JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY



# Effect of the Type of Frying Culinary Fat on Volatile Compounds Isolated in Fried Pork Loin Chops by Using SPME-GC-MS

MARÍA ROSARIO RAMÍREZ,* MARIO ESTÉVEZ, DAVID MORCUENDE, AND RAMÓN CAVA

Tecnología de los Alimentos, Facultad de Veterinaria, Universidad de Extremadura, Cáceres 10071, Spain

The effect of the type of frying culinary fat (olive oil, sunflower oil, butter, and pig lard) on volatile compounds isolated from fried pork loin chops (m. *Longissimus dorsi*) was measured by SPME-GC-MS. Frying modified the fatty acid composition of lipids from pork loin chops, which tended to be similar to that of the culinary fat used to fry. Volatile compounds formed from the oxidation of fatty acids increased, such as aldehydes, ketones, alcohols, and hydrocarbons. Besides, each culinary fat used modified the volatile profiles in fried meat differently. Sunflower oil-fried pork loin chops presented the highest aldehyde aliphatic content, probably due to their highest content of polyunsaturated acids. Hexanal, the most abundant aldehyde in fried samples, presented the most elevated content in sunflower oil-fried pork loin chops. In addition, these samples presented more heterocyclic compounds from the Maillard reaction than other fried samples. Volatiles detected in olive oil-fried pork loin chops were mainly lipid-derived compounds such as pentan-1-ol, hexanal, hept-2-enal, nonanal, decanal, benzaldehyde, and nonan-2-one. Butter-fried pork loins were abundant in ketones with a high number of carbons (heptan-2-one, nonan-2-one, undecan-2-one, tridecanone, and heptadecan-2-one). Pig lard-fried pork loin chops presented some Strecker aldehydes isolated in only these samples, such as 2-methylbutanal and 3-(methylthio)propanal, and a sulfur compound (dimethyl disulfide) related to Strecker aldehydes.

**KEYWORDS: Culinary oil and fat; deep-frying; meat; volatile compounds**

## INTRODUCTION

Food frying is one of the oldest known culinary practices, often utilized in the food industry due to the significant sales of these products and vast quantity. From the consumers' standpoint, fried food palatability is related to unique organoleptic and sensory characteristics, including flavor, texture, and appearance (*1*).

Frying produces significant modifications in food, such as the loss of constitutional water, changes in the fatty acid profile due to the exchange between frying media and fat from food (*2*, *3*), and the development of heat-induced chemical reactions (*4*, *5*). Moreover, during frying, minor compounds from culinary fats, which could influence volatile generation, such as tocopherols, phytosterols, and carotenes, are incorporated in the food (*6*).

The generation of volatile compounds in meat and meat products has been largely studied because of the role of flavor in the overall acceptability of them, because, after appearance and tenderness, flavor is the most important characteristic perceived by consumers. The development of aroma and flavor in cooked meat is a complex process in which different compounds react to produce intermediary compounds or volatiles responsible for the characteristic flavor. The flavor of cooked meat derives mainly from thermally induced reactions, principally lipid oxidation reactions and the Maillard reaction (*7*).

Lipids are probably the most important precursor volatiles of meat due to lipid degradation, which provides several compounds responsible for the aroma of cooked meat. Many works have dealt with the role of the fatty acid profile in the overall meat flavor (*8*, *9*) because lipid degradation is the most important factor that contributes to the volatile formation. Variations in the fatty acid profile of meat by frying (*2*, *3*) modify the substrate where thermo-oxidation reactions would take place. Therefore, these modifications of the oxidative susceptibility of fried meat and the possible influence of some of those aforementioned minor compounds from vegetable oils with antioxidant activity could affect the formation of volatiles and modify the aromatic profile detected in fried meat using different culinary fats.

On the other hand, the Maillard reaction, which occurs between amino compounds and reducing sugars, is one of the most important routes to flavor compounds in cooked foods,

* Author to whom correspondence should be addressed (telephone +34 927 257 169; fax +34 927 257 110; e-mail rramirez@unex.es).

10.1021/jf049207s CCC: $27.50 
Published on Web 11/18/2004

including meat, because it provides a large number of compounds that contribute to the flavor of cooked meat (*10*). Thus, heterocyclic compounds, especially those containing sulfur, are responsible for "savory", "meat", "roast", and "boiled" flavors (*7*). Besides, compounds from the Maillard reaction can also react with other components of meat such as aldehydes and other carbonyls formed during lipid oxidation, which have been shown to react readily with Maillard intermediates. Such interactions contribute to the achievement of the optimum and characteristic flavor of cooked meat (*7*).

The objective of this work is evaluate the effect of different types of culinary fats (olive oil, sunflower oil, butter, and pig lard) on the volatile formation of fried pork loin chops by using the technique of solid-phase microextraction (SPME) sampling combined with gas chromatography−mass spectrometry (GC-MS).

## MATERIALS AND METHODS

**Materials.** Olive oil with an acidity value of 0.4 (percent oleic acid), refined sunflower oil with an acidity value of 0.2 (percent oleic acid), butter, and pig lard were used as culinary fats. Pork loins (m. *Longissimus dorsi*) from Large-White × Duroc pigs were sliced using a slicing machine, obtaining 15 mm thickness chops.

**Methods.** *Frying.* Five pork loin chops (∼80 g/chop) were fried in each culinary fat (olive oil, sunflower oil, butter, and pig lard). Frying was performed at 160 °C in domestic stainless steel−Teflon-coated fryers with 2 L capacities. The food/oil ratio was 20 g/500 mL. The time of frying was 2 min, and the samples were turned after 1 min of frying. The temperature of the culinary fat during frying was checked using a thermocouple. Once fried, samples were drained for ∼2 min and dried on a paper towel to eliminate oil remaining on the loin slice surface. Samples were frozen and kept at −70 °C until analyses were made.

