Changes in the Volatile Profile, Fatty Acid Composition and Other Markers of Lipid Oxidation of Six Different Vegetable Oils during Short-Term Deep-Frying

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Abbreviations

FAME, fatty acid methyl ester; IV, iodine value; MUFA, monounsaturated fatty acid; *p*-AV, *p*-anisidine value; PUFA, polyunsaturated fatty acid; PC, principal component; PV, peroxide value; SCCs, sulphur containing compounds; SFA, saturated fatty acid SPME, solid-phase microextraction.

Abstract

Oil deterioration during deep-frying influences the quality of fried foods to a great extent. In this study, the frying performance of six vegetable oils, i.e., hemp, lupin, oat, rapeseed, soy, and sunflower, was evaluated following short-term (60 min) deep-frying of French fries at 180 °C. The frying oils were investigated for fatty acid profile, volatile compound composition, and parameters of oxidative stability, such as iodine, peroxide, and *p*-anisidine values. The examination showed that the content of ΣPUFA in hemp oil decreased significantly ($p < 0.05$) after 60 min of deep-frying, although the degree of change was relatively small (close to 1.5%). Similarly, soy oil presented a fatty acid profile prone to oxidation, and generated the highest level of peroxides at the end of the thermal treatment (PV = 16.6 ± 2.3 mEq O_2 kg^{-1}). As for the volatile compound composition of the oils, sunflower oil was extensively affected by the deep-frying treatment with a significant decrease ($p > 0.05$) in total terpenes, accompanied by a considerable rise in total aldehydes. Oppositely, the proportions of MUFA and PUFA of lupin and oat oils remained stable ($p > 0.05$) during the short-term deep-frying, indicating high stability of these oils. The research provided new data for evaluating the suitability of these oils for household food preparations.

Keywords: Hemp oil, Lupin oil, Oat oil, Oxidative stability, Rapeseed oil, Short-term deep-frying, Soy oil, Sunflower oil, Vegetable oils, Volatile aroma compounds.

1. Introduction

In order to satisfy the growing demand for edible oils (Rahoveanu, Rahoveanu, & Ion, 2018), novel crops need to be considered. Amongst the unconventional oil crops, hemp, lupin, and oat are of commercial interest since they can be used to produce oil economically, following the extraction of protein and fibre (Carvajal-Larenas, Linnemann, Nout, Koziol, & van Boekel, 2016). Due to the request from the food and cosmetics industries, several countries have started expanding the cultivation of these crops. For example, in the year 2016 more than 90,000 tonnes of hempseeds were produced worldwide, with Canada and France being the main producers (FAOSTAT, 2018), whereas about 500,000 tonnes of foods containing lupin ingredients were available in 2018 in the EU food market (Department of Primary Industries and Regional Development of Western Australia, 2018). However, hemp, lupin, and oat remain underdeveloped compared to the traditional oil crops, e.g., rapeseed, sunflower, and soy. Indeed, the literature provides a limited number of studies focusing on oils from hemp, lupin, and oat (Sbihi, Nehdi, Tan, & Al-Resayes, 2013; Teh & Birch, 2013) (Ben Halima, Ben Saad, Khemakhem, Fendri, & Abdelkafi, 2015). Several studies have focused on the fatty acid composition of these oils as unheated products (Mikulcova, Kasparkova, Humpolicek, & Bunkova, 2017; Rybinski et al., 2018) and the changes occurring as a result of cooking, e.g., deep-frying, are largely unexplored. On the contrary, common edible oils from rapeseed, soy, and sunflower, have been widely investigated, although research has focused on their behaviour under long-term cooking, e.g., several cycles of deep-frying, as operated by the food industry (Giuffre, Capocasale, Zappia, & Poiana, 2017; Molina-Garcia, Santos, Cunha, Casal, & Fernandes, 2017). Nevertheless,

66 changes in the chemical and sensory characteristics of oils take place even during
67 short-term deep-frying (Akil et al., 2015), which is relevant to household practises.

68 Deep-frying is a cooking technique carried out in both industrial and domestic
69 kitchens, performed by submersing the food in oil heated at temperature ≥ 180 °C
70 (Liu, Wang, Cao, & Liu, 2018). Deep-fried foods are popular amongst consumers
71 because of their sensory characteristics, e.g., flavour, colour, and texture (Miyagi,
72 2017). The chemical and sensory stability of frying oils is dependent on the
73 temperature and the length of the deep-frying process (Giuffre et al., 2017). Several
74 types of vegetable oils, e.g., oils from olive, rapeseed, sunflower, soy, coconut,
75 peanut, and palm, were studied in deep-frying experiments (Santos, Cunha, & Casal,
76 2017). Deep-frying causes thermal oxidation that leads to changes in the fatty acid
77 composition of oil and development of peroxides (Wang et al., 2016). Peroxides (and
78 hydroperoxides) are primary oxidation products characterised by high instability that
79 degrade easily into secondary products. Most of the secondary products have low
80 molecular weight and are volatile, with aldehydes, ketones, alcohols, acids, and
81 furans being the dominating compounds (Perestrelo, Silva, Silva, & Camara, 2017).
82 These molecules influence the aroma of the oil and can be identified by headspace
83 solid-phase-micro extraction coupled with gas chromatography and mass
84 spectrometry (HS-SPME/GC-MS) (Sghaier et al., 2016).

85 In this study, three novel vegetable oils (hemp, lupin, and oat) and three
86 conventional oils (rapeseed, soy, and sunflower), all from commercial sources, were
87 studied to evaluate the changes in the volatile profile and fatty acid composition
88 during short-term deep-frying. In addition, parameters of the oxidative stability, e.g.,
89 iodine, peroxide, and *p*-anisidine values were evaluated through established

analytical methods to assess the suitability of the selected oils for short-term deep-frying.

2. Materials and methods

2.1. Chemicals and materials

Lupin oil from blue sweet lupin *(Lupinus angustifolius)* was purchased from Prolupin GmbH (Grimmen, Germany). Oat oil (Sweoat® Oil PL4) was purchased from Swedish Oat Fiber AB (Bua, Sweden). Hemp, rapeseed, soy, and sunflower oils were purchased from a local supermarket in Turku, Finland. The oils were cold-pressed and unrefined, apart from rapeseed oil that was a conventional refined oil. Being commercial products, the frying oils were without addition of antioxidants and were used before their best before date (oils were stored for about six months after production). Fresh potatoes were purchased from a local supermarket. The reference compounds for volatile analysis were purchased from Sigma-Aldrich (Espoo, Finland): hexanal, nonanal, α-pinene, camphene, β-pinene, myrcene, α-phellandrene, δ-3-carene, α-terpinene, *p*-cymene, limonene, γ-terpinene, terpinolene, eucalyptol, terpinen-4-ol, bornyl acetate, β-caryophyllene, and a homologous series of *n*-alkanes (C7−C22). *p*-Anisidine (≥ 99%) was purchased from Sigma-Aldrich (Espoo, Finland). The Supelco 37-Component FAME Mix and Supelco SPME fibre (DVB/CAR/PDMS) were purchased from Sigma-Aldrich (Espoo, Finland). Potassium iodide, sodium thiosulphate (anhydrous, reagent grade), potato starch, and all the general laboratory reagents were purchased from VWR International Oy (Helsinki, Finland).

2.2. Deep-frying conditions

Food can influence the characteristics of the frying medium to a different extent. In order to evaluate the quality of the frying oils as utilised by consumers, potatoes were selected to perform the deep-frying experiment because of their great popularity worldwide. The deep-frying procedure was adapted from Akil et al. (2015). A domestic deep-fryer (DeLonghi, F28-311; 310x360x240 mm) of 1.2 L capacity was employed in the short-term deep-frying experiment, performed at 180 °C (the thermostat of the deep-fryer was set at 180 °C, however, the real oil temperature was periodically controlled by an analytical temperature probe that measured 183 ± 2 °C). Prior to frying, potatoes were peeled, washed, drained, and cut in uniform pieces (approximately 7 x 1 x 1 cm^3). Oils (1.0 L) were introduced in the deep-fryer, and after equilibrating for 15 min at 180 °C, potatoes were deep-fried for 5 min in batches of 200 ± 2 g. After each frying batch, 80 mL of oil were filtered and collected in glass bottles. Subsequently, the oil volume was replenished to 1 L, followed by 15 min of re-equilibration to 180 °C. This sequence was repeated three times, and four samples were taken at different time points: unheated (0 min), T20 (20 min), T40 (40 min), T60 (60min). Following each collection, the oil samples were cooled in an ice bath, flushed under nitrogen and stored in the dark at -20 °C until analysis. Figure 1 illustrates the design of the deep-frying experiment. This short-term deep-frying method was chosen to reproduce the conditions that are typically employed in household kitchens.

