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Research Article

Determination of Volatile Compounds of Sucuk with Different Orange Fiber and Fat Levels [1]

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Abstract

In this study, the effects of fat (10, 15 and 20%) and orange fiber (0, 2 and 4%) levels on volatile compounds of sucuk (Turkish dry-fermented sausage) were investigated. The volatile compound profile of sucuk samples was analyzed by gas chromatography/mass spectrometry (GC-MS) by using a solid phase microextraction (SPME). 75 volatile compounds were identified in the sucuk samples. The volatile compounds identified were 11 aldehydes, 7 aliphatic hydrocarbons, 2 acids, 10 esters, 1 furan, 5 alcohols, 8 aromatic hydrocarbons, 6 ketones, 6 sulphur compounds, and 19 terpenes. It was determined that the use of orange fiber had significant ($P<0.05$) effects on a few volatile compounds (allyl methyl sulfide, copaene and caryophyllene). Alpha-thujene was significantly affected by fat level ($P<0.05$). Fat level also showed very significant effect on p-xylene and allyl methyl sulfide ($P<0.01$). As result, orange fiber and fat level had no significant effects on volatile profile of sucuk samples.

Keywords: *Sucuk, SPME, Volatile compounds, Orange fiber, Fat*

Farklı Oranlarda Portakal Lifi ve Yağ İçeren Sucukların Uçucu Bileşiklerinin Belirlenmesi

Özet

Bu çalışmada, sucuğun (Türk tipi kuru-fermente sosis) uçucu bileşikleri üzerine yağ (%10, 15 ve 20) ve portakal lifi (% 0, 2 ve 4) oranlarının etkileri araştırılmıştır. Sucuk örneklerinin uçucu bileşik profili katı faz mikroekstraksiyon (SPME) tekniği kullanılarak gaz kromatografisi/kütle spektrometresi (GC/MS) ile analiz edilmiştir. Sucuk örneklerinde 75 uçucu bileşik tanımlanmıştır. Uçucu bileşik olarak 11 aldehit, 7 alifatik hidrokarbon, 2 asit, 10 ester, 1 furan, 5 alkol, 8 aromatik hidrokarbon, 6 keton, 6 sülfürlü bileşik ve 19 terpen belirlenmiştir. Portakal lifi kullanımının az sayıda uçucu bileşik (allil metil sülfit, kopaen ve karyofillen) üzerinde önemli ($P<0.05$) etkiye sahip olduğu belirlenmiştir. Alfa-thujen, yağ oranından önemli şekilde etkilenmiştir ($P<0.05$). Ayrıca yağ oranı p-ksilen ve allil metil sülfit üzerine çok önemli ($P<0.01$) etki göstermiştir. Sonuç olarak, portakal lifi ve yağ oranının sucuk örneklerinin uçucu bileşikleri üzerinde önemli bir etkiye sahip olmadığı belirlenmiştir.

Anahtar sözcükler: *Sucuk, SPME, Uçucu bileşikler, Portakal lifi, Yağ*

INTRODUCTION

Sucuk is a dry fermented sausage, one of the most important of traditional Turkish meat products. It is produced with beef meat and/or water buffalo meat/sheep meat, sheep tail fat and/or beef fat. Curing ingredients (nitrite and/or nitrate), salt, sucrose, and various spices including cumin, garlic, pimento, red and black peppers are also used in sucuk production. After stuffing the mixture into natural sausage casings (air-dried bovine small intestines) or small casings of similar characteristics, fermentation and drying steps are carried out under controlled or natural climatic conditions [1,2]. According to Communique of Meat and Meat Products of Turkish Food Codex (No: 2012/74) [3], sucuk must have 40% maximum moisture content and 5.4 maximum pH value.

Volatile compounds generated during processing of dry fermented sausage have great importance on aromatic character of the product. Raw meat contains a large number of aroma precursors which are converted

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to volatile compounds during ripening [4]. These compounds have the ability to influence the quality of products due to their effects on sensorial properties. Volatile compounds generated during production and storage can occur as a result of a) ingredients used in formulation, b) conditions used in production, c) microbial activities of starter and endogenous microorganisms such as amino acid catabolism, carbohydrate fermentation and d) lipid oxidation [5,6].