*Fat Extraction.* Lipids were extracted from 2 g of meat samples with chloroform/methanol (1:2), according to the method described by Bligh and Dyer (*12*).

*Lipid Extract Fractionation.* Total lipid extracts were fractionated into neutral lipids (NL), free fatty acids (FFA), and polar lipids (PL) on aminopropyl cartridges following the procedure described by Monin et al. (*13*). The lipid extract was fractionated by passing 30−60 mg of lipid dissolved in 4 mL of hexane through an aminopropyl column. NL were eluted with 4 mL of $CHCl_3$/2-propanol (2:1), FFA with 4 mL of diethyl ether/acetic acid 2%, and PL with 4 mL of MeOH/HCl (9:1).

*Fatty Acid Profile Determination.* Fatty acid methyl esters (FAMEs) were prepared by transesterification using methanol in the presence of sulfuric acid (5% of sulfuric acid in methanol) following the method of Cava et al. (*14*). FAMEs were analyzed using a Hewlett-Packard, model HP-5890A, gas chromatograph, equipped with a flame ionization detector (FID). The derivatives were separated on a semicapillary column (Hewlett-Packard FFAP-TPA fused-silica column, 30 m length, 0.53 mm i.d., and 1.0 $\mu$m film thickness). The injector and detector temperatures were held at 230 °C. The column oven temperature was maintained at 220 °C. The flow rate of the carrier gas ($N_2$) was set at 1.8 mL/min. Identification of FAMEs was based on retention times of reference compounds (Sigma). Fatty acid composition was expressed as percent of total FAMEs.

*Analysis of Volatiles in Raw and Fried Meat Samples.* In the development of the analysis a SPME fiber (Supelco Co. Canada) coated with divinylbenezene/carboxen/poly(dimethylxilosane) (DVB/CAR/PDMS) 50/30 $\mu$m was used. The sampling technique to extract volatile compounds from the headspace was the following: 0.5 g of meat was homogenized with 2 mL of distilled water during 1 min. Then 0.5 g of sodium chloride was added, and the mix was placed in 5 mL vials with a silicone stopper. The fiber was exposed to the headspace of the sample during 30 min of immersion in water at 50 °C. The analyses were performed on an HP5890GC series II gas chromatograph (Hewlett-Packard) coupled to a mass selective detector, Agilent model 5973. Volatiles were separated using a 5% phenyl−95% dimethylpolysiloxane column (30 m × 0.25 mm i.d., 1.0 $\mu$m film thickness; Restek). The carrier gas was helium at 18.5 psi, resulting in a flow of 1.6 mL/min at 40 °C. The SPME fiber was desorbed and maintained in the injection port at 220 °C during the first 30 min of the run. The injection port was in the splitless mode, and the temperature program was isothermal for 10 min at 40 °C and then raised at the rate of 7 °C $min^{-1}$ to 250 °C and held for 5 min. The GC-MS transfer line temperature was 270 °C. The MS operated in the electron impact mode with an electron impact energy of 70 eV and a multiplier voltage of 1650 V and collected data at a rate of 1 scan $s^{-1}$ over a range of *m*/*z* 40−300. *n*-Alkanes (Sigma R-8769) were co-injected with the samples to calculate the retention indices (RI) for volatiles. The compounds were identified by comparison with reference compounds (Sigma, Aldrich), by comparison of RI with those described by Kondjoyan and Berdagué (*15*), and by comparison of their mass spectra with those contained in Wiley and NIST libraries.

*Data Analysis.* For the analysis of the effect of the type of culinary fat (olive oil, sunflower oil, butter, and pig lard) on the fatty acid profile and volatile compounds detected, an analysis of variance (ANOVA) was used. HSD Tukey's tests were used when the ANOVA detected significant differences ($p < 0.05$) between treatments.

**Table 1.** Fatty Acid Composition (Percent) of the Different Types of Culinary Fats Used To Fry Pork Loin Chops

| | culinary fats[a] | | | |
|---|---|---|---|---|
| | OO | SO | BT | PLD |
| C12:0 | 0.0 | 0.0 | 3.7 | 0.0 |
| C14:0 | 0.0 | 0.1 | 13.4 | 1.2 |
| C16:0 | 10.1 | 6.3 | 39.5 | 23.0 |
| C17:0 | 0.0 | 0.0 | 0.7 | 0.5 |
| C18:0 | 3.3 | 3.8 | 11.0 | 12.6 |
| C20:0 | 0.4 | 0.3 | 0.0 | 0.2 |
| SFA[b] | 13.8 | 10.5 | 68.2 | 37.5 |
| C16:1 | 0.7 | 0.2 | 3.5 | 2.6 |
| C17:1 | 0.1 | 0.0 | 0.4 | 0.4 |
| C18:1 | 78.7 | 30.5 | 27.4 | 42.1 |
| C20:1 | 0.3 | 0.2 | 0.2 | 1.0 |
| MUFA[c] | 79.8 | 30.9 | 31.5 | 46.1 |
| C18:2 | 5.6 | 58.6 | 3.7 | 14.6 |
| C18:3 | 0.6 | 0.1 | 0.2 | 1.0 |
| C20:2 | 0.1 | 0.0 | 0.0 | 0.6 |
| C20:4 | 0.0 | 0.0 | 0.1 | 0.0 |
| PUFA[d] | 6.3 | 58.7 | 3.9 | 16.2 |

[a] OO, olive oil; SO, sunflower oil; BT, butter; PLD, pig lard. [b] SFA, saturated fatty acids (C12:0, C14:0, C16:0, C17:0, C18:0, C20:0). [c] MUFA, monounsaturated fatty acids (C16:1, C17:1, C18:1, C20:1). [d] PUFA, polyunsaturated fatty acids (C18:2, C18:3, C20:2, C20:4).