2.3. Analysis of fatty acid composition and calculation of iodine value (IV)

2.3.1. Preparation of fatty acid methyl esters (FAMEs)

Fatty acid methyl esters were prepared by adapting the sodium methoxide method of Stoffel et al. (1959). Briefly, 5 mg of oil were dissolved in 10 mL of hexane. Then, 500 µL of this solution were transferred into a glass tube and 1 mL of 1,2,3-triheptadecanoylglycerol (0.018 mg mL^{-1}) was added as internal standard. The mixture was evaporated to dryness under a stream of nitrogen. Subsequently, the dried samples were suspended in 1 mL of dry diethylether; then 25 µL of methylacetate and 25 µL of sodium methoxide were added, and the mixture was incubated for 5 min with shaking. The reaction was stopped by addition of 6 µL of acetic acid (glacial). This was followed by centrifugation at 2000 × *g* for 5 min. Then the supernatant was collected and evaporated to dryness under a stream of nitrogen. The dry residue containing the FAMEs was dissolved in 1 mL of hexane and analysed with the gas chromatograph.

2.3.2. GC-FID analysis of FAMEs

FAMEs were quantified by GC-FID analysis, as described by Kumar et al. (2016). The instrument employed was a GC-2010 equipped with AOC-20i auto injector (Shimadzu,Kyoto, Japan). The chromatographic separation of FAMEs was obtained using a capillary DB-23 column (60 m × 0.25 mm i.d.; 0.25 µm film thickness) from Agilent (Palo Alto, CA). Helium was used as the carrier gas. Splitless injection was used, and the split valve was opened after 1 min. The injection volume was 0.5 µL, and inlet temperature was 270 °C. The initial oven temperature was 130 °C (held for 1 min). The oven temperature was programmed to rise at a rate of 4.5 °C min^{-1} to

159 170 °C and 10 °C min^{-1} to 220 °C (held for 3.5 min), and further at 10 °C min^{-1} to 230
160 °C and 60 °C min^{-1} to 240 °C (held for 7 min). The detector temperature was 280 °C.
161 Peaks were identified by comparing their retention times to an external standard
162 mixture, Supelco 37 Component FAME Mix (Supelco). FAMEs were quantified in
163 relation to the internal standard and corrected with response factors calculated
164 based on the analysis of the standard mixture. The results are expressed as
165 percentage (%) composition of fatty acid (FA weight percentage (%) of total fatty
166 acids).

167 Iodine values (IV) of oils were calculated on the basis of the fatty acid composition,
168 using the AOCS official method (method Cd 1c-85) (AOAC, 2011).

169

170 *2.4. Oxidative stability of oils*

171 Quality indices of the oxidative stability of oils were investigated. The peroxide value
172 of oils was determined using the AOCS official method (method Cd 8-85) (AOAC,
173 2011) and expressed as meq O_2 kg^{-1}. The *p*-anisidine value (*p*-AV) was determined
174 using the AOCS official method (method Cd 18-90) (AOAC, 2011).

175

176 *2.5. Analysis of Volatile Organic Compounds (VOCs)*

177 *2.5.1. Extraction by SPME*

178 Volatile organic compounds were extracted as described by Marsol-Vall et al. (2018).
179 Each oil sample (2.0 g) was placed in a 20-mL headspace vial. After pre-conditioning
180 the samples for 10 min at 45 °C, the HS-SPME of the volatile fraction was performed
181 with a 2 cm SPME fibre CAR/PDMS/DVB (Carboxen/ Polydimethylsiloxane/

Divinylbenzene; 50/30 µm) from Supelco, at 45 °C for 30 min, applying agitation using a TriPlus RSH multipurpose autosampler (Thermo Scientific, Reinach, Switzerland).

2.5.2. GC-MS conditions

GC-MS analyses were performed with a Trace 1310 (Thermo Scientific) gas chromatograph coupled to a TSQ 8000 EVO mass spectrometer (Thermo Scientific). The SPME fibre was desorbed into the injection port equipped with an 0.8 mm i.d. SPME liner (Restek, Bellefonte, PA) at 240 °C for 3 min. Compounds were separated with a Supelco SPB-624 column (60 m x 0.25 mm i.d.; 1.4 µm film thickness), using helium as carrier gas (1.2 mL min^{-1}). The oven temperature was programmed from 50 °C (held for 2 min) to 220 °C at a rate of 5 °C min^{-1}, then held for 12 min at 220 °C. Mass spectra were recorded in electron impact (EI) mode at 70 eV within the mass range *m/z* 40–300. The transfer line and the ionization source were thermostated at 250 and 220 °C, respectively. The system was operated using Xcalibur 4.0 (Thermo Scientific).

VOCs were identified based on authentic standards when available, or tentatively identified by comparing the experimental spectra with those from Wiley 7 and Essential Oils mass spectral libraries (Wiley, New York, NY). Positive match was considered when library direct match was higher than 800. Linear retention indices (RI) were calculated using an *n*-alkane mixture (C7: C22). The software Xcalibur 4.0 was used to perform the peak detection and integration. The peak detection settings were set at an area/noise ratio > 20, and an area under the peak higher than 2 10^6 units. Semi-quantitative data (percentage of total volatile composition) were directly

206 calculated from the peak areas of the total ion chromatogram (TIC), assuming no
207 differences in the response factors among all the volatiles quantified.

208

209 *2.6. Statistical analysis*

210 All the analytical determinations were performed with four replicates, n = 4. Data are
211 expressed as mean ± standard deviation (SD). Univariate analysis was performed
212 using SPSS 23.0 for Windows (IBM, Armonk, NY, USA). Data were analysed using
213 one-way ANOVA to compare the groups, and the Tukey's HSD test was performed
214 to allow for multiple comparisons. Differences among groups were considered
215 significant at $p < 0.05$. PCA was performed using SIMCA-P^+ 15.0 (Umetrics, Umeå,
216 Sweden). The variables included in the model were selected on condition that they
217 were present at least in 40% of the observations.

218

3. Results and discussion

3.1. Fatty acid composition

Figure 2 shows that hemp had a unique fatty acid profile and differentiated from the other oils mainly due to the presence of γ-linolenic (18:3n-6) and nonadecanoic (19:0) acids. The concentrations of these two fatty acids were affected by the treatment as both fatty acids significantly decreased ($p < 0.05$) following deep-frying (Table 1a). The reduction was particularly noticeable (from 2.82 to 2.77%, $p < 0.001$) for γ-linolenic acid after 60 min of deep-frying (T60). Compared to the other oils, unheated hemp oil provided the highest amount of total PUFA (75.0 %), which showed low resistance to deep-frying, as the total percentage of PUFA lowered to 73.9% at T60 ($p < 0.001$). Amongst the PUFA, linoleic (18:2n-6, 54.0%) and α-linolenic (18:3n-3, 18.1%) acids were the most abundant in the unheated oil. The high content of α-linolenic acid distinguished the fatty acid composition of hemp oil from the rest of the oils, since the other oil samples showed much lower levels of α-linolenic acid. To the authors' knowledge, this is the first investigation examining the effects of deep-frying on the fatty acid composition of hemp oil. However, results from the present study agree with those reported for unheated hemp oil by other authors, in which linoleic acid resulted the main fatty acid, followed by α-linolenic and oleic (18:1n-9) acids (Aladic et al., 2015; Gao & Birch, 2016; Mikulcova et al., 2017; Teh & Birch, 2013).

Due to the availability of varieties of lupin low in alkaloids (*L. angustifolius*), the cultivation of lupin in Europe and South America is expanding (Schweiggert, Cornfine, Eisner, & Hasenkopf, 2010). However lupin oil is hardly available on the market. Lupin beans are employed mostly as sources of protein and dietary fibre, whereas the oil is considered a by-product. Data from the present investigation

showed that linoleic acid was the most abundant fatty acid in unheated lupin oil (38.5%), followed by oleic (31.3%) and palmitic (16:0, 11.1%) acids. These three fatty acids accounted for more than 80% of the total fatty acids. The tested lupin oil from *L. angustifolius* provided higher amount of linoleic acid and lower amount of oleic acid than oils from white lupins (*L. albus*), which contained about 50% of oleic acid and 20% of linoleic acid (Rybinski et al., 2018; Sbihi et al., 2013), suggesting that the fatty acid composition is influenced by genetic differences amongst the cultivars. The tested unheated lupin oil contained relatively high percentages of palmitic (11.1%) and stearic (5.99%) acids. These two fatty acids contributed to a large extent to the content of ΣSFA (22.6%), which was the highest amongst the selected oils (Table 1a). PCA analysis (Figure 2b) revealed that lupin differed from the other oils due to the presence of 22:0, 22:6 (n-3), 22:3 (n-3), 23:0, and 24:0. Lupin located in the lower left quarter of PC1, and was the richest in these long-chain fatty acids, which were scarce in the other samples.