Sucuk has a large number of volatile compounds. This product is especially rich in terms of terpens from spices which could play an important role in the overall aroma. On the other hand, sulfur compounds (diallyl disulfide, 1, propene 3-thiobis, disulfide methyl 2-phenyl), acids (especially acetic acid), and aldehydes (p-cumic aldehyde) are also found at significant levels. The profile of these compounds could display some differences due to differences in raw materials, spices and other ingredients, and production conditions [7].

Turkey is an important orange producer [8]. Citrus fruits such as orange are mainly processed to fruit juice and millions of tones waste are released every year around the world which could cause an environmental problem. In order to eliminate this problem, citrus wastes are processed to valuable by products like dietary fibers [9]. As well as their physiological properties, dietary fibers could improve the product's texture and decrease the cooking loss with their technological properties [10].

In order to understand the effects of dietary fiber in dry-fermented sausages, many researches have been carried out in the last decade [11-13]. However, the effects of fiber addition and decrease of fat level on volatile compound profile of sucuk have not been examined, yet. In this study, the effects of different levels of fat and orange fiber on volatile compound profile of sucuk were investigated by using gas chromatography/mass spectrometry (GC-MS) with solid phase microextraction (SPME).

MATERIAL and METHODS

Production of Orange Fiber

Cooked and dried orange fiber was obtained according to a method offered by Fernandez-Gines et al.[14].

Sausage Formulation and Processing

Two replicates were designed for the study (Experiment I and Experiment II). Nine sucuk batters were prepared for each experiment based on fat level (90% lean meat + 10% sheep tail fat, 85% lean meat + 15% sheep tail fat, and 80% lean meat + 20% sheep tail fat) and orange fiber (0%, 2% and 4%). As a parallel research project to that of Yalınkılıç et al.[12], the following ingredients (g/kg) and ripening conditions were used; 25 g/kg NaCl, 10 g/kg garlic, 7 g/kg red pepper, 4 g/kg sucrose, 5 g/kg black pepper, 2.5 g/kg pimento, 9 g/kg cumin, and 0.15 g/kg $NaNO_2$ [1]. The amount of orange fiber was calculated over the total mixture and added to batters at different levels (0, 2 and 4%). *Lactobacillus plantarum* GM77 and *Staphylococcus xylosus* GM92 strains [15] were used as starter culture approximately in the level of 10^6 cfu/g, as explained by Kaban and Kaya [1]. Sucuk batters were prepared in a laboratory-type cutter (MADO Typ MTK 662, Dornhan, Schwarzwald). Prepared batters were stuffed into collagen casings (38 mm, NaturinDarm, Germany) using a laboratory-type filling machine (MADO Typ MTK 591, Dornhan/ Schwarzwald). Sucuk samples were fermented and dried in an automatic climate unit (Reich, Klima-Rauchertechnik, Stuttgart). The ripening process was carried out under the following conditions: first day 22°C, second and third days 20°C and 18°C for the following days. In the first three days relative humidity (RH) was 90±2% and on the other days the RH was decreased gradually to 82±2%.

Sampling Procedure

Volatile compound profile examination was carried out using ripened sucuk samples from nine different groups at day 9.

Analysis of Volatile Compounds

Analysis of volatile compounds was done according to a method specified by Kaban [7]. The extraction of headspace volatile compounds was performed using a SPME device (Supelco, Bellefonte, PA), using fibers of 75 µm, carboxen/polydimethylsiloxane (CAR/PDMS). For each sample, 5 g minced sample was weighed into a 40 ml headspace vial and sealed with a PTFE-faced silicone septum (Supelco, Bellefonte, PA, USA). The vial was left at 30°C in a thermo block (Supelco, Bellefonte PA, USA) during 1 h to equilibrate its headspace. Then, a SPME fiber was exposed to the headspace while maintaining the sample at 30°C during 2 h.

The volatile compounds adsorbed by the fiber were desorbed from the injection port of the gas chromatography (GC, Agilent Technologies 6890N) for 6 min at 250°C with the purge valve off (splitless mode). DB-624 (J&W Scientific, 30 m, 0.25 mm i.d., 1.4 µm film) capillary column was used in separation of volatile compounds. The GC was equipped with a mass selective detector (MS, Agilent Technologies 5973). Helium was used as carrier gas. The GC oven temperature was started to increase as programmed when the fiber was inserted and held at 40°C for 5 min and subsequently programmed from 40 to 110°C at 3°C/min and at a rate of 4°C/min from 150°C, then at a rate of 10°C/min from 210°C where it was held for another 15 min. GC-MS interface was maintained at 240°C. Mass spectra were obtained by electron impact at 70 eV, and data were acquired across the range 30-400 amu. The compounds were determined by comparing the

results with mass spectra from a database developed by NIST and WILEY or standards molecules (for calculating Kovats Index, Supelco 44585-U, Bellefonte PA, USA) and by matching their retention indices with those in the literature. Quantification was based on a total ion chromatogram on an arbitrary scale.

