± 1.9A,a	3.9 ± 1.3AB,ab	2.3 ± 1.8AB,a	1.9 ± 1.2A,a	1.7 ± 1.0A,a	2.9 ± 1.8A,a



L_5	5.4 ± 1.5A,ab	3.7 ± 1.6A,a	2.0 ± 1.0A,a	4.4 ± 1.7A,a	4.4 ± 2.4A,ab	1.6 ± 0.8B,a	2.4 ± 1.4A,a	1.4 ± 0.8A,b	1.7 ± 1.0A,a
L_10	2.9 ± 2.0B,a	3.1 ± 2.4AB,a	3.3 ± 2.1A,a	4.3 ± 2.8A,a	3.7 ± 1.8AB,ab	4.1 ± 3.0A,a	2.1 ± 2.2A,a	2.7 ± 2.2A,a	3.0 ± 2.6A,a
OIL Heat treatment 2 (Canning)									
Control	1.0 ± 0.0B,a	1.0 ± 0.0B,a	1.3 ± 0.5B,a	3.0 ± 1.4A,a	1.0 ± 0.0B,b	1.3 ± 0.8B,a	1.3 ± 0.8A,a	1.1 ± 0.4A,ab	1.0 ± 0.0B,a
F_5	3.0 ± 2.0AB,b	3.0 ± 0.8AB,ab	2.1 ± 0.7AB,a	4.3 ± 1.6A,a	3.0 ± 2.2AB,ab	1.4 ± 0.5AB,a	2.0 ± 1.5A,a	1.3 ± 0.8A,a	1.7 ± 1.2AB,a
F_10	3.4 ± 2.5AB,a	3.9 ± 2.5AB,a	3.3 ± 2.0AB,a	3.4 ± 2.6A,a	3.6 ± 1.9AB,b	3.0 ± 2.0AB,a	1.9 ± 1.2A,a	1.7 ± 1.0A,a	2.4 ± 1.6AB,a
L_5	3.9 ± 1.7A,b	3.6 ± 1.7AB,a	1.7 ± 0.8B,a	4.1 ± 1.2A,ab	3.6 ± 2.4AB,ab	1.6 ± 0.8AB,a	1.7 ± 1.0A,a	1.6 ± 0.8A,ab	1.6 ± 1.0AB,a
L_10	3.9 ± 1.8A,a	4.4 ± 1.8A,a	4.7 ± 2.7A,a	3.6 ± 2.2A,a	4.6 ± 2.9A,ab	4.0 ± 3.2A,a	2.6 ± 2.1A,a	2.3 ± 1.9A,a	2.9 ± 2.1A,a

513 **Figures**

514 **Figure 1.** Heatmap of the volatile profile of truffled products before and after heat  
515 treatments, according to the relative percentage of area values detected by SPME-GC-  
516 MS. Colors range from white to green (sunflower oil) or orange (honey), showing the  
517 highest 90 percentile in the darkest color. In the sample names, C refers to control  
518 (without truffle), F and L indicate aromatization with fresh or lyophilized truffle, 5 and  
519 10 correspond to truffle amount, and T0-T2 to heat treatment (T0: none, T1:  
520 pasteurization, T2: sterilization).

521 **Figure 2.** PCA results for truffled oil samples: (a) score plot for VOC profile variation  
522 among samples, and (b) loading plot for the VOCs detected by HS-GC-MS. In (a), sample  
523 color indicates the quality of representation for the sample ( $\cos^2$ ). In the sample names,  
524 C refers to control (without truffle), F and L indicate aromatization with fresh or  
525 lyophilized truffle, 5 and 10 correspond to truffle amount, and T0-T2 to heat treatments  
526 (T0: none, T1: pasteurization, T2: sterilization). In (b), compounds are identified with  
527 numbers corresponding to those in Table 1, and arrow color indicates the contribution of  
528 a compound to the PCA components (contrib).