## RESULTS AND DISCUSSION

**Analysis of the Fatty Acid Composition in Raw and Fried Meat. Table 1** shows the major fatty acids of the olive oil, sunflower oil, butter, and pig lard used. Olive oil was rich in monounsaturated fatty acids (MUFA), mainly oleic acid (C18:1*n*−9, 78.7%), whereas sunflower oil was characterized by a high content of polyunsaturated fatty acids (PUFA), in which linoleic acid (C18:2*n*−6) was the majority (58.6%). Butter was very rich in saturated fatty acids (SFA), lauric acid (C12:0), myristic acid (C14:0), and palmitic acid (C16:0) (3.7, 13.4, and 39.5%, respectively). Pig lard showed moderate levels of SFA (37.5%), mainly myristic acid, palmitic acid, and stearic acid (C18:0), and a relatively large proportion of MUFA, principally oleic acid (42.1%).

**Table 2** shows the fatty acid composition of neutral lipids, free fatty acids, and polar lipids of intramuscular fat from raw and fried pork loin chops. Frying modified the composition of meat lipids, which tended to be similar to that of the culinary frying fat used (*2*, *3*). Findings indicate that changes in the fatty acid composition of fried samples were due to the exchange

**Table 2.** Fatty Acid Composition (Percent) of Intramuscular Lipid Fractions in Raw and Fried Meat in Different Culinary Fats

| | | fried loins | | | | | ANOVA |
|---|---|---|---|---|---|---|---|
| | raw | OO | SO | BT | PLD | SEM | *p* value |
| neutral lipids | | | | | | | |
| C16:0 | 26.5b | 19.3c | 17.0c | 28.9a | 25.3b | 0.96 | 0.000 |
| C18:0 | 15.2a | 8.6c | 8.9c | 12.6b | 11.8b | 0.53 | 0.000 |
| SFA[a] | 43.8a | 29.2c | 27.1c | 46.8a | 39.4b | 1.65 | 0.000 |
| C16:1 | 2.8b | 2.5b | 2.0c | 3.6a | 3.5a | 0.13 | 0.000 |
| C18:1 | 42.4b | 59.1a | 37.5c | 41.2b | 43.8b | 1.55 | 0.000 |
| MUFA[b] | 46.7b | 62.5a | 40.4c | 45.9b | 48.6b | 1.53 | 0.000 |
| C18:2 | 8.2bc | 6.9bc | 31.3a | 5.0c | 10.2b | 2.03 | 0.000 |
| C18:3 | 0.5b | 0.5b | 0.3c | 0.4c | 0.6a | 0.03 | 0.000 |
| PUFA[c] | 9.4bc | 8.2bc | 32.5a | 6.2c | 11.8b | 2.02 | 0.000 |
| free fatty acids | | | | | | | |
| C16:0 | 20.4 | 16.2 | 17.9 | 22.6 | 20.1 | 0.82 | 0.111 |
| C18:0 | 13.8 | 13.0 | 11.1 | 13.1 | 12.6 | 0.33 | 0.098 |
| SFA | 36.2ab | 33.0b | 32.2b | 40.9a | 36.9ab | 0.95 | 0.015 |
| C16:1 | 2.8 | 1.9 | 1.8 | 2.5 | 2.8 | 0.16 | 0.092 |
| C18:1 | 39.6a | 39.5a | 30.2b | 31.0b | 32.1b | 1.09 | 0.000 |
| MUFA[b] | 43.8a | 42.2ab | 34.1c | 35.1c | 37.1bc | 1.00 | 0.000 |
| C18:2 | 14.8b | 17.2b | 25.3a | 15.2b | 17.6b | 0.87 | 0.000 |
| C18:3 | 1.0c | 1.3ab | 1.9ab | 2.3a | 2.2a | 0.15 | 0.009 |
| PUFA[c] | 19.1c | 24.8b | 33.7a | 24.0bc | 26.0b | 1.11 | 0.000 |
| polar lipids | | | | | | | |
| C16:0 | 12.5a | 11.0ab | 9.5b | 11.9ab | 12.2ab | 0.36 | 0.046 |
| C18:0 | 17.4b | 19.7a | 19.0ab | 18.1ab | 17.5b | 0.27 | 0.011 |
| SFA[a] | 30.3 | 30.9 | 28.8 | 30.3 | 29.8 | 0.27 | 0.141 |
| C16:1 | 0.9a | 0.6b | 0.5b | 0.6ab | 0.6b | 0.04 | 0.002 |
| C18:1 | 17.1 | 14.1 | 14.9 | 15.7 | 15.2 | 0.48 | 0.388 |
| MUFA[b] | 18.6 | 15.1 | 16.3 | 17.0 | 16.4 | 0.04 | 0.094 |
| C18:2 | 28.5 | 29.7 | 29.2 | 29.7 | 30.7 | 0.50 | 0.294 |
| C18:3 | 0.9 | 0.9 | 1.4 | 0.8 | 0.7 | 0.31 | 0.256 |
| PUFA[c] | 50.8 | 53.7 | 54.7 | 52.5 | 53.5 | 0.60 | 0.303 |

[a] SFA, saturated fatty acids (C12:0, C14:0, C16:0, C17:0, C18:0, C20:0). [b] MUFA, monounsaturated fatty acids (C16:1, C17:1, C18:1, C20:1). [c] PUFA, polyunsaturated fatty acids (C18:2, C18:3, C20:2, C20:4).

between frying oils and NL and FFA fractions, whereas PL composition remained unchanged after the frying process. With regard to the NL composition, the fatty acid composition of the samples fried with pig lard did not differ markedly from that of raw loin chops. However, SFA (46.8%) increased in pork loin chops fried in butter. In the case of frying in olive oil, a rise of MUFA (62.5%) was found, mainly due to the increase of oleic acid (59.1%), whereas the rise of PUFA (32.5%) in samples fried in sunflower oil was mainly due to the increase of linoleic acid (31.3%). FFA profiles also reflected the fatty acid profile of the culinary fat. SFA (40.9%) percentages significantly increased in butter-fried loin chops, and PUFA (33.7%) percentages significantly rose in samples fried in sunflower oil. According to these results, frying produced an exchange between fat in the pork loin chops and the frying medium, which caused significant changes in the fatty acid composition.

