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Influence of roasting conditions on the flavor quality of sesame seed oil

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Abstract : Sesame seeds were roasted for 30, 60, 90, and 120 min at different temperatures (100, 200, and 300℃) and extracted to investigate an adequate condition for producing the high quality sesame oil. Sesame seeds roasted at 200℃ for 90 min gave the high yield of oil. The oil contained the low content of brownish-black precipitates and exhibited an excellent organoleptic quality when judged by descriptive sensory analysis. Thirty one volatile flavor compounds, which are the largest number of volatiles among the oil samples prepared, were identified from the oil sample. The oil contained relatively high concentrations of furfurals (sweet candy-like flavor) and pyrazines (roasted-like flavor), that are considered as good contributors to sesame seed oil flavor, and low concentations of aldehydes (C5~C10) and ketones, which are considerd as bad contributors (oxidized fat-like and painty-like flavors). These results suggest that the roasting condition of 200℃ for 90 min was the best for the oil production in terms of the overall aroma and taste quality under the test conditions (Received July 13, 1993; accepted November 4, 1993).

Introduction

The roasted sesame seed (*Sesamin indicum, L.*) has a typical sweet and pleasant aroma. The resulting sesame oil extracted from the roasted sesame seeds by the mechanical press also has the favorite flavors. Traditionally both the roasted sesame seed and oil have been used as condiments in many oriental foods because of their good matching flavors with other oriental spices.

Usually, the roasting temperature and time for extracting the sesame seed oil depends on processors. The home-made oil was prepared with the sesame seeds roasted at very high temperature has a strong roasted flavor, nearly almost burning flavor. As results, a lot of burnt-browning pigments possessing bitter taste are formed and precipitated during storage which have been considered as mutagens/carcinogens.[1-6] During roasting sesame seeds, many flavor compounds are also formed by Maillard reaction of amino acids and carbohydrates, autoxidation of lipids, and the cyclization of many breakdown products. Such compounds were found to be aldehydes, ketones, alcohols, and pyrazines.[7] However, none of these compounds could be defined as a characteristic sesame seed oil flavor compound. It has been known that aldehydes, ketones, and alcohols are formed from the oxidation of linoleic acid,[7-9] which is a major fatty acid of sesame seed. Aldehydes, ketones, furan, pyrrole, pyridine,

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and pyrazine were produced from the Maillard reaction by heating foods containing reducing sugar and amino acids.[10-11] Sesame seeds contained 3～4 % of each D-glucose and D-fructose,[12] 0.2% of sucrose,[12] and 20～30% of protein,[13-15] which might be involved in the formation of characteristic flavor compounds of sesame seed oils.

Production of the volatile compounds by the Maillard reaction was controlled by heating temperature,[16] time,[16] and pH of reaction medium.[17] Thus, roasting temperature and time of sesame seeds should be important factors for the production of flavor compounds in sesame seed oil. In this study, we investigated the appropriate roasting temperature and time for the formation of the best flavor profiles and for the less formation of browning pigment with high yield of sesame seed oil. The investigation was carried out by analysing volatile flavor compounds, by descriptive sensory analysis of organoleptic quality of the oil, and by determining the yields and contents of precipitates in the sesame seed oil.

Materials and Methods

Materials

Sesame seeds (*Sesame indicum L.* cv. *Suwon 21*) cultivated in Kyeong-nam, Korea in 1991 were obtained. The seed sample was composed of 7.6% moisture, 20.4% crude protein, 49.9% crude oil, 16.9 % carbohydrate, and 5.2% ash which were analyzed by the method of AOAC.[18]

Preparation of sesame seed oil

After washing and drying by air, 3 Kg of sesame seed was roasted for 30, 60, 90, and 120 min on a temperature-controlled Electric heating pan at individual roasting temperature, 100, 200, and 300℃. About one forth of each sample was successively removed during roasting for 30 min, 60 min, 90 min, and 120 min.