Statistical Analysis

All data from Experiment I and Experiment II were subjected to variance analysis (complete randomized design, two replications) and differences between means were evaluated by Duncan's multiple range test using the SPSS 13.0.0.246 for Windows (SPSS, Inc., Chicago, III., USA). The results of statistical analysis are shown as mean values ± standard deviation in tables.

RESULTS

Seventy five volatile compounds including eleven aldehydes, two acids, five alcohols *(Table 1)*, seven aliphatic hydrocarbons, eight aromatic hydrocarbons, one furan *(Table 2)*, ten esters, six ketones, six sulphur compounds *(Table 3)*, and nineteen terpenes *(Table 4)* were identified in sucuk samples.

Eleven different aldehyde compounds were identified and quantified (Table 1). Both fat and fiber level had no effect on aldehyde profile (P>0,05). Within aldehyde compounds, 2-methyl-3-phenyl propanal had the highest peak area and it was followed by hexanal. An increase in fat level from 10 to 20% increased acetaldehyde amount which was not statistically significant (P>0.05). On the other hand, an increase in fiber level caused a slight rise in levels of pentanal, hexanal, heptanal, 2-methyl-3-phenyl propanal, 2-hexanal and 2-heptenal. In contrast to aldehydes, only two acids; acetic acid and butanoic acid were identified *(Table 1)*. Both fiber and fat levels had no effects on acid profile (P>0.05). Five different alcohols were detected by solid phase microextraction (SPME) method *(Table 1)*. Each identified alcohol was not significantly affected by both fiber and fat levels (P>0.05). The highest peak was obtained for 4-(1-methylethyl) benzene methanol. Second highest peak was obtained for ethanol.

Seven aliphatic hydrocarbons were determined in samples, but these compounds were not affected by fat

Table 1. *Aldehyde, acid and alcohol compounds of sucuk produced by using different fat and orange fiber levels*
Tablo 1. *Farklı yağ ve portakal lif oranları kullanılarak üretilen sucukların aldehit, asit ve alkol bileşikleri*

Compound	KI	S	Fat Level (%)			S	Fiber Level (%)			Fat Level x Fiber Level S
			10	15	20		0	2	4	
Aldehydes										
Acetaldehyde	623	NS	0.18±0.09a	0.24±0.15a	0.45±0.54a	NS	0.26±0.14a	0.19±0.17a	0.41±0.54a	NS
Pentanal	742	NS	0.40±0.33a	0.38±0.33a	0.42±0.28a	NS	0.29±0.22a	0.33±0.32a	0.57±0.31a	NS
Hexanal	849	NS	0.76±0.56a	0.80±0.84a	0.79±0.43a	NS	0.53±0.28a	0.63±0.42a	1.20±0.81a	NS
2-Furancarboxaldehyde	902	NS	0.01±0.01a	0.00±0.00a	0.01±0.01a	NS	0.01±0.01a	0.01±0.01a	0.01±0.01a	NS
2-Hexanal	922	NS	0.01±0.01a	0.03±0.02a	0.03±0.03a	NS	0.01±0.02a	0.02±0.02a	0.03±0.03a	NS
Heptanal	955	NS	0.38±0.30a	0.47±0.61a	0.42±0.34a	NS	0.27±0.25a	0.31±0.24a	0.69±0.57a	NS
2-Heptenal	1022	NS	0.04±0.03a	0.04±0.02a	0.05±0.04a	NS	0.03±0.02a	0.04±0.03a	0.06±0.04a	NS
Octanal	1054	NS	0.16±0.14a	0.44±0.48a	0.17±0.16a	NS	0.23±0.21a	0.32±0.51a	0.24±0.19a	NS
Nonanal	1163	NS	0.54±0.15a	0.54±0.13a	0.53±0.12a	NS	0.50±0.16a	0.50±0.06a	0.61±0.12a	NS
Propanal, 2-methyl-3-phenyl-	1334	NS	2.65±1.03a	2.30±0.80a	2.94±1.34a	NS	2.19±0.59a	2.45±0.10a	3.25±1.29a	NS
Benzaldehyde, 4-methoxy	1369	NS	0.10±0.04a	0.08±0.04a	0.10±0.09a	NS	0.12±0.08a	0.09±0.04a	0.07±0.05a	NS
Acids										
Acetic acid	717	NS	2.07±0.91a	1.54±0.26a	1.52±1.03a	NS	1.76±0.41a	1.74±0.59a	1.64±1.29a	NS
Butanoic acid	890	NS	0.05±0.02a	0.05±0.02a	0.04±0.02a	NS	0.04±0.01a	0.05±0.01a	0.05±0.03a	NS
Alcohols										
Ethanol	539	NS	0.35±0.44a	0.12±0.10a	0.11±0.10a	NS	0.26±0.45a	0.17±0.18a	0.14±0.08a	NS
1-Hexanol	931	NS	0.03±0.03a	0.05±0.02a	0.05±0.03a	NS	0.04±0.03a	0.05±0.03a	0.04±0.03a	NS
Benzyl Alcohol	1136	NS	0.15±0.06a	0.12±0.04a	0.08±0.05a	NS	0.12±0.03a	0.12±0.05a	0.12±0.08a	NS
Phenylethyl Alcohol	1211	NS	0.22±0.25a	0.06±0.02a	0.09±0.04a	NS	0.15±0.25a	0.13±0.12a	0.09±0.04a	NS
Benzenemethanol, 4-(1-methylethyl)	1382	NS	2.06±0.70a	1.23±0.85a	1.95±0.91a	NS	2.00±0.81a	1.63±0.76a	1.60±1.09a	NS