**Analysis of Volatiles in Raw and Fried Meat. Table 3** summarizes GC-MS data [expressed as area units (AU) $\times 10^6$] obtained from the analysis of volatile compounds from raw and fried pork loin chops in different culinary fats. Seventy-one different compounds were identified and were classified according to their chemical nature into 11 classes: acids, ketones, aldehydes, alcohols, hydrocarbons, terpenes, sulfur compounds, furans, nitrogen compounds, phenolic compounds, and others. ANOVA results showed significant differences ($p < 0.05$) among groups in most of the compounds detected, except in seven compounds (propan-2-one, heptanal, octanal, nonanal, hept-1-en-3-ol/oct-1-en-3-ol, octan-1-ol, and cyclooctane), which indicates that modifications in the volatile profile of raw meat changed by frying and with the type of culinary fat used.

Thirty-three volatiles were detected in raw pork loin chops (m. *Longissimus dorsi*), 32 volatiles in olive oil-fried pork loin chops, 41 volatiles in sunflower oil-fried pork loin chops, 35 volatiles in butter-fried pork loin chops, and 41 volatiles in pig lard-fried pork loin chops.

**Analysis of Volatiles in Raw Meat.** The most abundant volatiles isolated in raw meat were carboxylic acids (acetic acid, pentanoic acid, hexanoic acid, benzoic acid, octanoic acid, nonanoic acid, and decanoic acid), ketones with low numbers of carbons (propan-2-one, butan-2-one, butane-2,3-dione, butan-2-one, and 3-hydroxy-1-(2-hydroxyphenyl)ethanone], alcohols (2-ethylhexan-1-ol, oct-1-en-3-ol, and octan-1-ol), hydrocarbons [1,1′-oxybis(ethane) and pentadecane], a nitrogen compound (benzamide), and some phenolic compounds [1,2,3,5-tetramethylbenzene, ethyl-(1-methylethenyl)benzene, 2-propenal-3-phenyl, 2-[1-(4-hydroxyphenyl)-1-methylethyl]phenol, 2,6-bis-(1,1-dimethylethyl)-4-methylphenol, and 4,4′-(1-methylethylindene)bis(phenol)]. Terpenes, sulfur compounds, and furans were not identified in the headspace of raw meat.

The detection of some compounds isolated only in raw meat, such as butane-2,3-dione, could be explained because of the enzymatic activity from pyruvate catabolism (*16*), which disappeared after cooking, whereas the significant presence of 3-hydroxybutan-2-one in raw meat has been reported to be a meat aging indicator (*11*). 2-Ethylhexan-1-ol was the most abundant alcohol in raw meat, and its presence has been described by other authors in raw and refrigerated meat (*11*). Its origin is not clear; some authors have reported that this compound originates from lipid oxidation (*17*), whereas others mentioned that it is produced by enzymatic degradation of amino acids by muscle and/or microbial proteolytic enzymes (*11*, *18*).

**Effect of Frying on Volatile Profiles.** Frying greatly changed the volatile profile of raw meat, mainly due to the increase of compounds from the Maillard reaction and volatiles closely related to lipid oxidation, such as aldehydes, whereas others such as acids decreased. However, changes in volatile compounds after frying did not affect all samples fried in different culinary fats in the same way, great differences being observed in the formation and/or diminution of some volatile compounds depending on the type of culinary fat used. In general, compounds with origin in the oxidative degradation of lipids were more abundant than those compounds formed by the development of Maillard reactions in fried samples. Lipid-derived volatile compounds comprise aliphatic aldehydes, alcohols, hydrocarbons, ketones, and alkylfurans, whereas volatile compounds formed via the Maillard reaction include heterocyclic nitrogen and sulfur compounds and non-heterocyclic compounds, such as Strecker aldehydes [3-(methylthio)-propanal, 2-methylbutanal, 3-methylbutanal, and bezeneacetaldehyde], alkenediones, and hydroxyketones as well as aliphatic and furan disulfides (*19*).

The volatile compounds found in the headspace of fried meat and not found in raw meat could have their origins in (i) the direct incorporation of aromatic compounds from culinary fats to meat without modifications; (ii) the incorporation to meat of compounds originated by the heating of frying fats; (iii) the interaction between compounds from raw meat and the culinary fat at high temperature, that is, due to modifications of composition of meat because of the heating and thermoinduced reactions such as Maillard and lipid degradation reactions (*7*).