The oil from the roasted seeds was extracted mechanically by the electric oil extractor (Han Il Gaedoli, Shin Han Il Electric Co., Seoul, Korea). The oil from the raw sesame seeds was extracted by the A.O.A.C. method[18] using the Soxhlet apparatus for comparison.

Determination of yields and precipitates of sesame seed oils

Yield (%) of oil was determined by substracting the weight of residue from that of the sesame seed used. Five replicated experiments were performed to obtain an average value of yields.

Three hundred mililiters of freshly-prepared sesame seed oil was left for 24 hr at room temperature to determine the amount of precipitates. The precipitate was filtered through the Watman No. 4 paper. The residues on the filter paper were washed three times with 30 m*l* of diethyl ether to remove the residual oil and dried at room temperature for 3 hr. Weight of the precipitate was calculated by subtracting the weight of the filter paper from the total weight.

Analysis of volatile flavor compounds

Volatile compounds of freshly-prepared sesame seed oil were quantitatively analyzed according to the method of Dunn and Lindsay.[19] The oil sample (50 g) containing internal standard (10 μg 4-decanol in ethanol; Aldrich Chemical Co., Milwaukee, WI, USA) were mixed with 250 m*l* of saturated sodium

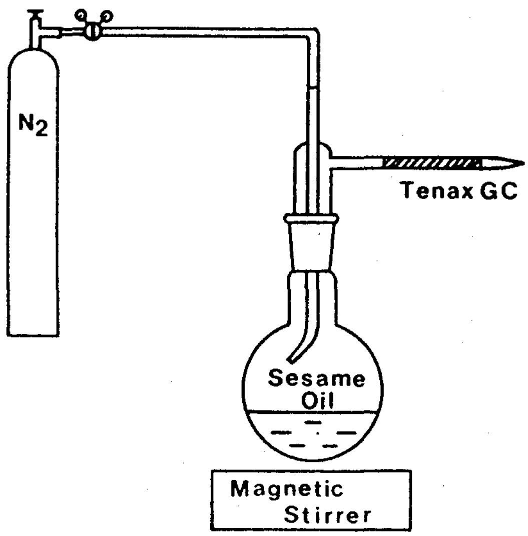

Fig. 1. An apparatus used for the collection of volatile compounds.

chloride solution in a 500 ml round bottom flask. The mixture were purged for 3 hr with nitrogen (300 ml/min) onto Tenax GC® (60~80 mesh; ENKA N. V., Holland) at room temperature (Fig. 1). The volatile compounds were eluted from the Tenax GC trap with redistilled diethyl ether into the concentrate tube (Laboratory Research Company, Los Angeles, CA, USA), and then concentrated under a stream of nitrogen to about 10 μl for GC analysis. The volatile flavor compounds in the diethyl ether concentrate (1 μl) were separated on a HP-5 capillary column (25 m×0.2 mm, i.d., 0.2 μm coating thickness; Hewlett Packard, Inc., Orlando, FL, USA) with a HP 5890 gas chromatograph (Hewlett Packard, Inc., Orlando, FL, USA) connected with a HP 5970 mass spectrometry. Nitrogen was used as carrier (2 ml/min) and make-up gases (30 ml/min). The oven temperature was programmed from 70℃ to 200℃ at 2℃/min after holding 3 min at 70℃. The injection port and detector temperature was set up at 270℃ and resulting voltage was at 1800.

The identification of volatile compounds was carried out by GC retention indices (I_E)[20] and the mass spectral data matching with Willey, REVE, and REVF NBS library. The peaks that could not be identified with these library were assigned by interpretation of fragmentation pattern and by sniffing the effluent carrier gas from the end of capillary column (HP-5) during GC analysis of the extract. Aromas were matched with GC peaks using timed notations marked on a integrator which was operated under conditions identical to analytical runs.