The fatty acid analysis of unheated oat oil revealed that oleic acid was the predominant (40.3%), followed by linoleic and palmitic acids (36.5 and 14.9 %, respectively). Ben Halima et al. (2015) wrote a review on the chemical composition of oats and reported that palmitic, oleic and linoleic acids make up about 90% of the total fatty acids of oat oil, regardless of the extraction method, crop variety and location of growth. It is worth noting that the high concentration of oleic acid might confer superior oxidative stability to oat oil (Dorni, Sharma, Saikia, & Longvah, 2018). In this investigation, the relative proportion of oleic acid remained stable ($p > 0.05$) during the short-term deep-frying, likewise the total content of MUFA. The concentration of α-linoleic acid was unaffected by short-term deep-frying (1.24% at T60; $p > 0.05$).

Analogous to hemp, rapeseed oil discriminated greatly from the other selected oils as shown in the PCA plot (Figure 2a). Unheated rapeseed provided the highest concentration of oleic acid (57.7%), and consequently of total MUFA (62.1%), whereas it had the lowest content of total SFA (9.11 %), being relatively low in palmitic and stearic acids (4.74 and 1.77%, respectively). Unheated rapeseed oil also contained a moderate amount of linoleic acid (19.0%). This fatty acid profile was comparable to that reported by Mba et al. (2017). Table 1b indicates that after 60 minutes of deep-frying, a loss of linoleic (from 19.0 to 18.8%; $p < 0.05$) and α-linolenic acids (from 8.40 to 8.09%; $p < 0.05$) occurred in the rapeseed oil. On the contrary, the relative proportions of stearic acid significantly increased at the end of the treatment (from 1.77 to 1.83%; $p < 0.05$).

Unheated soy oil was characterised by high levels of linoleic acid (52.9%), followed by oleic, palmitic, and α-linolenic acids (18.8, 10.1, and 8.89%, respectively). The fatty acid composition of the selected soy oil found confirmation in the literature (Liu et al., 2018). As it can be observed in Table 1b, the deep-frying process caused a significant increase ($p < 0.001$) of ΣMUFA already at T20 (from 20.4 to 22.6%), opposite to a decline ($p < 0.01$) in ΣPUFA (from 61.8 to 58.9% at T20). These changes were mainly due to an increase in the proportion of oleic acid (from 18.8 to 20.9% at T20; $p < 0.001$), along with a decrease in linoleic and α-linolenic acids (50.9% and 8.00%, at T 20 respectively; $p < 0.001$). Indeed, linoleic and α-linolenic acids are readily prone to oxidation since they contain two and three double bonds, respectively; whereas oleic acid is less sensitive, as it contains only one unsaturation. It is noteworthy that after an initial increase at 20 min, the proportions of palmitic, stearic and oleic acids remained stable ($p > 0.05$) up to the end of the deep-frying process (T60). This suggests that the decomposition of soy oil was more

294 influenced by the rise of temperature (180 °C) than the length of the cooking
295 process. In line with this observation, previous authors (Liu et al., 2018; Wang et al.,
296 2016) argued that deep-frying affects the quality of fatty acids from soy oil; however,
297 once triggered, the changes tend to be stable during the process.

298 Amongst the studied oils, unheated sunflower provided the highest amount of linoleic
299 acid (58.5%) and the lowest amount of α-linolenic acid (0.12%) (Table 1b). In
300 addition, sunflower oil provided moderate amounts of oleic acid (26.1%) and of ΣSFA
301 (12.9%), with palmitic acid being the most abundant SFA (6.44%). Our data are in
302 agreement with the fatty acid profile reported by Dorni et al. (2018) and Aladedunye
303 and Przybylski (2014). With regard to the composition in SFA, unheated sunflower
304 and soy oils showed similar profiles, mostly due to the concentrations of tridecanoic,
305 myristic, and stearic acids. The PCA model (Figure 2a) showed that sunflower oil
306 clustered with soy oil. Short-term deep-frying caused clear changes in the fatty acid
307 composition. The relative content of ΣSFA increased after 40 min of heating (from
308 12.9 to 13.3%; $p < 0.01$), whereas that of ΣPUFA decreased already after 20 min
309 (from 59.8 to 58.4%; $p < 0.01$). These changes were mainly due to tridecanoic,
310 pentadecanoic, and palmitic acids increasing, whereas linoleic acid decreased.
311 During deep-frying, linoleic acid is oxidized and degraded to aldehydes of about six
312 carbons, which can polymerise and generate fatty acids with skeleton ≥ 12 carbons
313 (Li, Li, Wang, Cao, & Liu, 2017). Although a high intake of PUFA such as linoleic
314 acid might provide protection against coronary heart diseases (Farvid et al., 2014),
315 an elevated content of linoleic acid as in sunflower is not desirable in frying oils,
316 since the high degree of unsaturation makes the oil prone to oxidation. In general,
317 the changes in the fatty acid composition after the deep-drying were significant ($p <$
318 0.05) but small in absolute values, likely due to the short frying time.

319 The iodine value (IV) provides information about the overall degree of unsaturation of
320 oils directly from the fatty acid composition (AOAC, 2011). It is a parameter
321 employed by the food industry to monitor the degree of hydrogenation of oils (Lirong,
322 Xufei, Xiuzhu, Zongyao, & Xingguo, 2018). The Codex Standard (FAO, 2013) for
323 vegetable oils provides guidelines on the levels of IV that conventional oils should
324 contain to be considered of good quality: approximately 100-150 g I_2 100 g^{-1} of oil.
325 The rapeseed, soy and sunflower oils included in this study possessed IV that fell in
326 this range. Oppositely, the Codex Standard does not provide references for novel
327 oils, such as hemp, lupin, and oat oils, the iodine values of which are still to be
328 comprehensively established. In this work, the short-term deep-frying treatment
329 produced a decrease in IV across the samples, apart from the oat oil. Oat had the
330 lowest IV (101 g I_2 100 g^{-1} of oil), nevertheless, IV did not statistically differ ($p > 0.05$)
331 between unheated and heated oil. This might be explained by the stability of MUFA
332 and PUFA in oat oil ($p < 0.05$) during short-term deep-frying, as well as the
333 antioxidants naturally present in the oil. The decline of IV in the other oils might be
334 ascribed to the development of volatile compounds and polymers from the
335 unsaturated fatty acids (Giuffre et al., 2017).

336

337 *3.2. Quality indices of oxidative stability*

338 The oxidative stability of the selected oils was determined by evaluating conventional
339 quality parameters of fatty acid oxidation. These parameters were the peroxide value
340 (PV) that assesses the formation of primary oxidation products, e.g., peroxides and
341 hydroperoxides, and the *p*-anisidine value (*p*-AV) that assesses the formation of
342 secondary oxidation products, namely non-volatile aldehydes (Teh & Birch, 2013).

343 The unheated oils had PV ranging from 3.50 to 8.96 mEq O_2 kg^{-1} in rapeseed and
344 oat oils, respectively (Figure 3a). All unheated oil samples had PV within the legal
345 limits, i.e., ≤ 20 and ≤ 10 mEq O_2 kg^{-1} for virgin and vegetable oils, respectively
346 (FAO, 2013). Hemp and lupin oils were the most sensitive to oxidation, as
347 statistically significant increases in PV (p < 0.01) were observed after 20 min of
348 deep-frying (7.46 and 6.96 mEq O_2 kg^{-1} for hemp and lupin, respectively). Soy oil
349 resulted relatively more stable as its PV rised significantly only after 60 min of deep-
350 frying (from 8.66 to 16.6 mEq O_2 kg^{-1}; p < 0.001). Being a refined oil with the majority
351 of peroxides removed, rapeseed oil presented the lowest PV, which remained steady
352 throughout the deep-frying process, suggesting good oxidative stability. This might
353 be explained by the high levels of ΣMUFA (Casal, Malheiro, Sendas, Oliveira, &
354 Pereira, 2010). On the other hand, peroxides (and hydroperoxides) are unstable
355 molecules and do not usually accumulate during cooking, instead they decompose
356 into secondary oxidation compounds (Giuffre et al., 2017). Indeed, as a result of
357 deep-frying the rapeseed oil yielded comparatively high *p*-anisidine values, reflecting
358 high levels of secondary oxidation products.