Aldehydes were the major class of volatiles in fried loin chops. Total amounts of straight-chain aldehydes increased with

**Table 3.** Volatile Compounds (Area Units × $10^6$) Detected in the Headspace of Raw Pork Loin Chops and and Pork Loin Chops Fried in Olive Oil (OO), Sunflower Oil (SO), Butter (BT), and Pig Lard (PLD)

| | | | fried loins | | | | | ANOVA | method of |
|---|---|---|---|---|---|---|---|---|---|
| | RI[a] | raw[b] | OO[b] | SO[b] | BT[b] | PLD[b] | SEM [c] | *p* value | identification[d] |
| acids | | | | | | | | | |
| acetic acid | 660 | 7.8a | 0c | 1.8b | 1b | 0.7b | 0.7 | 0.000 | a |
| pentanoic acid | 893 | 4.7a | 1.2b | 0c | 0c | 0c | 0.5 | 0.001 | a |
| hexanoic acid | 970 | 2.5a | 1.2b | 0c | 0.9b | 0.6b | 0.2 | 0.000 | a |
| benzoic acid | 1276 | 42.3a | 0c | 0c | 0c | 3.2b | 3.9 | 0.000 | b |
| octanoic acid | 1179 | 0b | 0b | 0b | 3.4a | 0b | 0.3 | 0.000 | a |
| nonanoic acid | 1275 | 15.6a | 0.8b | 0c | 2.3b | 0.2b | 1.5 | 0.000 | a |
| decanoic acid | 1373 | 3.2a | 0b | 0b | 0b | 0b | 0.3 | 0.000 | a |
| ketones | | | | | | | | | |
| propan-2-one | 503 | 7.4 | 6.4 | 5.0 | 5.8 | 4.1 | 0.5 | 0.182 | b |
| butane-2,3-dione | 593 | 3.6a | 0b | 0b | 0b | 0b | 0.3 | 0.000 | a |
| butan-2-one | 597 | 9.7a | 0b | 0b | 0b | 0b | 1.0 | 0.000 | a |
| 3-hydroxybutan-2-one | 711 | 6.0a | 0c | 1.9b | 0c | 1.3c | 0.5 | 0.000 | a |
| 2,5-dimethylhexan-3-one | | 0b | 0b | 0b | 0b | 1.2a | 0.1 | 0.000 | c |
| heptan-2-one | 888 | 0b | 0b | 0b | 1.5a | 0b | 0.2 | 0.000 | a |
| octane-2,3-dione | 980 | 0c | 7.0a | 9.9a | 1.8b | 2.0b | 1.1 | 0.015 | a |
| nonan-2-one | 1093 | 0b | 0b | 0b | 2.3a | 0b | 0.2 | 0.000 | b |
| undecan-2-one | 1282 | 0b | 0b | 0b | 6.8a | 0b | 0.6 | 0.000 | b |
| 1-(2-hydroxyphenyl)ethanone | | 5.8a | 0b | 0b | 0b | 0b | 0.5 | 0.000 | c |
| tridecanone | | 0b | 0b | 0b | 5.9a | 0b | 0.5 | 0.000 | c |
| heptadecan-2-one | 1910 | 0b | 0b | 0b | 3.0a | 0b | 0.3 | 0.000 | b |
| aldehydes | | | | | | | | | |
| 3-methylbutanal | 654 | 0c | 2.4a | 0.9b | 1.6ab | 2.1a | 0.2 | 0.000 | a |
| 2-methylbutanal | 662 | 0b | 0b | 0b | 0b | 1.7a | 0.1 | 0.000 | a |
| pentanal | 669 | 4.9b | 26.2ab | 37.7a | 9.0b | 7.3b | 3.5 | 0.003 | a |
| hexanal | 799 | 16.2c | 235.4b | 655.3a | 77.6c | 77.2c | 46.9 | 0.000 | a |
| hex-2-enal | 848 | 0b | 0b | 1.5a | 0b | 0b | 0.1 | 0.000 | b |
| heptanal | 900 | 20.3 | 6.6 | 11.8 | 11.7 | 7.0 | 2.1 | 0.194 | a |
| 3-(methylthio)propanal | 911 | 0b | 0b | 0b | 0b | 1.7a | 0.1 | 0.000 | b |
| hept-(*E*)-2-enal | 956 | 0b | 4.8a | 0b | 0b | 0b | 0.5 | 0.000 | b |
| benzaldehyde | 962 | 17.7a | 16.1a | 0b | 24.7a | 29.1a | 2.4 | 0.000 | a |
| benzeneacetaldehyde | 1042 | 0d | 0.6bc | 0.4c | 1.2a | 0.8ab | 0.1 | 0.000 | b |
| octanal | 1004 | 23.9 | 17.7 | 14.5 | 23.5 | 11.8 | 2.8 | 0.608 | a |
| oct-(*E*)-2-enal | 1060 | 0d | 3.1ab | 4.9a | 2.9bc | 1.9c | 0.4 | 0.003 | b |
| nonanal | 1104 | 55.5 | 34.3 | 37.5 | 49.2 | 35.2 | 4.7 | 0.530 | a |
| non-2-enal | 1160 | 0b | 0b | 0b | 1.7a | 0b | 0.2 | 0.000 | b |
| decanal | 1204 | 4.3a | 1.9b | 2.0b | 3.1ab | 1.9b | 0.2 | 0.000 | a |
| deca-(*E*,*Z*)-2,4-dienal | 1309 | 0b | 0b | 1.2a | 0b | 0b | 0.1 | 0.000 | a |
| dodecanal | 1412 | 14.9a | 0c | 1.8b | 0c | 2.6b | 1.2 | 0.000 | b |
| alcohols | | | | | | | | | |
| pentan-1-ol | 766 | 0c | 3.4ab | 6.0a | 1.4bc | 1.2b | 0.5 | 0.001 | a |
| hexan-1-ol | 865 | 0b | 0b | 2.2a | 0b | 0b | 0.2 | 0.000 | a |
| oct-1-en-3-ol | 980 | 5.8ab | 10.7ab | 20.2a | 9.3ab | 4.2b | 1.8 | 0.063 | a |
| 2-ethylhexan-1-ol | 1028 | 18.4a | 0c | 1.1b | 0c | 0c | 1.6 | 0.000 | a |
| octan-1-ol | 1075 | 4.7 | 4.4 | 4.2 | 5.0 | 2.6 | 0.5 | 0.654 | a |
| hydrocarbons | | | | | | | | | |
| 1,1′-oxybis(ethane) | | 2.9a | 0c | 0c | 0c | 2.2b | 0.4 | 0.004 | c |
| 2-methylpentane | 573 | 0b | 2.5a | 2.3a | 1.9a | 1.8a | 0.2 | 0.000 | b |
| 3-methylpentane | 577 | 0b | 3.0a | 0b | 3.5a | 29a | 0.4 | 0.002 | b |
| methylcyclopentane | 535 | 0c | 22a | 22a | 26a | 15b | 0.3 | 0.013 | b |
| 3-ethyl-2-methylhexa-(*Z*)-1,3-diene | | 0b | 0b | 1.4a | 0b | 0b | 0.1 | 0.002 | c |
| dec-2-ene(*Z*)/pentene-2,3-dimethyl | | 0b | 2.7a | 0b | 0b | 1.7a | 0.3 | 0.000 | c |
| cyclooctane | | 4.9 | 3.3 | 3.6 | 6.5 | 4.6 | 0.5 | 0.247 | c |
| pentadecane | 1500 | 3.7a | 0b | 0b | 0b | 0b | 0.4 | 0.000 | b |
| terpenes | | | | | | | | | |
| 1-limonene | 1031 | 0c | 53.4a | 3.5b | 3.4b | 4.4b | 5.0 | 0.000 | a |
| sulfur compounds | | | | | | | | | |
| dimethyl disulfide | 744 | 0b | 0b | 0b | 0b | 0.8a | 0.1 | 0.000 | b |
| furans | | | | | | | | | |
| 2-pentylfuran | 994 | 0c | 0.8b | 1.8a | 0c | 0.7b | 0.2 | 0.000 | a |
| nitrogen compounds | | | | | | | | | |
| pyridine | 751 | 2.4a | 0b | 3.1a | 0b | 0.8b | 0.3 | 0.000 | a |
| 2,5-dimethylpyrazine | 996 | 0b | 0b | 0.9a | 0b | 0b | 0.1 | 0.000 | b |
| 2-ethyl-3,5-dimethylpyrazine | 1136 | 0b | 0b | 0.8a | 0b | 0b | 0.1 | 0.000 | b |
| nitrobenzene | | 0b | 0b | 0.6a | 0b | 0b | 0.1 | 0.000 | c |
| benzamide | | 6.9a | 0b | 0b | 0b | 0b | 0.6 | 0.000 | c |
| phenolic compounds | | | | | | | | | |
| methylbenzene | 772 | 0b | 42.8a | 30.1a | 48.0a | 38.0a | 4.6 | 0.001 | b |
| 1*H*-indole-2-methyl-3-phenyl | | 0b | 0b | 0.4a | 0b | 0b | 0.0 | 0.000 | c |
| 1,2-dimethylbenzene | 868 | 0c | 1.8b | 2.8a | 0c | 1.0b | 0.2 | 0.000 | b |
| 1,3,5-trimethylbenzene | 1003 | 0c | 0.5b | 0.9a | 0c | 0.9a | 0.1 | 0.000 | b |
| 2-propenal-3-phenyl | | 13.2a | 0b | 0b | 0b | 0b | 1.2 | 0.000 | c |
| naphthalene | 1186 | 0c | 1.5a | 0c | 0.5b | 0c | 0.1 | 0.000 | b |