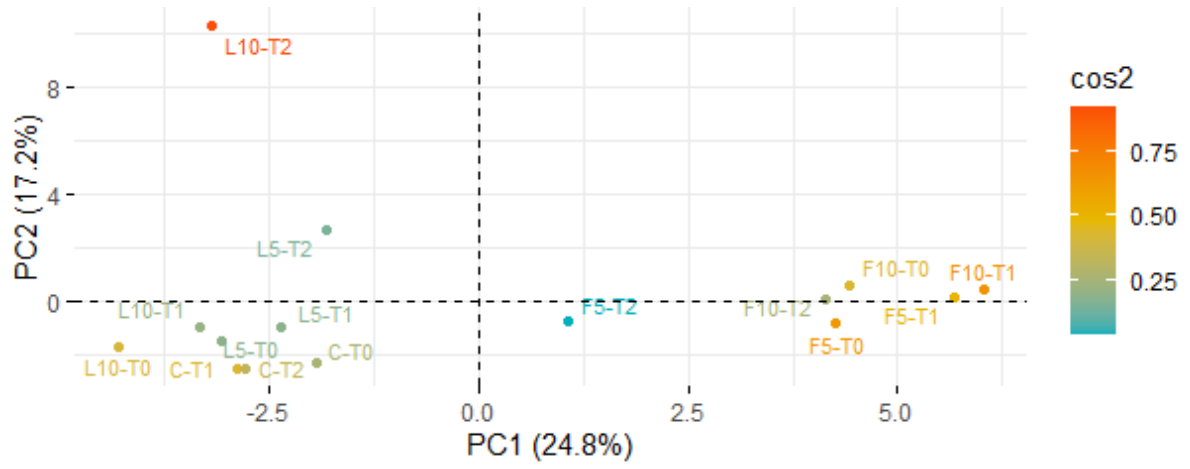
529 **Figure 3.** PCA results for truffled honey samples: (a) score plot for VOC profile variation  
530 among samples, and (b) loading plot for the VOCs detected by HS-GC-MS. In (a), sample  
531 color indicates the quality of representation for the sample ( $\cos^2$ ). In the sample names,  
532 C refers to control (without truffle), F and L indicate aromatization with fresh or  
533 lyophilized truffle, 5 and 10 correspond to truffle amount, and T0-T2 to heat treatments  
534 (T0: none, T1: pasteurization, T2: sterilization). In (b), compounds are identified with  
535 numbers corresponding to those in Table 1, and arrow color indicates the contribution of  
536 a compound to the PCA components (contrib).

537 **Figure 4.** PCA biplot corresponding to odorous attributes in the sensory analysis of the  
538 truffle products. Arrow color indicates the contribution of a compound to the PCA  
539 components (contrib) and sample color indicates the quality of representation for the  
540 sample (cos<sup>2</sup>). In the sample names, O corresponds to oil and H to honey products, C  
541 refers to control (without truffle), F and L indicate aromatization with fresh or lyophilized  
542 truffle, 5 and 10 correspond to truffle amount, and T0-T2 to heat treatments (T0: none,  
543 T1: pasteurization, T2: sterilization).



