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Comparison of Volatile Compounds in Sesame Oil and Sesame Cake Extract

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Abstract

Sesame (*Sesamum indicum*) is one of the most important oilseed crops in the world due to its high level of lipid content and health benefits. Its seeds have high amounts of fat, protein, mineral, phenolic and volatile compounds. In addition, its oil, cake and shell also contain a high proportion of these components. Sesame cake is a by-product of the edible oil industry which could be recovered and used as a value-added product. In this study, it was aimed to compare volatile compounds in cold press sesame oil and its cake extract. The volatile compounds were extracted using purge and trap extraction and identified by gas chromatography–mass spectrometry (GC–MS). The main volatile compounds were composed of aldehydes, alcohols, terpenes, pyrazines, ketones, furans and lactones. These compounds were identified as the dominant volatile groups and their amounts were significantly lesser in sesame cake samples than the sesame oil. Among these volatiles, hexanal was found in high levels in sesame oil while it was absent in the cake extract.

Keywords: Sesamum indicum, sesame oil, aroma compounds, GC-MS volatile

1. INTRODUCTION

Sesame (*Sesamum indicum)* is an oil bearing plant that contains %50-60 oil and %25 protein in its seeds (Dong et al., 2012; Li et al.,2016). This plant, whose homeland is thought to be Asia or East Africa, has been cultivated since ancient times. Today, although the sesame production is widespread, the leading sesame producers are Sudan, Venezuela, India, Nigeria, China, Mexico and Burma. In Turkey, sesame is grown mainly in Southeast Anatolia, Mediterranean, Aegean and Marmara regions (Arslan et al., 2018). World sesame production is approximately 3.5 million tons per year. Sudan is the country that produces the most sesame seeds (981000 tons) in the world. In Turkey sesame oil production is 33600 tons in 2018 (FAOSTAT, 2018). Sesame seeds and oils are rich in health beneficial components. Sesame seeds contain high amounts of oil, protein, mineral substances, phenolic compounds and volatile compounds. In general, oil can be obtained from sesame in two different ways as from roasted or unroasted seeds. To preserve the beneficial and delicate compounds like volatiles in seeds, cold press is generally preferred in obtaining the oil. Aroma is one of the most important criteria that determines the quality and consumer acceptability of a food product. The overall aroma of sesame oil significantly depends on the roasting conditions, oil extraction conditions, and the changes in the relative concentrations of the volatiles. The aroma profile of sesame oil is closely related to its manufacturing processes as mentioned previously (Tamura et al., 2011; Dong et al., 2012). Applied processes like roasting in obtaining sesame oil were reported to have a favorable effect on the volatile compounds such as pyrazines, furans and other thermally driven compounds (Shimoda et al., 1997). Dong et al. (2012), investigated the roasting effect on the volatiles of sesame oil and reported that the process had an increasing effect on pyrazines, furans and sesamol compounds.

There is limited information in the literature on comparison of volatile compounds between cold press sesame oil and sesame cake extract. Therefore in this study, it was aimed to compare volatile compounds in cold press sesame oil and its cake extract by the application of purge and trap extraction and GC-MS characterization.

2. MATERIALS AND METHODS

2.1. Materials

Cold pressed sesame oil and sesame cake samples were purchased from a local producer in Adana, Turkey. The standard aroma compounds were purchased from Sigma-Aldrich (Steinheim, Germany). Dichloromethane, was supplied by Merck (Darmstadt, Germany). All chemicals and solvents used in this study were of analytical and chromatography grade.

2.2. Methods

2.2.1. Extraction and Analysis of Volatile Compounds

The aroma compounds of sesame oil and its cake were determined by purge and trap (PT) method. The PT extraction process consisted of a source of nitrogen controlled by a flow-meter (LZT 4-M, Union-Tek Instrument, China). The needle of the source of N_2 and the cartridge were installed through the septum of the vial containing sample to purge and trap the aroma compounds. The temperature of the vial sample was controlled by a thermostat, 3 ml of sample transferred into a 20 ml vial, then the sample was pre-incubated at the extraction temperature (60°C) for 10 min. and incubated for 90 min. under the N_2 flow. The details of the extraction was performed according to Sonmezdag et al., 2018.

2.2.2. GC-MS analysis of volatile compounds

The GC system consisted of an Agilent 6890 equipped with a flame ionization detector (FID), and an Agilent 5973N – mass selective detector (MSD). Aroma compounds were separated on a DB-Wax (30 m x 0.25 mm, 0.5 μm thickness; J&W Scientific, Folsom, CA). A total of 3 μL of extract was injected each time in pulsed splitless (40 psi; 0.5 min) mode. The injector and FID were set at 270 and 280 °C, respectively. The flow rate of carrier gas (helium) was 1.5 mL/min. The oven temperatures was first increased from 50 to 200 °C at a rate of 5 °C/min and then to 260 °C at 8 °C/min with a final hold at 260 °C for 5 min. The mass detector was operated in scan mode, with electronic impact ionization energy of 70 eV. The GC–MS interface and ionization source temperatures were set at 250 and 180 °C, respectively. Identification and quantification were performed in full scan mode scanning a mass range of m/z 30–300 at 2.0 scan/s. The compounds were identified by comparing their mass spectra output with those in Wiley 9 and NIST 11 mass spectral data libraries and an in-house library created from previous laboratory studies using similarity searching of MS fragmentation (Sevindik et al., 2020). Compounds were taken into account if they had a similarity match of at least 80% with the MS fragmentation in the databases. The chromatogram obtained was analysed, and each peak was checked by determining the per cent area on the chromatogram, the retention time, the spectrum and the base peak and then referring to the characteristic mass spectra of compounds listed on the National Institute of Standards and Technologies (NIST) using the software of Agilent ChemStation. Each sample was analysed in triplicate. Linear Retention Indices (LRI) was calculated Linear retention index calculated on DB-WAX capillary column.