**Table 3. (Continued)**

| | RI[a] | raw[b] | fried loins OO[b] | fried loins SO[b] | fried loins BT[b] | fried loins PLD[b] | SEM[c] | ANOVA *p* value | method of identification[d] |
|---|---|---|---|---|---|---|---|---|---|
| phenolic compounds (continued) | | | | | | | | | |
| 1,2,3,5-tetramethylbenzene | 1108 | 36.2a | 0c | 1.3b | 0c | 1.2b | 3.1 | 0.000 | b |
| ethyl(1-methylethenyl)benzene | | 5.4a | 0c | 3.3b | 0c | 0c | 0.5 | 0.000 | c |
| 2-[1-(4-hydroxyphenyl)-1-methylethyl]phenol | | 14.2a | 0c | 0c | 1.4b | 0c | 1.2 | 0.000 | c |
| 2,6-bis(1,1-dimethylethyl)-4-methylphenol | | 14.1a | 3.8b | 2.6b | 3.3b | 2.1b | 1.0 | 0.000 | c |
| 4,4′-(1-methylethylindene)bis(phenol) | | 390.7a | 26.1b | 18.1b | 13.0b | 10.0b | 33.0 | 0.000 | c |
| others | | | | | | | | | |
| benezene, isothiocyanato | | 0b | 0b | 3.0a | 0b | 0b | 0.3 | 0.014 | c |
| 1-methylbenzene-2,4-diisocyanato | | 0b | 0b | 1.1a | 0b | 0b | 0.1 | 0.000 | c |
| butyl hydroxyanisole | | 0b | 0b | 0b | 0b | 8.5a | 0.7 | 0.000 | c |

[a] RI, relative indices in agreement with literature values. [b] Letters a–d in the same row indicate statistically significant differences. [c] SEM, standard error of the mean. [d] Method of identification: a, mass spectrum and retention time identical with a reference compound; b, mass spectrum and retention index from lthe iterature in accordance; c, tentative identification by mass spectrum.