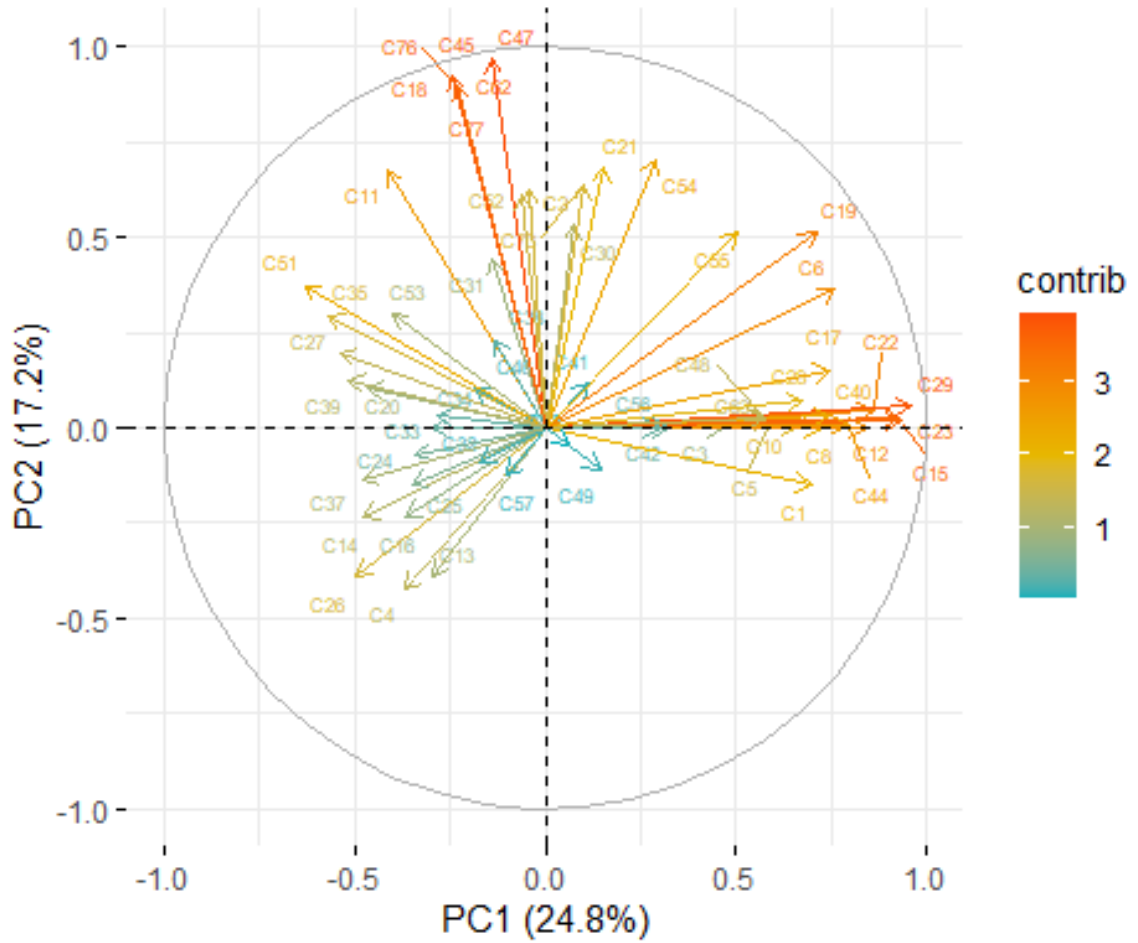
546 **Figure 2**

547 A)



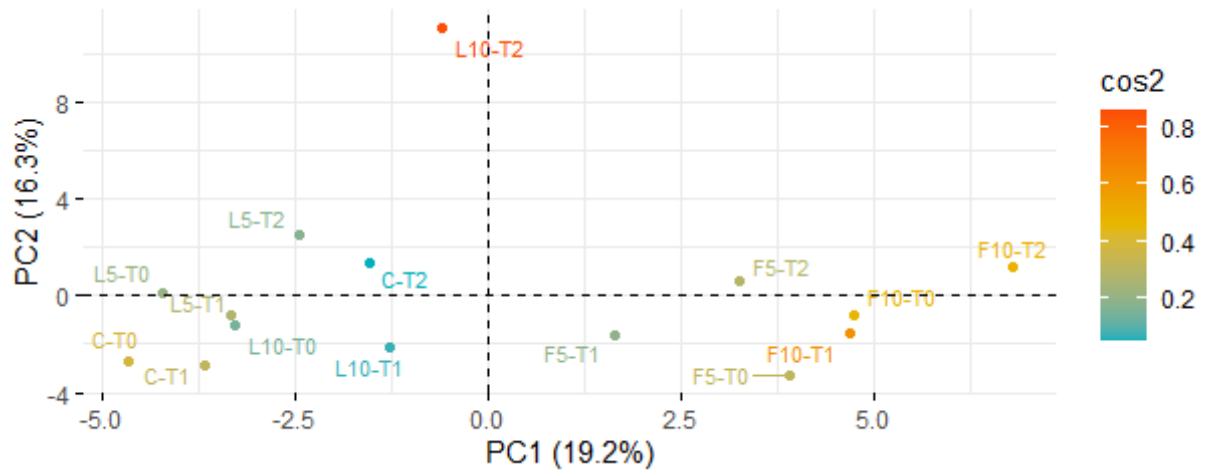