S: *significance,* ***NS:*** *Not Significant; Results are expressed in Arbitrary Area Units (×10⁻⁶) as means of 3 replicates of each sausage;* ***KI:*** *Kovats index calculated for DB-624 capillary column (J & W scientific: 30 m, 0.25 mm id, 1.4 lm film tickness) installed on a gas chromatograph equipped with a mass selective detector*

Table 2. *Aliphatic hydrocarbon, aromatic hydrocarbon and furan compounds of sucuk produced by using different fat and orange fiber levels*
Tablo 2. *Farklı yağ ve portakal lif oranları kullanılarak üretilen sucukların alifatik hidrokarbon, aromatik hidrokarbon ve furan bileşikleri*

Compound	RI	S	Fat Level (%)			S	Fiber Level (%)			Fat Level x Fiber Level S
			10	15	20		0	2	4	
Aliphatic hydrocarbons										
Hexane	600	NS	0.19±0.17a	0.29±0.43a	0.09±0.11a	NS	0.23±0.18a	0.24±0.42a	0.10±0.18a	NS
Heptane	700	NS	0.03±0.03a	0.04±0.04a	0.04±0.03a	NS	0.03±0.03a	0.04±0.04a	0.04±0.04a	NS
Undecane	1100	NS	0.10±0.10a	0.07±0.04a	0.13±0.18a	NS	0.18±0.17a	0.06±0.04a	0.05±0.03a	NS
Dodecane	1200	NS	0.10±0.08a	0.07±0.05a	0.11±0.04a	NS	0.13±0.07a	0.09±0.04ab	0.06±0.05b	NS
Tridecane	1300	NS	0.12±0.06a	0.10±0.04a	0.17±0.12a	NS	0.15±0.06a	0.11±0.06a	0.14±0.13a	NS
Tetradecane	1400	NS	0.11±0.04a	0.11±0.20a	0.12±0.05a	NS	0.14±0.03a	0.11±0.04a	0.10±0.03a	NS
Pentadecane	1500	NS	0.17±0.06a	0.19±0.02a	0.19±0.04a	NS	0.19±0.03a	0.18±0.05a	0.18±0.04a	NS
Aromatic hydrocarbons										
Toluene	789	NS	0.48±0.49a	0.29±0.06a	0.40±0.15a	NS	0.36±0.15a	0.35±0.10a	0.46±0.49a	NS
p-Xylene	892	**	0.05±0.03b	0.06±0.01b	0.11±0.04a	NS	0.08±0.06a	0.08±0.02a	0.06±0.01a	**
Styrene	933	NS	0.13±0.05a	0.10±0.07a	0.16±0.09a	NS	0.14±0.10a	0.16±0.04a	0.09±0.06a	NS
1-methyl-2-(1-methylethyl)-benzene	1062	NS	6.71±1.50a	10.18±3.39a	10.63±4.43a	NS	8.45±2.54a	8.58±3.51a	10.49±4.75a	NS
Eugenol	1460	NS	0.12±0.12a	0.04±0.03a	0.06±0.09a	NS	0.10±0.09a	0.07±0.10a	0.06±0.09a	NS
1,2-dimethoxy-4-(2-propenyl)-benzene	1482	NS	1.34±0.60a	0.87±0.60a	0.97±0.42a	NS	1.22±0.58a	0.93±0.43a	1.03±0.69a	NS
1-methoxy-4-(1-propenyl)-benzene	1342	NS	0.21±0.07a	0.13±0.10a	0.17±0.07a	NS	0.20±0.10a	0.13±0.05a	0.17±0.09a	NS
2-Methoxy-1,3-bis(trimethylsilyl)benzene	1491	NS	0.06±0.02a	0.03±0.02a	0.04±0.02a	NS	0.05±0.03a	0.04±0.02a	0.04±0.02a	NS
Furan										
Furan, 2-pentyl-	1021	NS	0.05±0.09a	0.03±0.02a	0.02±0.02a	NS	0.02±0.2a	0.02±0.02a	0.06±0.09a	NS