frying, probably due to the heat-induced oxidation of unsaturated fatty acids from culinary fats and muscle (*9*). Aliphatic aldehydes derived from oxidative degradation of PUFA are probably the most interesting lipid-derived volatiles, because they have low odor threshold values and may play an important role in the flavor of the fried pork samples (*9*, *20*). Frying increased the amounts of most aldehydes [pentanal, hexanal, hex-2-enal, oct-(*E*)-2-enal, non-2-enal, deca-(*E*,*Z*)-2,4-dienal], whereas others remained unchanged (heptanal, octanal, and nonanal) and yet others decreased after frying (decanal and dodecanal). In a previous work, the authors (*3*) also found a significant increase in lipid oxidation in raw meat after frying in different culinary fats, measured as thiobarbituric acid reactive substances (TBA-RS). Hexanal, the most abundant aldehyde in fried samples, increased after frying with respect to raw meat by 14.5-, 40.45-, 4.8-, and 4.8-fold in olive oil-fried samples, sunflower oil-fried samples, butter-fried samples, and pig lard-fried samples, respectively. Contrarily to aldehydes, ketones with a small number of carbons such as butan-2-one, butane-2,3-dione, and 3-hydroxybutan-2-one were missing from the headspace of fried loin samples, and being detected in only raw meat. Some ketones were isolated in all fried samples such as octane-2,3-dione, whereas others were found in only some fried samples, such as high-carbon-number ketones in butter-fried loin chops. With regard to alcohols, frying increased the amounts of some alcohols such as pentan-1-ol and oct-1-en-3-ol, but others such as 2-ethylhexan-1-ol significantly decreased in sunflower-oil-fried samples and were not detected in loin chops fried in olive oil, butter, or pig lard. After frying, a significant reduction of acids in the headspace of fried meat was observed. In this sense, acetic acid, pentanoic acid, hexanoic acid, benzoic acid, nonanoic acid, and decanoic acid decreased or were not detected in fried samples. Finally, hydrocarbons (linear and cyclic) contents increased in fried samples with respect to raw meat, with the exception of pentadecane, which was detected in only raw meat. Ketones, alcohols, and acids have been associated with the oxidative degradation of unsaturated fatty acids (*17*), and all of them may contribute as precursors to the desirable cooked meat flavor. The contribution of hydrocarbons to meat aroma is not considered to be important, but their presence in various meat aromas has been described (*21, 22*).

Frying generated 2-pentylfuran in the headspace of all fried samples, except in butter-fried loin chops, in which this compound was not detected. 2-Pentylfuran is a non-carbonyl oxidation product from linoleic and other $n-6$ PUFAs, which was frequently found in meat products (*23*). This compound shows a relatively low threshold, and, therefore, its presence could play an important role in the overall flavor of fried loin chops, along with being an indicator of lipid oxidation.

Four Strecker aldehydes were identified in fried loin chop headspaces [3-(methylthio)propanal, 2-methylbutanal, 3-methylbutanal, and benzeneacetaldehyde]. 3-Methylbutanal and benzeneacetaldehyde were isolated from the headspace of all fried samples, wheres 2-methylbutanal and 3-(methylthio)-propanal were isolated only in loin chops fried in pig lard. 2- and 3-methylbutanal are products of the Strecker degradation of the amino acids isoleucine and leucine and benzeacetaldehyde, of the amino acid phenylalanine (*23*). Due to their low threshold value, these compounds have been described as contributors to the desirable overall roasted flavor of cooked meat (*24*).

A small number and small amounts of nitrogen compounds (nitrobenzene, 2,5-dimethylpyrazine, 2-ethyl-3,5-dimethylpyrazine, and pyridine) were isolated in the headspace of samples fried in sunflower oil and pig lard. Nitrogen-containing volatile flavor compounds originate from the breakdown of proteins, free amino acids, and nucleic acids, whereas sulfur-containing volatile flavor compounds are derived from sulfur-containing amino acids (*7*). Mottram et al. (*25*) isolated 27 pyrazines in well-done grilled pork. Differences in the temperatures reached during cooking, much lower in frying than in grilling, and/or the method of isolation of volatiles could be likely responsible of the less presence of this type of compounds.

One terpene, 1-limonene, was detected in loin chops after frying, but the amounts detected in fried samples were different depending on the type of culinary fat used. This compound has been described to proceed from the essential oils of some plants (*26*), although other authors have described this volatile like an off-flavor compound originated from lipid oxidation (*27*).

**Differences in Volatile Profiles in Loin Chops Fried in Different Culinary Fats.** Some volatile compounds were found in only one of the groups of fried meat, so these compounds are characteristics from fried meat in certain fats and they probably play an important role in the distinctive aroma of meat fried in a particular culinary fat.

Sunflower oil-fried pork loin chops showed large amounts of lipid-derived products, mainly aldehydes, ketones, and alcohols. As a result, total aldehydes were higher in sunflower oil-fried samples than in the other fried pork loin chops: OO, 349.1; SO, 769.5; BT, 206.2; PLD, 180.3 ($\times 10^6$ AU). Besides hexanal, which is the major aldehyde, pentanal and octen-2-al presented significantly higher levels in samples fried in sunflower oil; and others, such as hex-2-enal and deca-(*E*,*Z*)-2,4-dienal, were only just detected in these samples. Five alcohols