359 Edible oils are considered to be acceptable when the *p*-anisidine value is below 10
360 (Giuffre et al., 2017). This indicates the nearly absence of non-volatile aldehydes,
361 (Casal et al., 2010). All the unheated oils had *p*-AV ≤ 10 (Figure 3b). As regard to the
362 influence of deep-frying on *p*-AV, the selected oils performed similarly, as *p*-AV
363 increased markedly (p < 0.05) after 20 min of deep-frying, with an average increase
364 of about 10-fold. Sunflower oil was the most prone to develop non-volatile aldehydes
365 as it gave the highest *p*-AV at all the time points (27.6, 27.5, 38.0 at T20, T40 and
366 T60, respectively). The *p*-AV of lupin and soy oils remained stable (p > 0.05) during
367 deep-frying, suggesting a higher resistance to fatty acid oxidation than the other oil

368 samples. Previous research has associated the development of aldehydes with the
369 total amount of PUFA, which are targets of thermal oxidative reactions (Aladedunye
370 & Przybylski, 2014; Gao & Birch, 2016; Nosratpour, Farhoosh, & Sharif, 2017; Wang
371 et al., 2016). This hypothesis is reinforced by the present study, in which strong
372 correlations were found between ΣPUFA and *p*-AV of the selected frying oils (Table
373 S2). The Sunflower oil, rich in ΣPUFA, produced high levels of *p*-AV ($r = -0.778$; $p < 0.001$). Nevertheless, the formation of *p*-AV cannot be solely attributed to the high
374 0.001). Nevertheless, the formation of *p*-AV cannot be solely attributed to the high
375 percentage of ΣPUFA. Our data showed that hemp oil yielded comparatively low
376 levels of *p*-AV, although it provided the greatest percentage of ΣPUFA. It is likely that
377 other factors played a role in the degradation of edible oils, such as the presence of
378 antioxidant compounds. This investigation makes firmer that short-term heat
379 treatments lead to the development of aldehydes regardless of the type of oil
380 employed (Akil et al., 2015). Compared to PV, the *p*-AV is a more reliable test, since
381 it measures oxidation products that are more stable than peroxides (Wang et al.,
382 2016). Nevertheless, the measurement of peroxides is compulsory prior to oil
383 commercialisation, despite the fact that the assay cannot be directly correlated to the
384 oxidative state of the sample. For this reason, peroxide and *p*-anisidine values
385 should be interpreted together to perform an assessment of the status of oils.
386 Considering these two parameters simultaneously, our results showed that short-
387 term deep-frying, as in household preparations, caused oil degradation of all the oils
388 employed in the study.

389

390 *3.3 Volatile organic compounds (VOCs)*

391 The volatile profile of unheated hemp oil consisted of 43.5% of alcohols, 15.9% of
392 aldehydes, 5.81% of alkanes, 7.57% of furans, and 17.4% of terpenes (Table S3a).

393 The profile changed markedly when the oil was exposed to deep-frying. Alcohols
394 decreased greatly, e.g., at T20 1-pentanol and hexanol reduced from 4.56 and
395 31.2%, to 0.85 and 1.88%, respectively. Similarly, terpenes decreased considerably,
396 i.e., from 17.4 in the unheated to 3.02% in T20 oils. On the contrary, the relative
397 content of aldehydes increased during deep-frying, i.e., from 15.9 in unheated to
398 52.7% in T20 oils. This increase was apparent in acrolein, (*E*)-2-heptenal, and 2,4-
399 heptadienal isomers, which after 20 min of deep-frying, reached 8.53, 7.56, and
400 10.9%, respectively. These conjugated aldehydes are oxidation products that
401 develop during deep-frying (Eskin & Shahidi, 2012; Fullana, Carbonell-Barrachina, &
402 Sidhu, 2004; Katragadda, Fullana, Sidhu, & Carbonell-Barrachina, 2010). Acrolein is
403 a very reactive α,β-aldehyde that is considered a potential health hazard (Rietjens et
404 al., 2018). Acrolein was detected at relatively high concentrations throughout the
405 frying process, e.g., 7.55% after 60 min. Alkanes behaved similarly to aldehydes,
406 peaking to 20.0% at T20. Amongst alkanes, pentane showed the greatest increase,
407 e.g., from 3.73% in unheated to 17.6% in T20 oils. Hexanol and total terpenes
408 tended to be negligible at the end of the deep-frying, whereas unsaturated aldehydes
409 increased when the frying time lengthened. This is the first investigation exploring
410 the effects of short-term deep-frying on the volatile profile of hemp oil.

411 Unheated lupin oil presented a volatile profile characterised by high concentrations
412 of alcohols (51.7%), aldehydes (17.8%) and lactones (9.46%). Acetic acid is a
413 volatile formed during the preliminary processing of the oil crops (Ivanova-Petropulos
414 et al., 2015). Amongst the selected oils, unheated lupin oil was the only sample to
415 provide acetic acid (8.67%). According to Asghar Amanpour et al. (2016), acetic acid
416 is a main aroma compound of olive oils. The short-term process produced several
417 changes in the aroma profile of lupin oil: acetic acid faded out upon deep-frying,

418 alcohols decreased to 7.19% after 20 min due to the drop in hexanol (3.75%), and
419 aldehydes increased greatly after 20 min (53.4%) and remained above 50%
420 throughout the process. In particular, hexanal, (*E*)-2-heptenal, 2,4-heptadienal
421 isomers, (*E*)-2-octenal, and nonanal affected deeply the volatile profile of the heated
422 lupin oil. Total alkanes increased sharply after 40 min of deep-frying, due to a rise in
423 the concentrations of pentane (5.79%). Total furans peaked to 26.4% at T60, owing
424 to the sharp increase in 2-pentylfuran (26.0%). 2-Pentylfuran is formed during
425 heating of oils, likely due to a degradation of linoleate (Vichi, Pizzale, Conte,
426 Buxaderas, & López-Tamames, 2003). Total lactones were negatively associated
427 with deep-frying, as they decreased steadily during the frying process, dropping to
428 1.46% after 60 min of treatment. In a previous study performed on the same lupin oil
429 by our research group, few terpenes were detected in the unprocessed oil, e.g., α-
430 thujene, *p*-cymene, α-phellandrene, and limonene (Multari et al., 2018). These
431 odour-active volatiles were linked to citrus, grass, and pine aromas. As these
432 volatiles were not found in lupin oil following heat treatment, it is clear that the deep-
433 frying process removed the compounds.

434 Amongst the selected oils, unheated oat oil presented a unique volatile profile. Oat
435 oil was the only to provide acetals, which represented 16.1% of total VOCs. Ethanol
436 was by far the most abundant compound (73.1%), representing nearly the totality of
437 alcohols (73.5%). The other classes of VOCs were found at minor concentrations,
438 e.g., aldehydes and esters represented 5.19% and 2.43% of total VOCs,
439 respectively. The aroma profile of oat changed markedly when the oil underwent
440 deep-frying, e.g., acetals and ethanol decreased to 0.62 and 22.7%, respectively,
441 after 20 min. Alcohols lowered to 5.96% at T60. On the contrary, aldehydes and
442 alkanes increased throughout the treatment. As in the other samples, (*E*)-2-heptenal,

443 (*E*)-2-octenal, nonanal, and (*E, E*)-2,4-decadienal were the aldehydes that increased
444 the most. Ketones increased considerably during deep-frying, although the trend was
445 not linear (4.98% in T60). In general, high temperature and long frying time favoured
446 the development of ketones in oat oil. Amongst ketones, 4-octen-3-one was the most
447 abundant, peaking to 1.88% of the total volatiles at T40.

448 Unheated rapeseed oil showed a volatile profile characterised by high concentrations
449 of alcohols (29.8%), with 1-pentanol (3.64%), 1-hexanol (11.3%), and 6-methyl-5-
450 hepten-1-ol (4.96%) being the most abundant compounds. Aldehydes represented
451 16.0% of total VOCs in unheated rapeseed oil. Nevertheless, only propanal (1.00%),
452 hexanal (5.03%), and heptanal (2.65%) were found at relatively high concentrations.
453 Alkanes made up 14.4% of total VOCs, and amongst them, three were unknown
454 branched alkanes. Regarding the other classes of compounds, ketones constituted
455 4.32% of total VOCs with acetone (2.61%) and 6-methyl-5-hepten-1-one (1.72%)
456 being the most abundant compounds. Terpenes constituted 5.36% of total VOCs
457 with α-pinene (2.63%) and α-terpinene (2.36%) as main compounds. It is noteworthy
458 that rapeseed oil was the only to provide sulphur containing compounds (SCCs),
459 such as ethanethiol, 3-butenyl isothiocyanate and dimethyl sulfone that added up to
460 12.4% of total VOCs. SCCs likely develop from the breakdown of glucosinolates
461 found in rapeseed (Barba et al., 2016). SCCs can inhibit the hardening of oils and
462 confer a brassica-like flavour, even when present at minor concentrations (Ivanova-
463 Petropulos et al., 2015). The volatile profile of rapeseed oil changed markedly
464 following short-term deep-frying. Ketones and terpenes faded out after 40 min of
465 treatment. Alcohols dropped to 5.32% at T60. SCCs are thermolabile compounds
466 and declined to 1.86% at T60. On the contrary, aldehydes increased greatly, peaking
467 to 73.2% at T60. The most abundant aldehydes were (*E*)-2-heptenal, (*Z,E*)-2,4-

heptadienal, (*E,E*)-2,4-heptadienal, (*E*)-2-octenal, nonanal, (*E*)-2-decenal, (*Z,E*)-2,4-decadienal, (*E,E*)-2,4-decadienal, and undecen-2-enal. The relative proportions of total alkanes remained stable during deep-frying, nevertheless, the quality of alkanes altered, with aliphatic alkanes outweighing the branched alkanes at the end of the treatment.