The concentration of each volatile compound was calculated by the area of each peak comparing with the area of internal standard assuming the response factor 1 using the following equation: the concentration (μg/g)=[the peak area of compound × weight (μg) of IS]/[the peak area of IS]

Descriptive sensory analysis of sesame seed oil

Descriptive sensory analysis was carried out by the method described by Moskowitz et al.[21] Thirty four sensory panel judges of 20 years or older were recruited with no attempt made to classify panelists by gender or age. The samples were tasted with white rice cake, and total aroma quality and taste quality were scored on 5 scale (0, weak or bad; 5, strong or good). The flavor descriptions were cany-like, roasted sesame seed like, painty, green, burnt, bitter, sweet, tart, and Gamchil (umami).

Results and Discussion

Fig. 2 shows the yields of oils from the three sesame seed samples roasted at 100℃, 200℃, and 300℃ for 30 min, 60 min and 120 min. The sesame seed roasted for 60 min at 100℃ or 200℃ yielded less oil substantially as compared with other treatments that did not show significant differences in

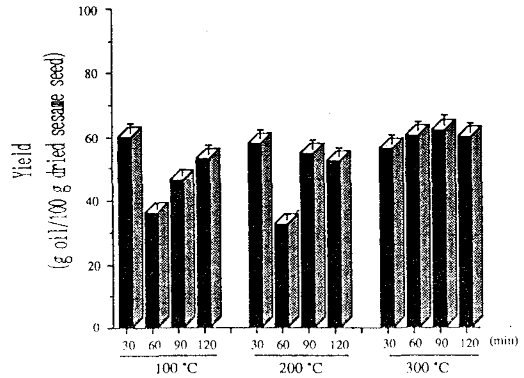

Fig. 2. Yields of sesame seed oils from sesame seeds roasted at different conditions.

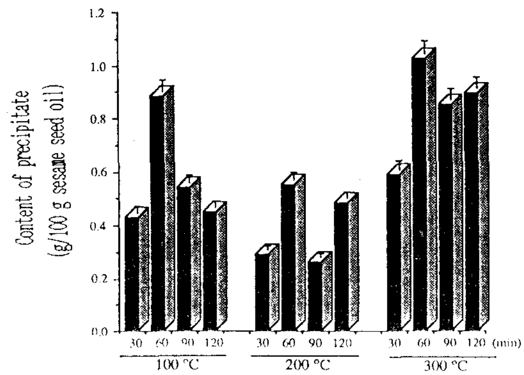

Fig. 3. Precipitate contents of the sesame seed oil from the sesame seeds roasted at different conditions.

the quantity of oils. Fig. 3 shows the amount of precipitates in the oils from the sesame seeds treated with the conditions shown in Fig. 2. In contrast to the yield, the levels of the precipitates in the treatment groups for 60 min at 100, 200 or 300℃ were significantly higher than those in other treatment groups. Around 100℃ is the suitable temperature for the browning reaction[22] and 300℃ is the temperature for the pyrolysis and polymerization that led to the formation of brownish-black precipitates. Hence, to give high yields and less precipitates under the test conditions, 200℃ seemed to be the optimum temperature for the roasting sesame seeds.

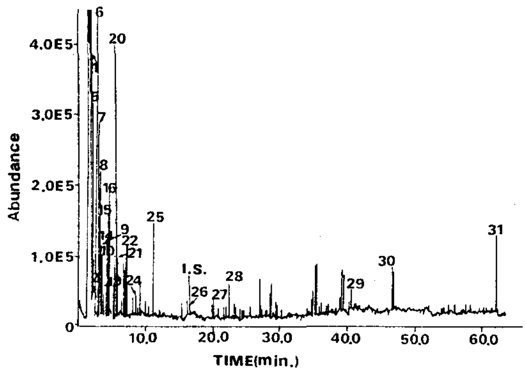

Fig. 4. Total ion chromatogram (TIC) of volatile compounds of oil from sesame seeds roasted at 200℃ for 90 min. Column, HP-5 crosslinked 5% phenyl methyl sillicone capillary (25 m×0.25 mm); temperature, 70~200℃ programmed at 2℃/min.

A typical total ion chromatogram (TIC) of the volatile compounds in the oil from sesame seeds roasted at 200℃ for 90 min, where the largest number of volatile flavor compounds (31 compounds identified) was appeared as shown in Fig. 4.