548

549 B)



550 **Figure 3**

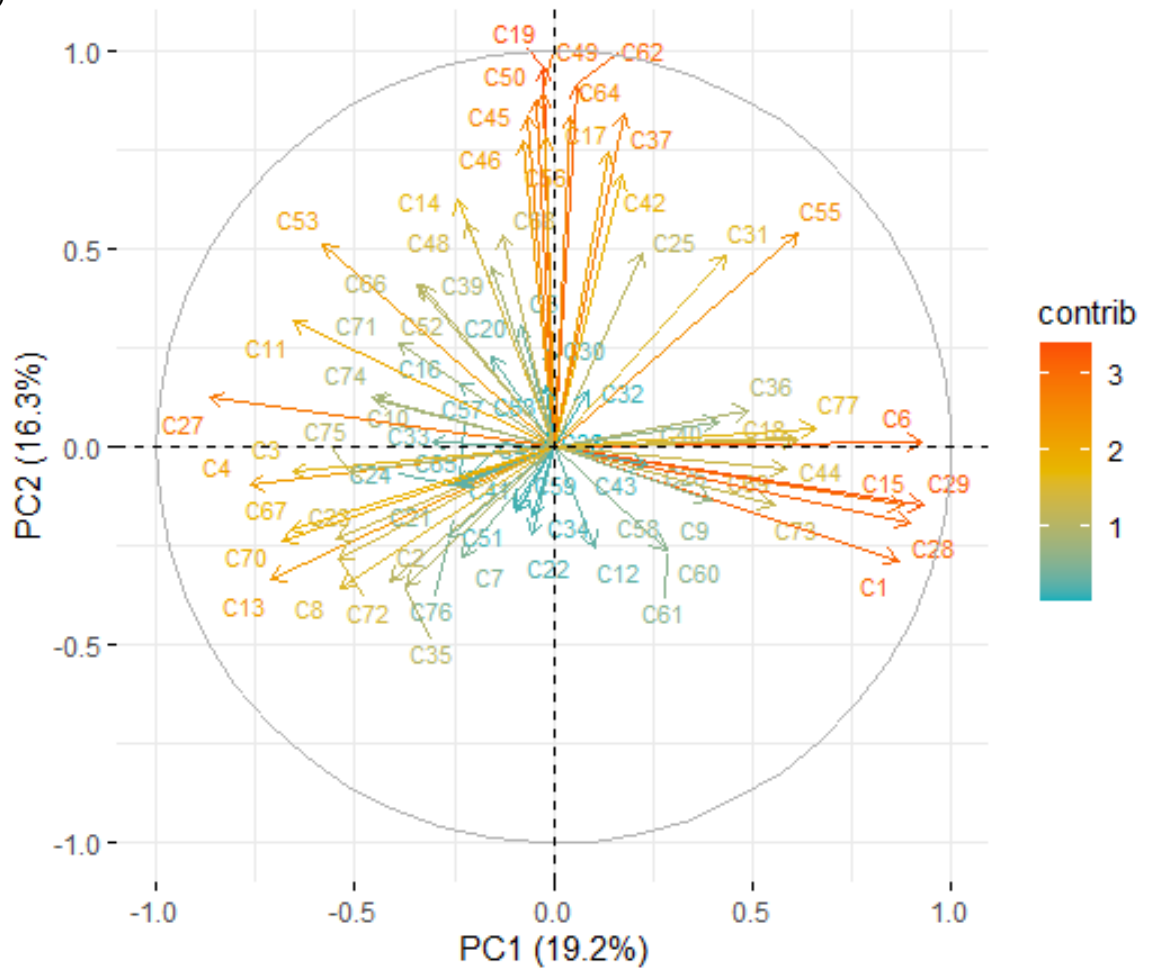
551 A)