The volatile profile of unheated soy oil was characterised by high concentrations of alcohols (31.7%). As shown in Table S3b, twelve alcohols were detected, including both saturated and unsaturated alcohols. Hexanol (13.1%), pentanol (4.07%), and 1-octen-3-ol (3.45%) were the most abundant. Generally, volatile alcohols originate from the oxidative degradation of unsaturated fatty acids (Xia & Budge, 2017). 1-Octen-3-ol first increased to 3.75% in T20, then decreased to 2.44% in T60. 1-Octen-3-ol has a mushroom-like odour (Zhang et al., 2018) and is generally detected in thermally treated oils rich in linoleic acid such as soy oil. Moreover, unheated soy oil was rich in aldehydes (47.4%), with hexanal making up 24.1% of the total VOCs. Alkanes, furans, and ketones were found at relatively low levels (5.22, 1.40, and 5.08%, respectively). After 20 min of deep-frying, alcohols and ketones decreased greatly, being 6.05% and 2.17% of the total VOCs, respectively. On the contrary, aldehydes increased to 62.3%. Aldehydes, such as (*E*)-2-heptenal, (*Z,E*)-2,4-heptadienal, (*E,E*)-2,4-heptadienal, (*E*)-2-octenal, (*E*)-2-decenal, (*Z,E*)-2,4-decadienal, and (*E,E*)-2,4-decadienal were also found by other researchers investigating the volatile profile of fried soy oil (Mildner-Szkudlarz, 2003; Zribi, Jabeur, Flamini, & Bouaziz, 2016). It is important to point out that although total aldehydes raised during the thermal treatment, hexanal dropped from 24.1% (unheated oil) to 11.3% at T60. This decrease in percentage was caused by the rise of the other aldehydes, which generated a redistribution of their relative proportions.

493 The deep-frying process affected also other classes of VOCs, with alkanes
494 increasing to 11.2% at T60, due to pentane rising to 8.01%, and furans peaking to
495 6.51%, due to 2-pentylfuran raising to 6.51% at T60.

496 Amongst the tested oils, the volatile profile of unheated sunflower oil stood out due to
497 the low percentages of alcohols (0.86%), aldehydes (2.42%) and alkanes (0.40%).
498 On the contrary, terpenes accounted for 92.9% of total VOCs. This chemical class
499 was composed of 21 compounds, of which α-pinene (72.7%), sabinene (7.54%), and
500 β-pinene (4.19%) were the most abundant. After 20 min of deep-frying, alcohols
501 increased to 1.32% and continued to increase throughout the treatment, peaking to
502 3.52% after 60 min. The same trend was observed for aldehydes that reached
503 52.5% at T60. The main aldehydes were hexanal, nonanal, (*Z,E*)-2,4-decadienal,
504 and (*E,E*)-2,4-decadienal. Similarly, alkanes and furans increased during deep-frying
505 due to the increases of pentane and 2-pentylfuran, to 13.5% and 3.90% at T60,
506 respectively. Other authors have reported that 2-pentylfuran is a major volatile
507 compound of fried sunflower oil (Doleschall, Recseg, Kemény, & Kővári, 2003). The
508 relative proportion of terpenes dropped to 7.36% of total VOCs at the end of the
509 thermal treatment.

510 PCA was applied to identify patterns amongst the VOC profiles of the selected oils.
511 For this purpose, 33 VOCs were included to perform the chemometric analysis.
512 These compounds were found in at least 40% of the observations. The model
513 yielded a 58.9% of explained variance when considering the two main principal
514 components (PC). From the score plot (Figure 4a), the discrimination between
515 unheated and deep-fried oils in PC1 is clear. On the negative side of PC1 were
516 located all the unheated oils, which are visibly scattered. Unheated lupin and
517 sunflower oils discriminated greatly from the other oils. α-Pinene decreased

progressively during deep-frying, explaining the positioning of unheated sunflower oil on the negative side of PC1, whereas the absence of alkenes accounted for the discrimination of unheated lupin oil and positioned it far-off the centre. As showed in Tables S3a and S3b, the unheated oils had VOC compositions much different from their deep-fried counterparts. On the contrary, once the oils were heated, no evident differences were observed amongst the different time points. For this reason, the deep-fried oils tended to group on the positive side of PC1. Nevertheless, some subgroups could be observed, e.g., deep-fried sunflower, rapeseed and soy oils occupied the lower right-hand quadrant, whereas deep-fried hemp and oat oils occupied the upper right-hand quadrant. Indeed, fried oat and hemp oils had high contents of alkanes, e.g., pentane and octane, which located on the upper positive side of PC1 (loading plot). Figure 4b shows the loading plot of the compounds that contributed most to the separation between the unheated and heated oils. Nearly all the aldehydes, such as (*E*)-2-octenal, nonanal, heptanal, (*E*)-2-heptenal, and butanal were situated on the positive side of the principal components. Aldehydes were major decomposition products of all the frying oils that by increasing over frying, located the fried oils along the positive side of PC1. Aldehydes derive from the β-scission of alkoxy radicals, which originate from the homolytic cleavage of the hydroperoxides (Fujisaki, Endo, & Fujimoto, 2002). The fact that deep-frying produces aldehydes is further corroborated here by the increase in the *p*-AV of the selected oils. The PCA loading plot (Figure S3b) shows a strong correlation between *p*-AV and several volatile aldehydes, e.g., *(E)*-2-hexenal, heptanal, nonanal, and *(E,E)*-2.4-heptadienal (supplementary file no. 3). Besides, clusters between aldehydes and alcohols, e.g., (i) pentanal, hexanal, and 1-penten-3-ol, (ii) 1-octen-e-ol, (*E*)-2-heptenal, and butanal, were displayed on the positive side of PC1,

543 indicating strong correlations between the two groups of compounds. The PCA plot
544 showed no evident correlations between VOCs and PVs, and as frying produced
545 little changes in IV, these two variables remained close to the origin of the plot. The
546 small linear hydrocarbons are other degradation products that were found on the
547 positive side of the loading plot. Heptane and octane derive form β-scission of oleic
548 acid, whereas pentane originate from β-scission of linoleic acid (Schaich, 2015). On
549 the contrary, (*E*,*E*)-2,4-decadienal and (*Z*,*E*)-2,4-decadienal were found on the
550 negative side of PC2, and contributed to the clustering of soy, sunflower, and
551 rapeseed deep-fried oils. It is important to point out that 2,4-decadienal derives from
552 the oxidation of linoleate, and gives a desirable “fried flavour” when present in small
553 amounts. In contrast, high amounts of this aldehyde give a rancid flavour (Frankel,
554 E.N., 1998).

555

556 **4. Study Limitations**

557 Limitation of this research includes: 1) the replenishment with fresh oil after each
558 cycle of frying might not be representative when no replenishment is performed
559 between batches; 2) No internal standard was used for the quantification of VOCs,
560 therefore, data represented the percentages rather than the absolute concentrations
561 of VOCs; 3) the deep-frying of potato affected the composition of the oils in a
562 different way when compared with other foods. These aspects should be taken into
563 consideration when the results of this study are interpreted.

564

565

5. Conclusions

In this study the fatty acid composition, markers of lipid oxidation, and the aroma profile of different vegetable oils were investigated following short-term deep-frying. Hemp oil was the most susceptible to oxidation during deep-frying, showing the greatest reduction in ΣPUFA. Rapeseed, soy, and sunflower oils reduced slightly their content in ΣPUFA and resulted relatively stable to short-term deep-frying. Lupin and oat oils demonstrated high resistance to oxidative degradation, as their fatty acid composition altered to a small extent after 60 min of deep-frying. The highest level of peroxides was observed in soy oil after 60 min. Regarding *p*-AV, the greatest increase was observed for sunflower oil followed by rapeseed oil. Oat oil had the lowest *p*-AV among the oil samples, indicating a low production of secondary non-volatile oxidation products. Several classes of VOCs were observed in the selected oils. In general, unheated hemp oil had the richest volatile profile, with more than 100 VOCs identified. Following deep-frying, the volatile composition of hemp oil changed markedly, and aldehydes developed into the most abundant compounds. Unheated sunflower oil was the richest in terpenes, although most of them were lost upon deep-frying.

In conclusion, oat oil showed the highest thermal stability during short-term deep-frying, as its fatty acid composition and volatile profile changed slightly, and developed the lowest levels of *p*-AV. This work investigated for the first time the effects of a thermal treatment on the volatile profile of hemp, lupin and oat oils.

Contributors

Multari S. and Marsol-Vall A. conceived the work, performed the experiments and wrote the manuscript jointly. Heponiemi P. performed the frying experiment. Suomela J.-P. and Yang B. supervised the analytical work and revised the manuscript. All the authors approved the final version of the manuscript for publication. The authors declare no competing financial interest.