The flavor compounds formed during roasting sesame seeds for 60, 90, and 120 min at 100℃ are listed in Table 1. Alkanes, aldehydes, alcohols, arenes, and pyrazines were identified as major compounds and their concentrations were generally increased with extending the roasting time. Alkanes and aldehydes are the breakdown products of polyunsaturated fatty acids and contribute to the green, oily, and oxidized fat-like flavor.[23] When roasting

Table 1. Concentrations of volatile compounds identified in the oil from sesame seeds roasted at 100℃

Compound[1]	TIC Peak No.[2]	Roasting Time (min.)			
		30	60	90	120
			μg/g		
Pentanal	1	0.65	1.10	1.34	6.38
1-Pentanol	2	0.24	0.35	0.58	1.20
Methylbenzene	3	–[3]	0.63	0.63	2.59
Hexanal	5	1.06	2.62	2.84	2.39
Methoxymethyl acetate	6	4.04	3.89	3.58	6.78
Methylpyrazine	7	0.08	0.40	0.81	0.92
2-Furfural	8	0.11	0.14	0.13	0.15
Furfuryl alcohol	9	0.04	0.06	0.11	0.10
1,2-Dimethylbenzene	11	0.01	0.03	0.02	0.01
Decanal	15	0.08	0.10	0.08	0.11
2,6-Dimethylpyrazine	16	–	0.05	0.11	0.10
2-Heptenal	19	0.01	0.04	0.02	0.06
2-Ethyl-4-pentenal	20	0.26	0.37	0.71	1.60
2,4-Nonadienal	22	–	0.03	0.07	0.04
An aldehyde	25	–	0.04	0.09	0.09
2-Methylheptadecane	26	–	0.02	0.13	0.10

[1]Identified by GC-MS and retention time.
[2]Peak numbers are shown in Fig. 4.
[3]Not detected.

time was extented, the concentrations of pentanal (painty), 2-ethyl-4-pentenal (painty), and hexanal (green) were increased. An aldehyde (peak no. 25; oxidized fat-like) and 2,4-nonadienal (cucumber-like) appeared in the sample roasted at 1000C for longer than 30 min. 2-Heptenal (oily, oxidized fat-like) known as an oxidized product of oleic and linoleic acids[23-25] are the major fatty acids of sesame seeds. The formation of 2,4-decadienal from unsaturated fatty acids in beef tallow has been explained via the mechanism of double bond hydration in the presence of water or the retro-aldol condensation of 2-octenal and ethanal derived from unsaturated fatty acids.[24] Hence the 2,4-nonadienal in the sesame oil was formed by chemical reaction of 2-heptenal with ethanal via the same mechanisms as 2,4-decadienal formation in the beef tallow. The concentrations of methylpyrazine and 2,6-dimethylpyrazine were increased with the longer roasting time. Methylpyrazine and 2,6-dimethylpyrazine with a roasted aroma and 2-furfural with sweet candy-like aroma could be formed through the Maillard reaction of amino acids with reducing sugar substrates in sesame seeds. These compounds may contribute to the sweet and roasted flavor notes of sesame seed oil, which could be an important

Table 2. Concentrations of volatile compounds identified in the oil from sesame seeds roasted at 200℃