S: *significance,* ***NS:*** *Not Significant, ** P<0.01; Results are expressed in Arbitrary Area Units (×10^{-6}) as means of 3 replicates of each sausage;* ***KI:*** *Kovats index calculated for DB-624 capillary column (J & W scientific: 30 m, 0.25 mm id, 1.4 lm film tickness) installed on a gas chromatograph equipped with a mass selective detector*

and orange fiber levels (P>0.05) *(Table 2)*. On the other hand, eight aromatic hydrocarbons were detected in all groups *(Table 2)*. Among these compounds, 1-methyl-2-(1-methylethyl)-benzene had the highest peak. Only p-xylene was very significantly (P<0.01) affected by both fat level and fat x fiber interaction. Only one furan compound was identified in the samples *(Table 2)*.

Ten ester compounds were identified in samples *(Table 3)*. None of them were affected by fiber and fat levels (P>0.05). On the other hand, six ketone compounds were detected in samples *(Table 3)* and only 3,5-octadien-2-one was significantly (P<0.05) affected by interaction of fat level and fiber level.

A total of six sulphur compounds were identified and quantified in experimental sucuk samples *(Table 3)*. Only allyl methyl sulfide was affected very significantly by fat level (P<0.01) and significantly by orange fiber level (P<0.05). In contrast, interaction of fat level and fiber level had no statistically significance (P>0.05).

Terpens were the largest volatile group in the present study with their nineteen identified compounds *(Table 4)*. β-myrcene had the highest peaks among all terpen compounds identified in sucuk samples. D-limonene, 3-carene, linalool and caryophyllene were also standing out with their high peak areas.

DISCUSSIONS

Biochemical reactions including glycolysis, proteolysis, lipolysis, autoxidation etc. play an important role in transformation of meat to fermented meat products. Volatile compounds resulted from such reactions have significant impact on characteristic flavor and consumer acceptance [16]. As a result of these reactions and components of sucuk formulation, seventy five volatile compounds were identified in experimental samples. In parallel to our findings, Kaban [7] identified ninety-two volatile compounds in sucuk belonging to different brands. In addition, forty volatile compounds were also identified in a different study on sucuk produced with same starter culture as in our study [1]. Besides, Olivares et al.[5] identified ninety five volatile compounds by using SPME in dry-fermented sausages. These differences in number and amount of volatile compounds in various

Table 3. *Ester, ketone and sulphur compounds of sucuk produced by using fat and orange fiber levels*
Tablo 3. *Farklı yağ ve portakal lif oranları kullanılarak üretilen sucukların ester, keton ve sülfürlü bileşikleri*