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780

781 **Tables**

782

783 **Table 1a.** Fatty acid composition of hemp, lupin and oat oils.

		hemp				lupin				oat			
	compound	**unheated**	**T20**	**T40**	**T60**	**unheated**	**T20**	**T40**	**T60**	**unheated**	**T20**	**T40**	**T60**
13:0	tridecanoic acid	0.80± 0.07**c**	1.44± 0.12**b**	1.79± 0.12**a,**b	1.92± 0.05**a**	1.09± 0.16**b**	1.68± 0.12**a**	1.77± 0.27**a**	1.79± 0.18**a**	1.04± 0.10**a**	1.45± 0.54**a,b**	1.83± 0.19**b**	1.93± 0.20**b**
14:0	myristic acid	n/d	n/d	n/d	n/d	0.22± 0.01**b**	0.22± 0.01**b**	0.23± 0.01**a,b**	0.24± 0.00**a**	0.16± 0.01**a**	0.17± 0.01**a**	0.17± 0.02**a**	0.17± 0.01**a**
14:1 (n-5)	myristoleic acid methyl ester	0.15± 0.03**c,b**	0.18± 0.03**b,a**	0.20± 0.03**a**	0.20± 0.01**a**	0.20± 0.01**a**	0.19± 0.02**a**	0.20± 0.02**a**	0.20± 0.01**a**	0.18± 0.01**a**	0.18± 0.04**a**	0.20± 0.01**a**	0.21± 0.01**a**
15:0	pentadecanoic acid	0.14± 0.01**a**	0.14± 0.00**a**	0.14± 0.00**a**	0.13± 0.00**a**	0.16± 0.01**a**	0.16± 0.01**a**	0.15± 0.00**a**	0.16± 0.00**a**	0.15± 0.00**a**	0.14± 0.00**b**	0.13± 0.00**b**	0.14± 0.01**b**
16:0	palmitic acid	5.93± 0.02**a**	5.90± 0.02**a**	5.92± 0.02**a**	5.91± 0.01**a**	11.1± 0.03**a**	11.1± 0.04**a**	11.1± 0.01**a**	11.2± 0.01**a**	14.9± 0.02**a**	14.9± 0.04**a**	14.9± 0.03**a**	14.9± 0.02**a**
16:1	palmitoleic acid	0.11± 0.00**a**	0.10± 0.00**a**	0.11± 0.00**a**	0.10± 0.00**a**	n/d	n/d	n/d	n/d	0.17± 0.01**a**	0.17± 0.00**a**	0.18± 0.00**a**	0.18± 0.00**a**
17:1	cis-10-heptadecenoic acid	0.15± 0.01**a**	0.14± 0.00**a**	0.14± 0.00**a**	0.14± 0.01**a**	0.16± 0.00**a**	0.15± 0.00**b**	0.15± 0.00**b**	0.14± 0.00**b**	0.16± 0.01**a**	0.14± 0.00**b**	0.14± 0.00**b**	0.14± 0.01**b**
18:0	stearic acid	2.93± 0.01**a**	2.89± 0.01**b**	2.90± 0.01**b**	2.88± 0.01**b**	5.99± 0.03**a**	5.98± 0.02**a**	5.98± 0.02**a**	5.98± 0.01**a**	1.96± 0.01**a**	1.91± 0.01**b**	1.91± 0.01**b**	1.91± 0.00**b**
18:1 (n-9)c	oleic acid	10.7± 0.01**a**	10.6± 0.02**b**	10.6± 0.02**b**	10.6± 0.01**b**	31.3± 0.15**a**	31.3± 0.06**a**	31.3± 0.09**a**	31.4± 0.05**a**	40.3± 0.06**a**	40.4± 0.26**a**	40.3± 0.09**a**	40.3± 0.10**a**
18:1 (n-7)	vaccenic acid	0.67± 0.00**a**	0.67± 0.00**a**	0.67± 0.00**a**	0.67± 0.00**a**	0.62± 0.01**a**	0.61± 0.00**a**	0.61± 0.00**a**	0.61± 0.01**a**	0.63± 0.00**a**	0.63± 0.01**a**	0.62± 0.00**a**	0.63± 0.00**a**

		hemp				lupin				oat			
	compound	**unheated**	**T20**	**T40**	**T60**	**unheated**	**T20**	**T40**	**T60**	**unheated**	**T20**	**T40**	**T60**
18:2 (n-6)c	linoleic acid	54.0± 0.04**a**	53.6± 0.09**b**	53.4± 0.09**b**	53.3± 0.07**b**	38.5± 0.17**a**	38.3± 0.06**a,b**	38.2± 0.11**b**	38.3± 0.05**a,**b	36.5± 0.04**a**	36.5± 0.24**a**	36.4± 0.09**a**	36.4± 0.09**a**
18:3 (n-6)	γ-linolenic acid	2.82± 0.00**a**	2.80± 0.01**b**	2.78± 0.01**b**	2.77± 0.00**c**	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d
18:3 (n-3)	α-linolenic acid	18.1± 0.02**a**	17.9± 0.02**b**	17.8± 0.02**c**	17.8± 0.02**c**	3.77± 0.02**a**	3.73± 0.01**b**	3.71± 0.01**b**	3.71± 0.01**b**	1.24± 0.00**a**	1.24± 0.01**a**	1.24± 0.00**a**	1.24± 0.02**a**
19:0	nonadecanoic acid	1.09± 0.01**a**	1.07± 0.00**a**	1.06± 0.00**b**	1.06± 0.00**b**	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d
20:0	eicosanoic acid	0.75± 0.00a	0.75± 0.00a	0.75± 0.00a	0.75± 0.00a	1.20± 0.28**a**	0.79± 0.03**b**	0.75± 0.01**b**	0.75± 0.00**b**	0.12± 0.00**a**	0.12± 0.00**a**	0.12± 0.00**a**	0.13± 0.00**b**
20:1 (n-9)	gondoic acid	n/d	n/d	n/d	n/d	0.23± 0.01**a**	0.23± 0.00**a**	0.23± 0.01**a**	0.23± 0.00**a**	0.71± 0.00**a**	0.73± 0.01**b**	0.73± 0.00**b**	0.73± 0.01**b**
22:0	docosanoic acid	0.27± 0.01**a**	0.27± 0.01**a**	0.28± 0.01**a**	0.27± 0.00**a**	1.80± 0.01**a**	1.81± 0.01**a**	1.81± 0.01**a**	1.80± 0.01**a**	n/d	n/d	n/d	n/d
22:1 (n-9)	erucic acid	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d
22:6 (n-3)	docosa-hexaenoic acid	n/d	n/d	n/d	n/d	0.11± 0.01**a**	0.11± 0.01**a**	0.10± 0.01**a**	0.10± 0.01**a**	n/d	n/d	n/d	n/d
22:3 (n-3)	docosa-trienoic acid	n/d	n/d	n/d	n/d	1.15± 0.01**a**	1.15± 0.01**a**	1.16± 0.00**a**	1.16± 0.01**a**	0.12± 0.01**a**	0.11± 0.01**b**	n/d	n/d
23:0	tricosanoic acid	n/d	n/d	n/d	n/d	0.76± 0.03**a**	0.79± 0.02**a**	0.78± 0.01**a**	0.76± 0.02**a**	0.41± 0.01**a**	0.27± 0.00**b**	0.17± 0.01**c**	0.10± 0.01**d**
24:0	tetracosanoic acid	n/d	n/d	n/d	n/d	0.30± 0.01**a**	0.32± 0.01**a**	0.33± 0.01**a**	0.32± 0.01**a**	n/d	n/d	n/d	n/d
others		0.99± 0.01**b,c**	0.96± 0.02**c**	0.96± 0.02**c**	1.04± 0.05**a,b**	1.48± 0.03**a**	1.38± 0.01**b**	1.38± 0.02**b**	1.23± 0.02**c**	1.21± 0.02**a**	0.93± 0.03**b**	0.97± 0.03**b**	0.96± 0.03**b**

	hemp				lupin				oat			
compound	**unheated**	**T20**	**T40**	**T60**	**unheated**	**T20**	**T40**	**T60**	**unheated**	**T20**	**T40**	**T60**
ΣSFA	11.9± 0.06**c**	12.5± 0.12**b**	12.8± 0.12**b,a**	12.9± 0.03**a**	22.6± 0.37**a**	22.9± 0.13**a**	22.9± 0.22**a**	23.0± 0.14**a**	18.7± 0.11**a**	18.9± 0.49**a**	19.2± 0.17**a**	19.3± 0.19**a**
ΣMUFA	11.7± 0.03a	11.7± 0.03a	11.7± 0.03a	11.7± 0.03a	32.4± 0.16**a**	32.5± 0.05**a**	32.5± 0.08**a**	32.5± 0.05**a**	42.2± 0.07**a**	42.3± 0.25**a**	42.2± 0.10**a**	42.2± 0.08**a**
ΣPUFA	75.0± 0.06**a**	74.4± 0.12**b**	74.1± 0.12**b,c**	73.9± 0.09**c**	43.5± 0.19**a**	43.3± 0.08**a,b**	43.2± 0.13**b**	43.3± 0.06**a,b**	37.9± 0.04**a**	37.8± 0.25**a**	37.6± 0.09**a**	37.6± 0.09**a**
iodine value	150± 0.12**a**	149± 0.24**b**	148± 0.59**b**	148± 0.18**b**	105± 0.29**a**	105± 0.17**a**	104± 0.31**b**	104± 0.05**b**	101± 0.12**a**	101± 0.65**a**	101± 0.22**a**	101± 0.25**a**

784 Data (relative %) are presented as mean ± SD and represent mean of four independent replicates. n/d = not detected. Values with

785 unlike letters (a-c) within the same row for a given oil differ significantly ($p < 0.05$).