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553 B)

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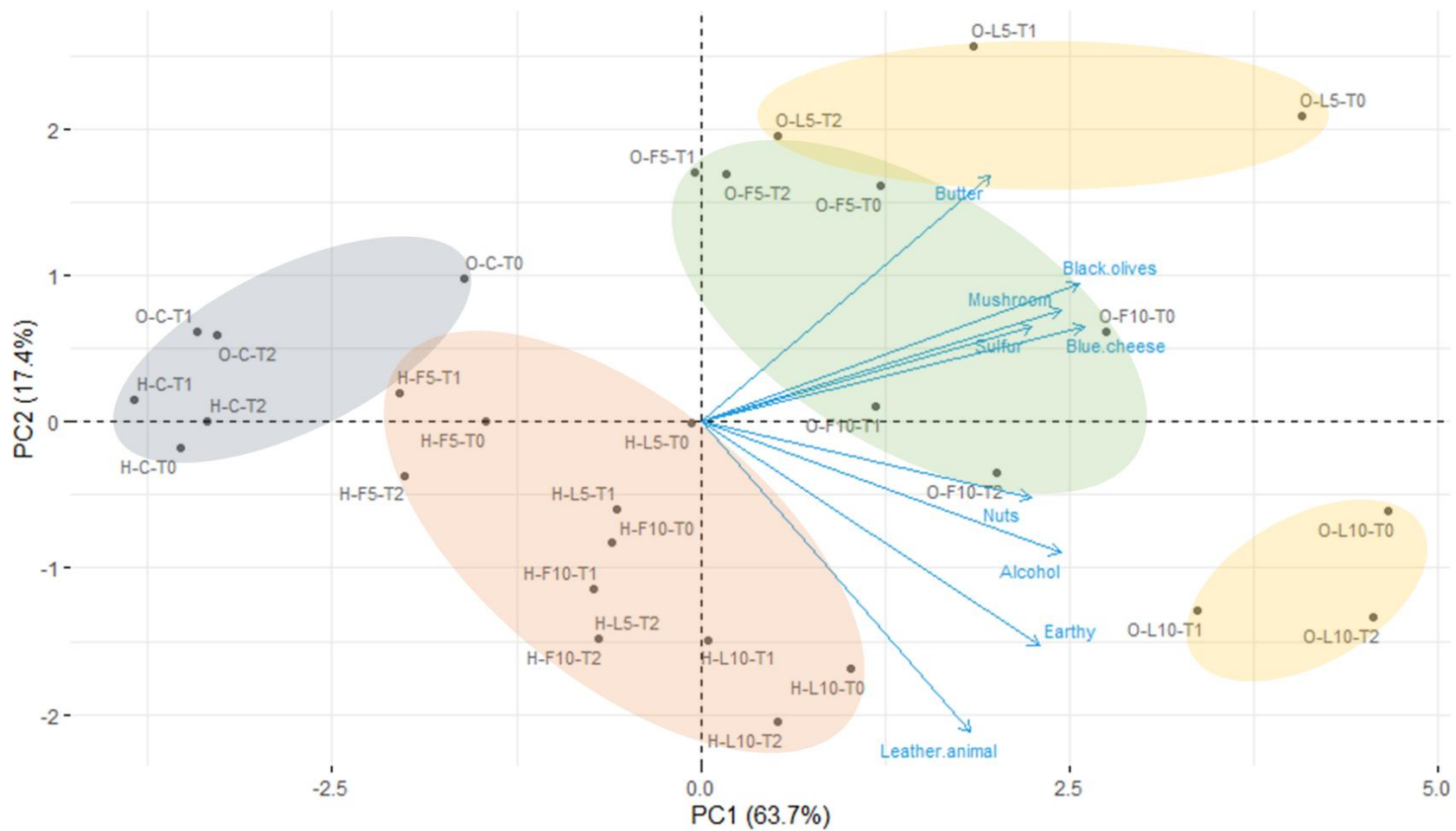


Figure S1.