Compound	KI	S	Fat Level (%)			S	Fiber Level (%)			Fat Level x Fiber Level S
			10	15	20		0	2	4	
Esters										
Acetic acid ethenyl ester	639	NS	0.06±0.08a	0.06±0.06a	0.02±0.02a	NS	0.07±0.09a	0.05±0.06a	0.02±0.02a	NS
Ethyl acetate	640	NS	0.28±0.10a	0.23±0.09a	0.15±0.14a	NS	0.24±0.11a	0.22±0.07a	0.20±0.17a	NS
Butanoic acid, ethyl ester	843	NS	0.07±0.09a	0.05±0.05a	0.02±0.02a	NS	0.03±0.03a	0.03±0.02a	0.07±0.09a	NS
Ethyl lactate	867	NS	0.04±0.05a	0.06±0.05a	0.04±0.03a	NS	0.04±0.05a	0.05±0.03a	0.05±0.05a	NS
Hexanoic acid, ethyl ester	1024	NS	0.06±0.10a	0.15±0.19a	0.14±0.22a	NS	0.09±0.20a	0.08±0.15a	0.17±0.17a	NS
2,4-Hexadienoic acid, methyl ester	1077	NS	0.54±0.31a	0.52±0.32a	0.47±0.35a	NS	0.60±0.33a	0.46±0.25a	0.47±0.37a	NS
Hexanoic acid, propyl ester	1126	NS	0.43±0.13a	0.34±0.22a	0.42±0.16a	NS	0.39±0.14a	0.45±0.23a	0.34±0.13a	NS
2,4-Hexadienoic acid, ethyl ester	1154	NS	0.17±0.18a	0.18±0.16a	0.13±0.12a	NS	0.13±0.13a	0.13±0.12a	0.21±0.20a	NS
Butanoic acid, hexyl ester	1224	NS	0.25±0.11a	0.26±0.20a	0.22±0.16a	NS	0.26±0.14a	0.27±0.18a	0.20±0.16a	NS
Octanoic acid, ethyl ester	1239	NS	0.06±0.06a	0.05±0.04a	0.07±0.03a	NS	0.06±0.05a	0.05±0.04a	0.07±0.06a	NS
Ketones										
2-Butanone, 3-methyl-	637	NS	0.06±0.05a	0.03±0.03a	0.04±0.03a	NS	0.04±0.03a	0.03±0.04a	0.06±0.04a	NS
2-Butanone, 3-hydroxy-	779	NS	0.32±0.08a	0.24±0.16a	0.22±0.21a	NS	0.26±0.17a	0.26±0.17a	0.25±0.17a	NS
2-Heptanone	948	NS	0.34±0.58a	0.03±0.01a	0.03±0.02a	NS	0.06±0.05a	0.07±0.09a	0.28±0.60a	NS
6-Methyl-5-hepten-2-one	1042	NS	0.07±0.11a	0.04±0.01a	0.04±0.02a	NS	0.03±0.02a	0.03±0.02a	0.07±0.11a	NS
3,5-Octadien-2-one	1141	NS	0.12±0.11a	0.13±0.06a	0.09±0.10a	NS	0.11±0.09a	0.14±0.08a	0.09±0,11a	*
2-Nonanone	1147	NS	0.04±0.07a	0.09±0.08a	0.05±0.03a	NS	0.05±0.09a	0.08±0.05a	0.04±0.03a	NS
Sulphur compounds										
Thiirane, methyl	574	NS	0.17±0.24a	0.04±0.05a	0.04±0.07a	NS	0.11±0.14a	0.03±0.03a	0.10±0.23a	NS
Sulfide, allyl methyl	731	**	0.39±0.29a	0.10±0.08b	0.12±0.13b	*	0.15±0.15b	0.12±0.13b	0.34±0.32a	NS
1-Propene, 1-(methylthio)	762	NS	0.05±0.06a	0.06±0.06a	0.06±0.06a	NS	0.07±0.07a	0.05±0.06a	0.06±0.05a	NS
1-Propene, 3,3'-thiobis-	888	NS	0.14±0.07a	0.12±0.10a	0.09±0.05a	NS	0.14±0.10a	0.09±0.05a	0.11±0.07a	NS
Disulfide, methyl 2-propenyl	958	NS	0.12±0.09a	0.09±0.06a	0.06±0.03a	NS	0.10±0.03a	0.07±0.03a	0.10±0.10a	NS
Diallyl disulphide	1139	NS	1.27±0.40a	0.96±0.25ab	0.89±0.24b	NS	1.20±0.32a	1.01±0.43a	0.90±0.21a	NS

S: *significance,* ***NS:*** *Not Significant, * P<0.05, ** P<0.01; Results are expressed in Arbitrary Area Units (×10⁻⁶) as means of 3 replicates of each sausage;* ***KI:*** *Kovats index calculated for DB-624 capillary column (J & W scientific: 30 m, 0.25 mm id, 1.4 lm film tickness) installed on a gas chromatograph equipped with a mass selective detector*

dry-fermented sausages can be explained by the differences in formulation, processing conditions, starter culture and biochemical reactions taking place during production [1,17,18].