Compound[1]	TIC Peak No.[2]	Roasting Time (min.)			
		30	60	90	120
			μg/g		
Pentanal	1	2.48	2.45	3.14	4.19
1-Pentanol	2	0.40	0.43	0.41	1.97
Methylbenzene	3	1.68	1.72	1.70	3.89
4-Methyl-1,3-dioxane	4	—[3]	—	—	0.49
Hexanal	5	2.34	4.67	5.05	8.26
Methoxymethyl acetate	6	5.85	6.62	6.45	8.86
Methylpyrazine	7	0.55	0.60	0.71	0.60
2-Furfural	8	0.42	0.63	0.85	0.81
Furfuryl alcohol	9	0.22	0.28	0.14	0.51
2,4-Dimethylcyclopentanone	10	—	—	0.26	0.09
1,2-Dimethylbenzene	11	0.03	0.08	0.04	0.18
Ethylbenzene	12	—	—	—	0.32
2-Heptanone	13	—	—	0.15	0.28
1,3,5,7-Cyclooctatetraene	14	—	—	0.27	0.42
Decanal	15	0.16	0.34	0.59	0.86
2,6-Dimethylpyrazine	16	0.40	0.63	0.68	0.39
2-Heptenal	19	0.06	0.06	0.07	—
2-Ethyl-4-pentenal	20	0.55	1.08	1.40	1.76
5-Methyl-2-furfural	21	—	0.21	0.23	—
2,4-Nonadienal	22	0.28	0.26	0.44	0.58
Benzeneacetaldehyde	24	—	0.10	0.21	0.16
An aldehyde	25	0.41	0.39	0.68	0.81
2-Methylheptadecane	26	0.13	0.11	0.14	—
1-Undecene	27	—	—	0.11	—
2-Propyl-1-heptanol	28	—	—	0.41	0.30
1-Heptacosanol	29	—	—	0.14	0.07
Amyl benzoate	30	—	—	0.42	0.23
Nonacosane	31	—	0.08	2.95	0.07

[1]Identified by GC-MS and retention time.
[2]Peak numbers are shown in Fig. 4.
[3]Not detected.

factor that people prefer sesame seed oils to other vegetable oils. Methoxymethyl acetate were first found in this study from the sesame seed oil, however, its origin and flavor characteristic are not clear. Furfuryl alcohol found in the sesame oil samples has been previously reported,[7] which was considered as a compound giving a typical sesame seed oil flavor, however, we can not agree with this aspect because other foods containing this compound do not have the typical flavor of sesame seed oil. Instead, we suspect the characteristic flavor of sesame seed oil might be derived from compounds like sesamol and sesamin typically present in sesame seeds. Further investigation about the breakdown products of sesamol and sesamin is being progressed.

The volatile flavor compounds of the sesame seed oil from sesame seeds roasted at 200℃ for 30, 60, 90, and 120 min are listed in Table 2. The sesame seed oil prepared by 200℃ for 90 min contained the largest number of flavor compounds among the sesame seed oil samples prepared in this study (Fig. 4). In addition to the flavor compounds identified from the sample roasted at 100℃, 4-methyl-1,3-dioxane, 2,4-dimethylcyclopentanone, ethylbenzene, 2-heptanone, 1,3,5,7-cyclooctatetraene, 5-methyl-2-furfural, 1-undecene, 2-propyl-1-heptanol, 1-heptacosanol, amyl benzoate, and nonacosane were identified from the oil sample prepared at 200℃. These compounds were not detected in the sample roasted

Table 3. Concentrations of volatile compounds identified in the oil fromsesame seeds roasted at 300℃

Compound[1]	TIC Peak No.[2]	Roasting Time (min.)			
		30	60	90	120
			μg/g		
Pentanal	1	6.39	5.25	3.85	3.96
1-Pentanol	2	1.76	1.74	1.27	2.11
Methylbenzene	3	1.89	1.47	1.55	2.70
4-Methyl-1,3-dioxane	4	0.34	0.42	–[3]	–
Hexanal	5	7.65	6.93	7.29	6.59
Methoxymethyl acetate	6	10.25	10.16	10.33	12.26
Methylpyrazine	7	0.55	0.41	0.14	0.07
2-Furfural	8	0.36	0.38	0.38	0.77
Furfuryl alcohol	9	0.19	0.18	0.14	0.21
2,4-Dimethylcyclopentanone	10	0.05	0.08	–	–
Ethylbenzene	12	0.08	0.10	–	–
2-Heptanone	13	0.23	–	–	–
1,3,5,7-Cyclooctatetraene	14	0.26	0.13	0.18	–
Decanal	15	0.45	0.38	0.20	0.23
2,6-Dimethylpyrazine	16	0.46	0.42	0.27	0.40
Ethylpyrazine	17	0.08	0.11	0.09	0.04
2,3-Dimethylpyrazine	18	–	0.10	0.04	0.04
2-Heptenal	19	0.88	0.90	0.72	0.95
2-Ethyl-4-pentenal	20	1.36	0.87	0.50	–
2,4-Nonadienal	22	0.42	–	–	–
Pyrrole	23	–	0.06	0.03	–
Benzeneacetaldehyde	24	0.24	0.04	–	–
An aldehyde	25	0.83	–	0.02	–
2-Methylheptadecane	26	0.27	0.06	0.03	–
2-Propyl-1-heptanol	28	–	0.08	0.18	0.04