	OIL														HONEY															
	C_T0	C_T1	C_T2	F5_T0	F10_T0	F5_T1	F10_T1	F5_T2	F10_T2	L5_T0	L10_T0	L5_T1	L10_T1	L5_T2	L10_T2	C_T0	C_T1	C_T2	F5_T0	F10_T0	F5_T1	F10_T1	F5_T2	F10_T2	L5_T0	L10_T0	L5_T1	L10_T1	L5_T2	L10_T2
Ethanol	9.32	7.51	6.49	16.35	9.96	10.46	23.74	27.10	18.12	8.40	2.24	6.44	2.67	1.38	4.16	4.33	2.65	2.84	10.00	11.22	4.93	9.21	5.76	9.89	2.14	1.83	1.59	1.32	1.68	1.41
2-propanone	0.00	0.00	6.72	3.59	6.37	9.09	4.80	3.29	6.33	3.82	2.49	11.75	8.67	14.78	12.49	11.28	5.95	8.64	5.80	2.33	9.22	4.31	8.56	2.41	5.50	7.99	5.44	6.66	2.64	4.18
Dimethyl sulphide	0.00	0.00	0.00	5.97	2.83	3.94	4.68	0.00	3.79	4.56	1.02	6.32	2.75	1.33	1.41	0.00	17.44	9.07	1.55	0.45	0.46	0.38	0.82	0.00	6.63	19.43	14.09	3.60	12.67	2.76
Methylene chloride	58.61	52.08	41.73	1.20	3.64	0.89	0.64	5.24	2.01	4.89	2.73	6.01	3.65	3.04	1.29	26.09	34.44	12.90	4.74	1.40	11.97	10.24	11.71	2.43	25.27	26.41	17.33	38.37	11.11	21.15
1-propanol	0.00	0.00	0.00	0.00	0.00	3.48	2.44	0.00	0.00	0.00	0.00	0.50	0.04	0.00	0.00	0.22	0.33	0.55	0.00	0.00	1.54	0.00	1.16	0.00	0.17	0.25	0.28	0.00	1.03	0.69
Propanal-2-methyl	0.00	0.00	0.00	5.51	6.52	6.41	6.12	0.00	5.54	3.98	0.00	0.00	0.00	5.24	3.43	0.00	0.00	0.00	3.70	3.74	2.09	2.55	1.72	3.35	0.00	0.00	0.00	0.00	0.00	1.68
2,3-butanedione	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.42	0.00	0.48	0.18	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.41	0.00	0.00
Isopropyl formate	0.00	0.00	0.00	0.12	0.07	0.06	0.10	0.00	0.26	0.00	0.00	0.00	0.00	0.00	0.00	0.77	0.32	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.13	0.13	0.00	0.23	0.00	0.00
Butanal	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.92	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3-methyl-2-butanone	0.00	0.00	0.00	0.47	0.45	0.22	0.58	0.00	1.62	0.00	0.00	0.00	0.00	0.00	0.00	0.78	0.88	0.43	1.75	0.00	3.37	0.54	1.13	0.13	3.20	2.48	5.05	1.72	6.92	1.72
Acetic acid	6.20	14.15	7.85	0.00	0.00	0.31	0.00	1.57	0.28	0.00	0.00	0.00	0.00	6.99	27.55	9.77	1.94	3.90	0.00	0.69	0.95	0.00	0.00	0.00	2.52	2.75	10.02	0.00	8.82	6.79
2-butanone	0.00	0.00	0.00	1.93	1.75	0.65	1.43	0.00	0.81	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.25	3.91	9.75	7.26	2.00	3.28	3.32	0.00	2.86	30.26	3.80	0.00
Hexane	20.97	11.53	14.30	0.00	0.00	0.88	0.00	0.54	0.67	0.00	0.00	0.00	0.00	0.00	0.00	9.00	8.16	2.84	1.74	0.00	0.00	0.00	0.00	0.00	6.43	11.65	6.28	0.00	0.00	0.00