Aldehydes are final products of lipid oxidation and have importance in volatile profile of food products in which they are present. Because of their low perception thresholds, aldehydes can affect the aroma even in trace amounts [19]. Hexanal was one of the outstanding aldehyde compound in samples. This compound has fatty, grassy and fruity odor which is also found in nearly 300 natural sources including meat, cheese and fruits [20]. Short chain aldehydes such as acetaldehyde are mostly originated from carbohydrate metabolism. Besides, some of aldehydes shown in *Table 1* are probably formed by the autoxidation of oleic and linoleic acids [19].

Acetic acid, had the highest peak area in samples, is a colorless volatile liquid with its strong vinegar odor [20]. This volatile acid is generally produced by microbial metabolism [6]. Likewise, 4-(1-methylethyl) benzene methanol and ethanol had the highest peaks, respectively. Within these alcohols, 4-(1-methylethyl) benzene methanol has an aromatic, burning taste and intense, caraway-like odor [20]. On the other hand, ethanol is mainly produced by carbonhydrate fermentation and catabolism of amino acids, and lipids are also responsible for its production [19].

Aromatic hydrocarbons are important volatile components for dry-fermented meat products and 1-methyl-2-(1-methylethyl)- benzene had the highest peak in current study. In contrast, 1,2-dimethoxy-4-(2-propenyl)-

benzene was found by Kaban and Kaya [1] in sucuk as the most abundant aromatic hydrocarbon in terms of peak area. p-xylene and toluene which were also detected in samples could be originated from animal feed and from catabolism of phenylalanine, respectively [19].

2,4-Hexadienoic acid methyl ester and hexanoic acid propyl ester were detected in high levels in samples. 2,4-Hexadienoic acid methyl ester has a fruity, sweet, anise odor. Hexanoic acid propyl ester has an ether-like odor with a pineapple-blackberry undertone. Moreover, hexanoic acid propyl ester is naturally found in some cheeses and some fruits [20].

Ketones are formed by β-oxidation and autoxidation of free fatty acids found in fermented meat products [19]. 3,5-octadien-2-one was the only ketone compound which was affected by interaction of main variation sources (fiber and fat). This compound has a pungent, herbaceous odor and soluble in fats. It's aroma and taste threshold values are 0.15 ppm (in water) and 1.0 ppm, respectively [20]. 3-hydroxy,2-butanone was another significant ketone compound found in samples. This compound known as acetoin is a yellowish liquid with a bland, woody, yogurt odor and a fatty creamy "tub" butter taste [20]. 3-hydroxy,2-butanone is also known to be produced by fermentation of sugars by lactic acid bacteria [19]. Similar ketones were also found in commercial sucuk samples but in low proportions [7].

Diallyl disulphide was the significant sulphur compound in sucuk samples. This compound is the characteristic garlic odor [20] and sulphur compounds are probably originated from garlic. Garlic is one of the main ingredient used in sucuk production [7].

Terpenes are important share in volatile profile of sucuk. As indicated by Kaban and Kaya [1] and Kaban [7], sucuk contains higher level of terpens originated from spices. β-myrcene which is known for its pleasant, sweet, balsamic, plastic odor [20] was one of the terpenes with its higher peak. D-limonene, 3-carene and caryophyllene were also outstanding among these nineteen terpenes detected in the study. D-limonene, 3-carene and caryophyllene are some of the major compounds found in essential oil of black pepper [19]. D-limonene is known as the most important and widespread terpene and has a pleasant, lemon-like odor. 3-carene is colorless or very pale-yellow liquid oil. Linalool has a typical nice floral odor. Caryophyllene's three different isomers are found in

Table 4. *Terpene compounds of sucuk produced by using different fat and orange fiber levels*
Tablo 4. *Farklı yağ ve portakal lif oranları kullanılarak üretilen sucukların terpen bileşikleri*