were identified in sunflower oil-fried samples (pentan-1-ol, hexan-1-ol, 2-ethylhexan-1-ol, oct-1-en-3-ol, and octan-1-ol), the content of pentan-1-ol, hexan-1-ol, and oct-1-en-3-ol being significantly greater in this group than in samples fried in other culinary fats. Other lipid oxidation derived compounds, such as ketones (e.g., octane-2,3-dione) and non-carbonyl oxidation products (e.g., 2-pentylfuran) were more abundant in the headspace of samples fried in sunflower oil than in other fried samples. Volatile differences with respect to other fried samples are in agreement with the large content of PUFA, principally linoleic acid (C18:2*n*−6), in sunflower oil and NL and FFA of intramuscular lipids from fried samples (**Tables 1** and **2**). In this sense, Warner and Mounts (*28*) have reported that foods fried in oils with high linoleic acids content developed rancid and fishy flavors. Additionally, previous studies indicated that altering the fatty acid composition of muscles can affect greatly the volatile composition and hence its characteristic flavor of cooked meat (*8*). Elmore et al. (*9*) have reported that the increase of PUFA contents in meat leads to a larger content of lipid oxidation products derived from the autoxidation of the more abundant unsaturated fatty acid during cooking and, consequently, to modifications in the aroma of cooked meat. Among 10 compounds, which were only just detected in the headspace of sunflower oil-fried meat, three were nitrogen compounds such as pyridine and pyrazines (2,5-dimethylpyrazine and 2-ethyl-3,5-dimethylpyrazine), the aroma of which is associated with roasted, grilled meat (*7*). Bicyclic pyrazines, such as 2,5-dimethylpyrazines, have been described in the volatile headspace from well-done grilled pork (*29*). The formation of pyrazines has been extensively studied, and a probable route to the formation of alkylpyrazines is from the condensation of two α-aminoketone molecules produced in the Strecker degradation of amino acids by dicarbonyl compounds (*7*). These heterocyclic compounds derived from the Maillard reaction (MRPs) were only just isolated in sunflower oil-fried samples probably due to a high development of Maillard reaction. In previous works (*3*, *30*), authors found that samples fried in sunflower oil showed a larger amount of water-extracted colored compounds associated with Maillard compounds and a significantly lower value of *L** measured as color instrumental than other samples. Other compounds detected in only sunflower oil-fried pork loin chops were several hydrocarbons such as 3-ethyl-2-methylhexa-(*Z*)-1,3-diene and phenolic compounds such as nitrobenzene, 1*H*-indole-2-methyl-3-phenyl, benzene-2,4-diisocyanate, and 1-methylbenzene-2,4-isothiocyanate. The latter compounds have been described as aroma components originated by lipid oxidation and have been abundantly isolated from cooked beef (*31*).

Olive oil-fried samples showed a similar trend to samples fried in sunflower oil. Headspace from olive oil-fried samples showed a large content of lipid oxidation derived volatiles, especially aliphatic aldehydes that were more abundant in these samples than in samples fried in butter and pig lard but much lower than in samples fried in sunflower oil. Olive oil-fried pork loin chops were rich in pentanal, hexanal, hept-(*Z*)-2-enal, benzaldehyde, and oct-(*E*)-2-enal; meanwhile, only two ketones (propan-2-one and octan-2,3-dione) were detected in samples fried in olive oil. Some lipid derived compounds such as pentan-1-ol, hexanal, hept-2-enal, nonanal, decanal, benzaldehyde, and nonan-2-one have been described as contributors of the characteristic flavor of olive oil (*32*) and might contribute to a distinctive flavor of meat fried in this culinary oil (*33*). A possible explanation for the large amounts of lipid-derived volatile compounds in olive oil-fried samples could be the thermal degradation of oleic and linoleic acids, the most abundant unsaturated fatty acid in olive oil and olive oil-fried samples. This is suggested by the high level of aldehydes derived from these fatty acids in loin chops fried in olive oil. Finally, there is a remarkable content of 1-limonene in these fried samples, significantly higher than in other fried meat, with its origin likely in olive oil.

In contrast, butter-fried loin chops contained large amounts of high-carbon-number ketones (heptan-2-one, nonan-2-one, undecan-2-one, tridecanone, and heptadecan-2-one), which were not detected in other fried samples. Ketones have been described in the headspace of cooked samples and have been associated with the "buttery" aroma note of cooked meats (*34*). Thereby, Elmore et al. (*9*) detected C5−C10 ketones in volatiles of cooked beef, which have been found in butter-fried samples from this experiment, whereas ketones with >10 carbons were not reported in cooked beef by the aforementioned author and were not detected in the other fried samples. Because of the large content of ketones in butter, these compounds in fried samples are likely to have their origin in butter or in thermal changes of ketones in butter during frying and their incorporation to meat during the lipid exchange between butter and fat meat (*2*, *3*).

In general, pig lard-fried meat showed fewer volatiles than loin chops fried in the other culinary fats. However, pig lard-fried samples presented seven volatiles that were detected in only these fried samples. Two of them were 2-methylbutanal and 3-(methylthio)propanal, which are Strecker aldehydes and which might play an important role in the aromatic profile of these samples, as previously described by Machiels et al. (*24*) in cooked beef. 3-(Methylthio)propanal is derived from sulfur-containing amino acids (*7*). Dimethyl disulfide is a sulfur compound isolated in only pig lard-fried samples, which is also derived from sulfur-containing amino acids. Concretely this compound is a product formed from a Strecker aldehyde, which results from methionine degradation (*35*). Finally, butyll hydroxyanisole (BHA), an antioxidant permitted in fat, was just detected in only pig lard-fried samples. It was probably added to pig lard during the elaboration process, which may have reduced lipid oxidation during frying.

In conclusion, the different fatty acid compositions of the pork loin chops fried in each culinary fat and the modifications of their oxidative susceptibility could be influential factors in the generation of volatile compounds. Differences in the volatile profiles, relative amounts, presence/absence of particular compounds, the relationships between the different compounds, and their aromatic characteristics might contribute to the overall distinctive flavor of samples fried in the different culinary fats.

## ACKNOWLEDGMENT

We thank Inmaculada Linares for excellent technical assistance.

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**Received for review May 18, 2004. Revised manuscript received September 21, 2004. Accepted October 2, 2004. M.R.R. is grateful to the University of Extremadura and to the Ministerio de Educación y Ciencia for a grant.**

JF049207S