Ethyl acetate	0.00	0.00	0.00	0.06	1.35	0.00	0.04	0.00	0.10	58.84	30.93	49.46	27.75	0.00	0.00	0.00	0.18	1.68	0.00	0.98	0.71	0.75	1.43	1.85	4.38	0.00	3.18	0.83	5.79	3.74
2-methyl-1-propanol	0.00	0.00	0.00	8.40	8.82	7.22	8.21	7.39	4.99	0.00	0.00	0.00	0.00	0.00	0.57	0.00	0.00	0.00	4.17	9.27	3.93	4.90	4.41	4.90	0.00	0.00	0.00	0.00	0.00	0.50
Acetaldehyde	0.00	0.34	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.01	0.29	0.00	0.00	0.00	0.00	0.08	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.43	0.00	
3-methyl-butanal	0.00	0.65	0.00	2.12	1.83	11.66	5.23	2.27	4.60	0.28	0.12	0.20	0.38	2.40	1.51	0.25	0.11	2.53	0.50	0.81	0.37	0.56	0.90	1.49	0.36	0.37	0.26	0.13	2.22	2.21
Butanal	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.50	0.00	0.00	0.00	0.00	0.00	0.35	0.81	0.86	0.00	0.00	0.00	0.31	0.00	0.00	
2-methyl-butanal	0.00	0.00	0.00	11.81	12.32	11.72	9.70	9.36	8.35	0.00	0.00	0.54	0.38	14.31	9.46	0.39	0.08	1.92	0.60	0.84	0.86	0.72	1.24	1.93	1.22	1.38	0.92	0.00	3.67	5.77
2-propanone-1-hydroxy	0.00	1.27	0.00	0.00	0.00	0.16	0.02	0.00	0.00	0.04	2.03	0.15	0.00	0.19	0.89	0.29	0.63	0.46	0.01	0.07	0.04	0.05	0.61	0.30	0.21	0.00	0.11	0.67	0.00	0.52
1-penten-3ol	0.00	0.00	0.00	0.00	0.00	0.00	0.39	0.00	0.00	0.00	0.00	0.00	0.00	0.39	0.00	0.00	0.20	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Methylpropylformate	0.00	0.00	0.00	2.38	2.50	1.79	1.46	2.49	1.68	0.16	0.02	0.00	0.00	0.69	0.20	2.13	0.05	0.00	0.83	0.14	0.41	0.08	0.07	0.55	0.00	0.01	0.00	0.00	0.11	0.23
2-pentanone	0.00	0.00	0.00	1.09	1.51	1.12	1.53	0.00	0.82	0.00	0.00	0.00	0.00	0.00	0.00	2.56	1.36	2.37	0.95	0.00	0.60	0.00	0.00	0.00	1.68	0.00	1.38	2.15	0.00	
2,3-pentadione	0.00	1.17	2.35	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.67	0.00	1.34	0.00	0.53	1.21	0.76	0.67	0.38	0.86	3.11	0.18	5.31	0.91	0.20	0.56
Pentanal	0.00	0.66	0.00	0.10	0.11	0.03	0.02	0.46	0.27	0.00	0.66	0.00	0.76	0.71	0.00	0.00	0.21	0.00	0.17	0.65	0.00	0.00	0.33	0.48	0.33	0.42	0.36	0.00	0.00	0.67
2-pentanol	0.00	0.91	1.91	0.00	0.00	0.00	0.00	0.00	0.00	0.88	1.35	0.00	0.00	0.00	0.00	0.00	0.00	1.29	0.75	0.29	0.23	0.60	0.17	0.33	0.00	0.00	0.31	0.00	0.00	
2-butanone,3-hidroxy	0.00	0.00	0.00	0.00	0.00	0.39	0.22	0.00	0.00	2.74	1.41	2.10	1.03	1.76	1.24	6.35	3.55	2.28	1.20	0.00	0.57	0.00	0.00	0.00	3.53	2.86	5.20	1.27	3.92	3.72
3-methyl-1-butanol	0.00	0.00	0.00	14.82	16.87	4.95	2.72	15.49	13.30	0.53	0.43	0.56	0.46	4.57	2.25	0.37	0.10	0.13	11.17	17.06	12.28	21.90	12.58	12.83	0.44	0.40	0.55	0.19	0.57	1.00