Compound	KI	S	Fat Level (%)			S	Fiber Level (%)			Fat Level x Fiber Level S
			10	15	20		0	2	4	
Terpenes										
alpha-thujene	944	*	0.23±0.05b	0.34±0.04a	0.26±0.06b	NS	0.26±0.08a	0.27±0.06a	0.29±0.07a	NS
α-pinene	957	NS	0.60±0.07a	0.63±0.10a	0.61±0.13a	NS	0.64±0.09a	0.62±0.09a	0.59±0.11a	*
Camphene	970	NS	0.04±0.02a	0.05±0.01a	0.04±0.02a	NS	0.06±0.03a	0.04±0.01a	0.03±0.01a	NS
β-pinene	988	NS	0.86±0.83a	0.75±0.81a	0.77±0.79a	NS	0.83±0.88a	0.71±0.78a	0.83±0.76a	NS
β-myrcene	1005	NS	5.92±2.34a	7.05±3.07a	6.97±4.95a	NS	7.35±3.81a	6.16±4.48a	6.43±2.20a	NS
α-phellandrene	1019	NS	0.72±0.22a	0.86±0.29a	0.61±0.55a	NS	0.68±0.29a	0.92±0.45a	0.58±0.32a	NS
3-carene	1026	NS	1.82±0.18a	1.98±0.45a	2.04±0.72a	NS	2.15±0.57a	1.89±0.61a	1.79±0.13a	NS
D-limonene	1054	NS	2.35±0.82a	3.36±1.03a	3.33±1.99a	NS	2.98±1.34a	3.11±1.93a	2.94±0.98a	NS
β-phellandrene	1065	NS	0.58±0.11a	0.66±0.14a	0.58±0.37a	NS	0.59±0.25a	0.67±0.25a	0.57±0.19a	NS
o-cymene	1070	NS	0.44±0.20a	0.43±0.17a	0.43±0.32a	NS	0.52±0.23a	0.42±0.24a	0.37±0.20a	NS
α-carene	1114	NS	0.05±0.05a	0.08±0.02a	0.06±0.05a	NS	0.07±0.05a	0.06±0.04a	0.07±0.05a	NS
Linalol	1161	NS	1.10±0.19a	1.13±0.35a	0.91±0.50a	NS	1.20±0.27a	1.05±0.25a	0.88±0.49a	NS
p-cymene	1138	NS	0.21±0.04a	0.23±0.06a	0.24±0.05a	NS	0.23±0.06a	0.02±0.05a	0.23±0.05a	NS
4-terpineol	1240	NS	0.18±0.12a	0.12±0.09a	0.05±0.08a	NS	0.10±0.11a	0.11±0.09a	0.14±0.13a	NS
α-terpineol	1267	NS	0.19±0.10a	0.08±0.07a	0.13±0.13a	NS	0.13±0.12a	0.11±0.08a	0.15±0.14a	NS
Carvacrol	1428	NS	0.06±0.03a	0.06±0.03a	0.04±0.01a	NS	0.06±0.02a	0.06±0.04a	0.04±0.02a	NS
Copaene	1433	NS	0.36±0.05a	0.26±0.09b	0.35±0.10a	*	0.40±0.07a	0.28±0.07b	0.29±0.08b	NS
β-elemene	1453	NS	0.11±0.08a	0.09±0.07a	0.12±0.08a	NS	0.15±0.10a	0.10±0.06a	0.07±0.05a	NS
Caryophyllene	1495	NS	2.89±1.11a	2.39±0.90a	2.98±0.85a	*	3.56±0.77a	2.61±0.55ab	2.09±0.90b	NS

S: *significance,* ***NS:*** *Not Significant, * P<0.05; Results are expressed in Arbitrary Area Units (×10⁻⁶) as means of 3 replicates of each sausage;* ***KI:*** *Kovats index calculated for DB-624 capillary column (J & W scientific: 30 m, 0.25 mm id, 1.4 lm film tickness) installed on a gas chromatograph equipped with a mass selective detector*

nature. Furthermore, 1R-alpha-pinene, another terpene found in samples, has a characteristic odor of pine [20].

Fat is a major component in dry-fermented sausages and has an important role both in development of textural parameters and sensory properties of dry-fermented sausages. It is known to be effective in volatile compound formation in such products. But taking into consideration of sucuk samples produced in current study, effects of different fat levels did not appear well, maybe due to the short ripening time of nine days. Thus, extending the ripening time with different fat levels may reveal more information about the exact role of fat in aroma formation in sucuk. On the other hand, orange fiber had little impact on volatile compound profile of sucuk. This result may be explained by processing steps applied in orange fiber production. Because of several washing steps and pasteurization process, many important volatile compounds could be moved away from the fiber. It could be suggested that sucuk samples with orange fiber has similar volatile profile with traditional one.

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