[1]Identified by GC-MS and retention time.
[2]Peak numbers are shown in Fig. 4.
[3]Not detected.

Table 4. Mean score from descriptive sensory analysis of oil obtained from roasted sesame seeds roasted at 200℃ for different time

Attribute	Roasting time (min.) 30	60	90	120
Aroma				
Candy	0.881	1.22	1.83	1.30
Roasted sesame like	1.75	1.41	2.14	2.20
Painty	0.97	1.23	0.44	0.96
Green	0.72	0.79	0.46	0.75
Burnt	0.73	1.22	0.88	0.91
Taste				
Bitter	0.70	1.47	0.70	0.61
Sweet	0.64	0.72	0.99	0.97
Tart	1.23	1.35	1.01	1.88
Gamchil[2]	0.68	0.45	0.97	1.10
Total aroma quality	0.77	1.16	1.67	1.29
Total taste quality	0.70	0.70	1.08	1.11

[1]Data (n=34) on profiling from Moskowits.[21)]
Using the checklist method and a 0~5 point category rating scale (0; weak or bad, 5; strong or good). Statistics: Tuckey's w (α=0.05) for candy, 0.45; roast sesame-like, 0.31; painty, 0.62; green, 0.35; burnt, 0.41; bitter, 0.60; sweet, 0.20; tart, 0.43; Gamchil, 0.38; total aroma, 0.40; and total taste, 0.21.
[2]Korean description for "Umami".

for 30 and 60 min. Alcohols and ketones are derived from alkyl radical of the break down products of unsaturated fatty acid peroxides.[25)] 2-Heptanone has painty odor contributing to the oxidized-fat like odor of oil samples. The origin and flavor characters of 2,4-dimethylcyclopentanone in sesame seed oil were not exactly defined, but the cyclopentanone is derived from the Maillard reaction of lactose with casein and cyclopenten-1-one was found from the Maillard reaction mixture of D,L-alanine and D-glucose. Therefore, it is possible that 2,4-dimethylcyclopentanone is formed from the Maillard browning in the sesame seed during heating process. 1-Undecene found in the oil sample roasted at 200℃ for 90 min was not found in any other samples analyzed in this study. This compound has been found in the cotton seed oil as thermal degradation product of unsaturated fatty acid.[8)] It has been reported that aromatic hydrocarbons including methylbenzene and ethyl benzene can be derived from thermal degradation of carbohydrate[26)] and oil.[8)] 5-Methyl furfural (sweet candy-like flavor) appeared in the sample roasted at 200℃ for 60 min and 90 min, but not in the samples roasted at other conditions used in this study. This may be because 5-methyl furfural first transiently formed during sugar caramelization and then transformed to other volatile compounds as Shibamoto suggested.[27)] This compound has sweet caramel-like odor, and is well known product of sugar caramelization,[27-30)] and have been found in meats,[27)] cooked eggs,[31)] roasted peanuts,[32)] and roasted sesame seeds.[7)]