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787 **Table 1b**. Fatty acid composition of rapeseed, soy and sunflower oils.

		rapeseed				soy				sunflower			
	compound	**unheated**	**T20**	**T40**	**T60**	**unheated**	**T20**	**T40**	**T60**	**unheated**	**T20**	**T40**	**T60**
13:0	tridecanoic acid	1.68± 0.07**a**	1.76± 0.11**a**	1.88± 0.30**a**	1.78± 0.10**a**	1.80± 0.20**a**	1.76± 0.56**a**	0.99± 0.11**b**	0.88± 0.05**b**	1.49± 0.19**c**	1.86± 0.13**b**	2.15± 0.03**a**	1.87± 0.05**b**
14:0	myristic acid	n/d	n/d	n/d	n/d	0.10± 0.00**a**	0.09± 0.00**a**	0.09± 0.00**a**	0.09± 0.00**a**	0.10± 0.00**a**	0.10± 0.02**a**	0.10± 0.01**a**	0.10± 0.01**a**
14:1 (n-5)	myristoleic acid methyl ester	0.12± 0.01**a**	0.12± 0.02**a**	0.12± 0.02**a**	0.12± 0.02**a**	0.11± 0.05**a**	0.10± 0.03**a**	0.11± 0.02**a**	0.13± 0.02**a**	0.08± 0.02**b**	0.12± 0.02**a**	0.12± 0.01**a**	0.11± 0.01**a,b**
15:0	pentadecanoic acid	0.13± 0.00**a**	0.14± 0.00**a**	0.14± 0.01**a**	0.14± 0.01**a**	0.13± 0.01**a**	0.14± 0.01**a**	0.13± 0.00**a**	0.14± 0.00**a**	0.13± 0.01**b**	0.16± 0.01**a**	0.16± 0.02**a**	0.16± 0.01**a**

	compound	rapeseed				soy				sunflower			
		unheated	T20	T40	T60	unheated	T20	T40	T60	unheated	T20	T40	T60
16:0	palmitic acid	4.74± 0.01**b**	4.80± 0.00**a**	4.80± 0.01**a**	4.84± 0.05**a**	10.1± 0.08**b**	10.2± 0.02**a**	10.3± 0.04**a**	10.3± 0.08**a**	6.44± 0.03**c**	6.47± 0.02**b,c**	6.49± 0.02**a,b**	6.50± 0.02**a,b**
16:1	palmitoleic acid	0.22± 0.00**a**	0.22± 0.00**a**	0.22± 0.00a	0.23± 0.01**a**	0.07± 0.00**b**	0.09± 0.01**a**	0.09± 0.01**a**	0.10± 0.00**a**	0.13± 0.00**a**	0.13± 0.01**a**	0.13± 0.01**a**	0.13± 0.00**a**
17:1	cis-10-heptadecenoic acid	n/d	n/d	n/d	n/d	0.14± 0.00**a**	0.14± 0.00**a**	0.13± 0.00**a**	0.14± 0.01**a**	0.13± 0.01**b,c**	0.14± 0.01**a,b**	0.14± 0.01**a,b**	0.12± 0.01**c**
18:0	stearic acid	1.77± 0.00**b**	1.79± 0.00**a,b**	1.79± 0.01**a**,**b**	1.83± 0.05**a**	3.83± 0.12**b**	4.17± 0.03**a**	4.18± 0.01**a**	4.18± 0.00**a**	3.36± 0.03**a**	3.37± 0.01**a**	3.38± 0.01**a**	3.38± 0.00**a**
18:1 (n-9)c	oleic acid	57.7± 0.10**a**	57.9± 0.06**a**	57.9± 0.15**a**	57.9± 0.19**a**	18.8± 0.13**b**	20.9± 0.16**a**	20.9± 0.03**a**	20.8± 0.01**a**	26.1± 0.06**a**	26.0± 0.07**a**	26.0± 0.02**a**	26.0± 0.01**a**
18:1 (n-7)	vaccenic acid	2.76± 0.01**b**	2.81± 0.01**a**	2.81± 0.02**a**	2.80± 0.01**a**	1.12± 0.01**b**	1.14± 0.01**a**	1.15± 0.01**a**	1.15± 0.00**a**	0.63± 0.00**a**	0.64± 0.00**a**	0.63± 0.00**a**	0.64± 0.00**a**
18:2 (n-6)c	linoleic acid	19.0± 0.03**a**	18.9± 0.02**b**	18.9± 0.05**b**	18.8± 0.06**b**	52.9± 0.36**a**	50.9± 0.35**c**	51.5± 0.07**b**	51.6± 0.01**b**	58.5± 0.10**a**	58.1± 0.13**b**	58.0± 0.04**b**	58.1± 0.12**b**
18:3 (n-6)	γ-linolenic acid	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d
18:3 (n-3)	α-linolenic acid	8.40± 0.01**a**	8.12± 0.01**b**	8.09± 0.02**b**	8.09± 0.03**b**	8.89± 0.06**a**	8.00± 0.05**c**	8.13± 0.01**b**	8.19± 0.00**b**	0.12± 0.01**a**	0.12± 0.02**a**	0.12± 0.02**a**	0.09± 0.01**b**
20:0	eicosanoic acid	0.51± 0.01**b**	0.52± 0.00**a,b**	0.52± 0.01**a,b**	0.53± 0.00**a**	0.31± 0.00**b**	0.32± 0.01**a,b**	0.32± 0.00**a,b**	0.33± 0.00**a**	0.62± 0.09**a**	0.22± 0.01**b**	0.21± 0.01**b**	0.22± 0.00**b**
20:1 (n-9)	gondoic acid	1.18± 0.08**a**	1.18± 0.08**a**	1.18± 0.08**a**	1.18± 0.05**a**	0.15± 0.00**b**	0.16± 0.01**a**	0.17± 0.00**a**	0.17± 0.01**a**	0.13± 0.01**a**	0.12± 0.00**a**	0.12± 0.00**a**	0.12± 0.01**a**
22:0	docosanoic acid	0.28± 0.01**a**	0.28± 0.00**a**	0.27± 0.00**a**	0.28± 0.01**a**	0.34± 0.01**a**	0.32± 0.02**a**	0.32± 0.01**a**	0.32± 0.01**a**	0.59± 0.01**a**	0.59± 0.01**a**	0.60± 0.01**b**	0.60± 0.00**b**
22:1 (n-9)	erucic acid	0.05± 0.00**b**	0.07± 0.00**a**	0.07± 0.01**a**	0.08± 0.00**a**	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d
22:3 (n-3)	docosa-trienoic acid	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	0.14± 0.01**a**	0.14± 0.00**a**	0.14± 0.01**a**	0.14± 0.01**a**

	compound	rapeseed				soy				sunflower			
		unheated	**T20**	**T40**	**T60**	**unheated**	**T20**	**T40**	**T60**	**unheated**	**T20**	**T40**	**T60**
24:0	tetracosanoic acid	n/d	n/d	n/d	n/d	0.09± 0.01**a**	0.08± 0.00**a**	0.10± 0.01**a**	0.09± 0.01**a**	0.20± 0.01**a**	0.19± 0.01**a**	0.19± 0.01**a**	0.20± 0.00**a**
others		1.38± 0.07**a**	1.33± 0.03**a**	1.40± 0.01**a**	1.42± 0.14**a**	1.22± 0.08**b**	1.46± 0.12**a**	1.41± 0.04**a**	1.40± 0.09**a,b**	1.13± 0.04c	1.44± 0.12a	1.35± 0.03a,b	1.23± 0.05b,c
ΣSFA		9.11± 0.06**b**	9.30± 0.11**a**	9.40± 0.27**a**	9.38± 0.16**a**	16.7± 0.43**a**	16.8± 0.50**a**	16.4± 0.13**a**	16.4± 0.09**a**	12.9± 0.14**b**	13.0± 0.14**b**	13.3± 0.04**a**	13.2± 0.05**a**
ΣMUFA		62.1± 0.09**a**	62.3± 0.09**a**	62.3± 0.21**a**	62.2± 0.19**a**	20.4± 0.08**b**	22.6± 0.14**a**	22.6± 0.05**a**	22.4± 0.03**a**	27.2± 0.04**a**	27.2± 0.07**a**	27.1± 0.03**a**	27.0± 0.08**a**
ΣPUFA		27.4± 0.04**a**	27.1± 0.03**b**	27.0± 0.07**b**	26.9± 0.09**b**	61.8± 0.42**a**	58.9± 0.40**c**	59.6± 0.07**b**	59.8± 0.01**b**	58.8± 0.11**a**	58.4± 0.14**b**	58.2± 0.03**b**	58.3± 0.13**b**
iodine value		105± 0.16**a**	105± 0.11**a**	104± 0.25**b**	104± 0.36**b**	128± 0.62**a**	123± 0.78**c**	124± 0.11**b**	125± 0.05**b**	112± 0.24**a**	111± 0.25**b**	110± 0.04**c**	110± 0.27**c**

788 Data (relative %) are presented as mean ± SD and represent mean of four independent replicates. n/d = not detected. Values with

789 unlike letters (a-c) within the same row for a given oil differ significantly ($p < 0.05$).