2-methyl-1-butanol	0.00	0.00	0.00	22.47	19.62	21.21	23.08	18.15	16.86	2.29	0.72	1.87	0.77	5.64	2.53	0.00	0.16	0.10	22.53	32.79	22.00	25.04	21.30	24.14	0.89	0.76	0.81	0.29	2.78	3.65
Dimethyl-disulphide	0.00	0.00	0.00	0.00	1.71	0.00	0.00	0.00	0.00	0.18	0.00	0.00	0.09	1.02	0.86	0.00	0.15	0.17	0.22	0.51	0.09	0.11	0.42	0.45	0.84	0.59	0.25	0.15	0.35	0.35
2-methyl-pentanal	0.34	0.00	0.00	0.00	0.00	0.00	0.00	0.87	0.45	0.00	0.45	0.00	0.00	0.00	0.80	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.29	0.00	0.00	0.00	0.00	0.00	0.14
Propanoic-ac-2methyl-ester	0.00	0.00	0.00	0.00	0.00	0.20	0.00	0.00	0.00	0.00	0.00	0.12	0.95	0.00	0.00	0.00	0.00	0.31	0.00	0.00	0.00	0.00	0.00	0.13	0.00	0.00	0.00	0.00	0.00	0.00
Isobutylacetate	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.67	0.28	0.36	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.36	0.00	0.00	0.01	0.00	0.00
Propanoic-ac-2methyl	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	21.46	4.59	1.58	0.00	0.00	0.09	0.00	0.04	0.03	0.08	0.00	0.07	0.00	0.00	0.13	0.87	0.00	0.00
1,3-butanediol	4.10	0.00	0.00	0.00	0.00	0.15	0.11	0.27	0.05	0.00	18.46	7.97	24.67	18.87	13.16	1.16	0.49	0.43	0.29	0.06	0.52	0.00	0.20	0.10	0.00	0.00	0.91	0.00	0.00	0.00
Octane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Hexanal	0.00	9.74	18.64	0.82	0.97	1.27	1.17	1.87	2.05	1.36	31.53	2.38	0.00	6.25	5.97	0.00	0.17	0.20	0.38	0.45	0.49	0.48	0.23	0.26	0.00	0.00	0.22	0.00	0.31	2.30
ethylbutanoate	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.28	0.00	0.00	0.00	0.00	0.30	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.51	0.00
Furfural	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.35	0.43	1.32	0.63	0.86	0.60	0.92	0.45	1.89	1.17	11.39	0.63	0.62	0.64	0.46	3.81	3.65	2.23	0.83	1.91	0.76	7.72	3.92
2-methylthio-ethanol	0.00	0.00	0.00	0.03	0.03	0.10	0.11	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.14	0.00	0.39	0.03	0.03	0.06	0.08	0.33	0.45	0.00	0.00	0.06	0.00	0.00	0.00
ethyl-2-methylbutanoate	0.00	0.00	0.00	0.17	0.15	0.17	0.20	0.00	0.10	0.00	0.00	0.00	0.55	0.55	0.00	0.07	0.07	0.00	0.00	0.18	0.00	0.10	0.00	0.00	0.25	0.00	0.00	0.00	0.04	0.00
ethyl-3-methylbutanoate	0.00	0.00	0.00	0.15	0.00	0.02	0.11	0.00	5.28	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.53	0.00	0.12	0.00	0.00	1.08	0.11	0.00					