In addition to the flavor compounds found in the 100℃ and 200℃ oils , ethylpyrazine, 2,3-dimethylpyrazine, and pyrrole were detected, but 1-undecene, 5-methyl 2-furfural, 1-heptacosanol, amyl benzoate, and nonacosane were disappeared in the oil sample from the sesame seeds roasted at 300℃ (Table 3). The oil sample from the sesame seeds roasted at 300℃ has higher amounts of pentanal, 1-pentanol, and methoxymethyl acetate were present than from other samples. Methylpyrazine and 2,6-dimethylpyrazine starting to be detected from 100℃ treatment were increased in their concentrations until roasted up to 200℃ for 90 min; thereafter decreased. Pyrazins have typically pleasant roasty flavor notes and have been found from many heat-treated foods.[31-36)] The formation of pyrazins has been proposed by several researchers: nonenzymatic browning reaction of amino acids and carbohydrate,[37)] of lactose and casein,[29)] of D-glucose and ammonia.[38)] More specific mechanism proposed by Koehler and Odell[39)] was through the condensation of a-amino carbonyl fragments, which is formed from amino acids and carbonyl compounds-glyoxal and pyruvaldehyde. Several kinds of a-amino carbonyl compounds can be produced from the reaction of carbonyl compounds and amino acids, and pyrazines were determined from a-amino carbonyl fragment.[40)] Therefore, methylpyrazine in the heated sesame seed oil could be formed through the condensation of 2-amino ethanal and 2-amino-3-hydroxy propanal. Shibamoto and Bernard[38)] reported that optimum temperature for the production of pyrazines were 120℃, and the pyrazine contents were decreased at the temperature higher than 120℃. Koehler and Odell[39] reported that pyrazines did not form at temperature lower than 100℃ and the optimum temperature was 150℃. Given the results of this study and previous reports, the optimum temperature for the formation of pyrazines could be affected by other constituents of reaction systems, and thus, the optimum temperature in sesame seed seem to be higher than in the model system (120∼150℃) due to, in part, the higher oil content of sesame seeds.

Roasting sesame seed at 200℃ for 90 min resulted in acceptable high yield of oil, low contents of precipitates, and large numbers of flavor compounds. Hence, the descriptive sensory analysis was carried out for the oil samples obtained from the sesame seeds roasted at 200℃ for 30, 60, 90, and 120 min. The oil obtained from the sesame seeds roasted for 90 min gave high score for the total aroma quality and taste quality (Table 4). This may be due to the facts that low scores of unfavorable flavor notes (painty, green, burnt, bitter, tart) and high scores of favorable flavor notes (roasted aroma-like, sweet, Gamchil) of this oil sample.

In conclusion, the oils from the sesame seeds roasted at 200℃ showed low contents of brownish-black precipitate with relatively high yield. Roasting sesame seeds at 200℃ for 90 min produced the largest number of volatiles among the oil sample prepared in this study. Among thirty one volatile flavor compounds identified from the oil, pyrazines and furfurals contribute to the roasted and sweet flavor notes of sesame seed oil considered as good flavor notes. Aldehydes (C5∼C10) and ketones seem to contribute to the painty, oily, and oxidized fat-like flavor notes of the oil. Organoleptic quality of sesame seed oil judging by descriptive sensory analysis was good when the oil was extracted from the sesame seeds roasted at 200℃ for 90 min in terms of the overall aroma and taste quality.

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참깨 복음조건이 참기름의 향미에 미치는 영향

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초록 : 참깨를 100, 200, 300℃ 에서 각각 30, 60, 90, 120분 동안 볶은 다음 짜낸 참기름의 수율과 갈색침전물의 생성량을 조사한 결과, 200℃ 에서 90분간 볶은 경우가 기름의 수율이 높고 갈색침전물함량이 낮았다. 관능검사에의한 참기름의 향과 맛을 평가하였을 때도 200℃ 에서 90분간 처리구가 가장 좋게 나타났으며, 가장 많은 수의(31 화합물) 휘발성향기성분이 분리, 동정되었다. 이 시료에는 다른 처리구에 비해 furfurals(달콤한 사탕냄새)과 pyrazines (고소한 냄새)의 함량이 높은 반면, 기름의 산화취를 내는 화합물인 aldehydes(C5~C10)와 ketones의 함량이 낮았다. 따라서 향기와 냄새 좋은 참기름을 얻기 위해서는 참깨를 200℃ 에서 90분간 볶는 것이 좋을 것으로 사료된다.