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Figures

Figure 1. Experimental design of the deep-frying experiment.

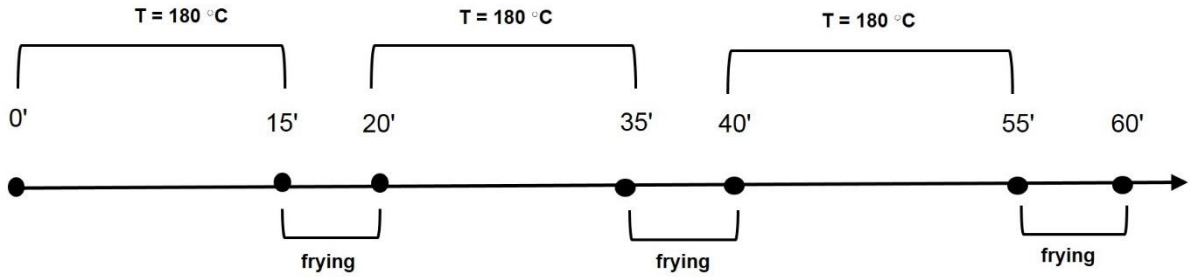
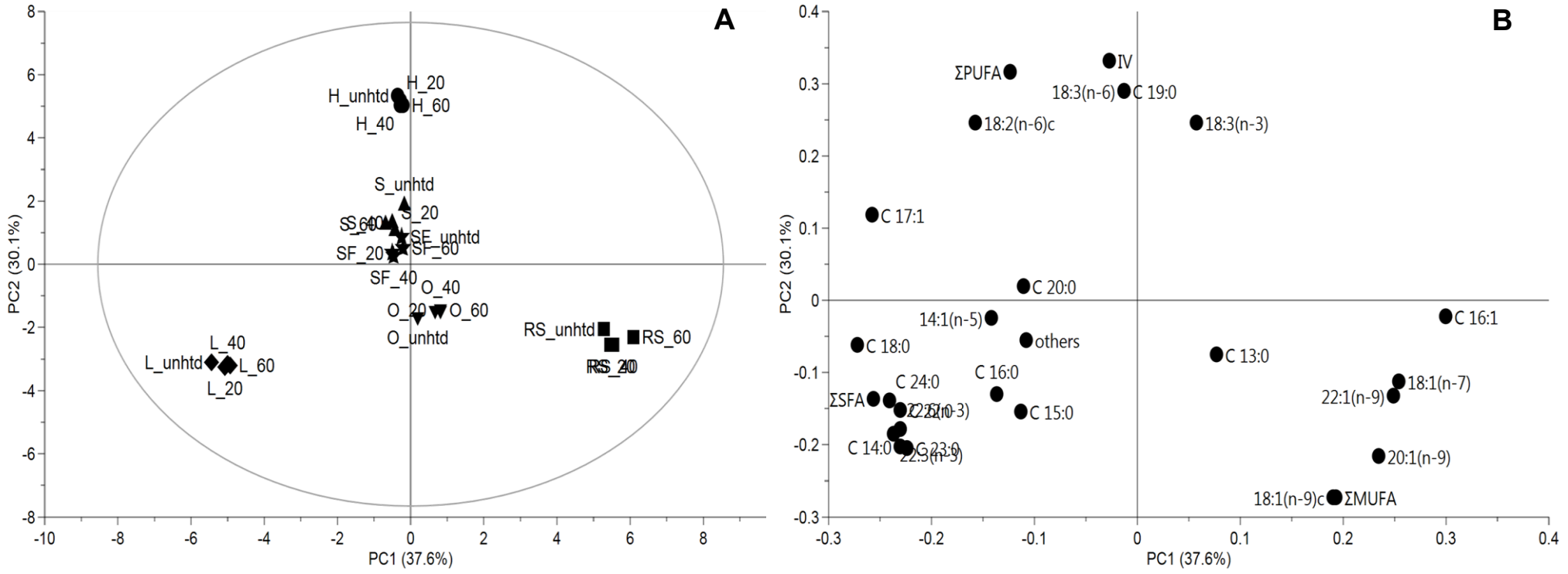
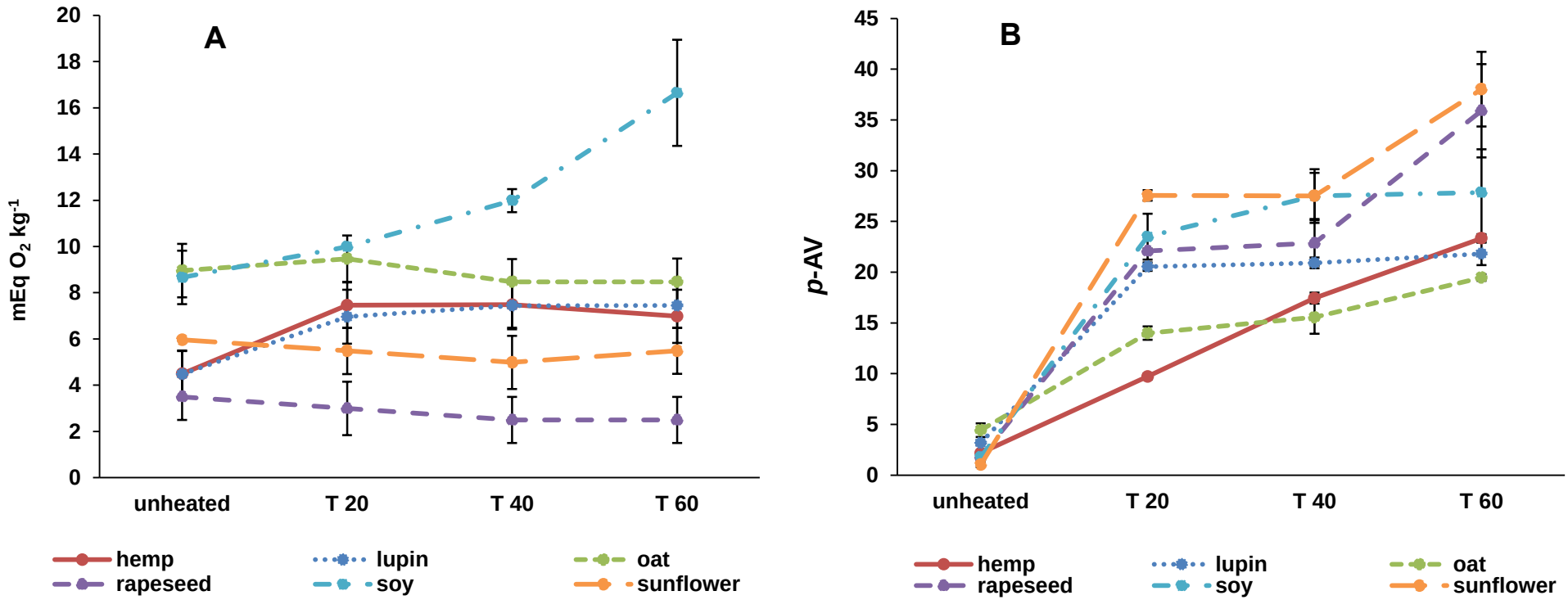


Figure 2. PCA of edible oils for fatty acids and iodine values in function of PC1 and PC2. (A) Scores plot (n = 27) for hemp (H, circle), lupin (L, diamond), oat (O, inverted triangle), rapeseed (R, square), soy (S, triangle) and sunflower (SF, 5-point star). Unheated, 20, 40, and 60 represent unheated oil at 20, 40, and 60 min of deep-frying, respectively. (B) Loadings plot (n = 26). Compounds coded according to Table 1a and 1b.



806 **Figure 3**. Peroxide (A) and *p*-anisidine (B) values of the edible oils.



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808 Data are presented as mean ± SD and represents mean of four independent measurements.

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Figure 4. PCA of edible oils for volatile compounds in function of PC1 and PC2. (A) Scores plot (n = 24) for hemp (H, circle), lupin (L, diamond), oat (O, inverted triangle), rapeseed (R, square), soy (S, triangle) and sunflower (SF, 5-point star). Unhtd, 20, 40, and 60 represent unheated oil at 20, 40, and 60 min of deep-frying, respectively. (B) Loadings plot (n = 33). Compounds numbered according to Table S3a and S3b.