3. RESULTS AND DISCUSSION

3.1. Volatile composition of samples

It is very important to choose the most suitable method for the determination of aroma compounds. Purge and trap extraction method gave the best results among the another tried-and-tested methods (liquid-liquid and solid phase extractions). A total of 23 volatile compounds were identified in sesame oil and cake samples. The main volatile compounds were composed of aldehydes, alcohols, terpenes, ketones, furans and lactones in the samples. The results (μg/l) of the GC-MS analyses of triplicate extractions were given in Table 1. All of the volatile groups except pyrazines were found to have bigger peak areas in sesame oil samples than the cakes. Aldehydes, the main significant components in flavor of oils (Xu-Yan et al., 2012; Zhao et al.,2013) were only found in sesame oils except nonanal. The major aldehyde compounds in sesame oil include pentenal, hexanal, 2-methyl-propanal, nonanal and 2-methyl-butanal. Hexanal was found in high level (255,1 µg/l) in sesame oil while it was not found in cake extract. In some studies, it was reported that hexanal is one of the important compounds among the aldehydes in sesame samples (Zhao et al., 2013; Hou et al.,2019). Other aldehydes can not be detected in cake also, except nonanal. But content of nonanal decreased in cake. Another important volatile group is the alcohols. Results show that the concentration of total alcohols were higher in sesame oil. 1-Pentanol, 1-hexanol, 1-heptanol and 1-octanol were identified in sesame oil while they can not be detected in sesame cake. 2-Ethyl-1-hexanol, 3-Penten-2-ol and 3-hexanol were detected in both samples. In addition 3-hexanol had the highest area among alcohols. Pyrazines were not detected in oil, but were detected in cake. Pyrazines are likely formed during the Maillard reaction between sugar, proteins, lipids and their lysates (Maga,2002). In literature, pyrazines, pyrroles, thiazoles, thiophenes, and furans have been identified as the major volatile compounds responsible for the aroma of sesame oil (Takei, 1988; Schieberle, 1996; Xu-Yan et al.,2012; Hou et al.,2019). Technological processes such as roasting temperature and duration and the method of extraction of the sesame oil have significant effects on the nature of the aroma. In this study, oil was obtained by cold pressing of raw sesames. Therefore, pyrazines were not detected in the oil, only 2,6-dimethyl-pyrazine and 2,5-dimethyl-pyrazine were detected at low concentrations in cake. A total of three different organic acids were found in the oil and cake samples examined and it was observed that acetic acid had the abundance. Acetic acid is reported to be a common volatile compound in oils (Zhao et al., 2013). In sesame oil, acetic acid had the highest concentration (61,4µg/l) among acids, followed by hexanoic acid and octanoic acid. Other compounds detected in the oil but not in the cake are D-limonene, 2-pentylfuran, dimethyl sulfoxide and 5-ethyl-2(5H)-furanone. In addition *p*-cymene was identified in both samples. As a result, when the cold pressed sesame oil and cake were compared in terms of volatiles, it was determined that the aroma compounds were higher in the oil. It can be concluded that the cold pressing preserved most of the volatiles and prevented their remaining in the cake.

Table 1. Aroma composition of sesame oil and sesame cake samples

No	LRI	Compounds	SO	SC
1	983	Pentenal	193,8	N
2	1053	Hexanal	255,1	N
3	1204	D-Limonene	15,6	N
4	1230	1-Pentanol	18,5	N
5	1248	*p*-Cymene	94,9	35,6
6	1245	2-Pentylfuran	23,9	N
7	1254	2-Methyl butanal	11	N
8	1328	3-penten-2-ol	169,9	113,1
9	1350	1-Hexanol	114,1	N
10	1378	3-Hexanol	251,1	225,6
11	1382	Nonanal	67,4	27,7
12	1388	2-Hexanol	27,7	25,2
13	1413	Acetic acid	61,4	N
14	1498	1-Heptanol	4,4	N
15	1532	2-Ethyl-1-hexanol	97,4	84,2
16	1554	1-Octanol	11,9	N
17	1675	Dimethyl sulfoxide	23,9	N
18	1802	2-Methyl-propanal	4,8	N
19	1810	Hexanoic acid	46,7	14,5N
20	2072	5-Ethyl-2(5H)-furanone	9,1	N
21	2073	2,6-Dimethyl- pyrazine	N	23,7
22	2075	2,5-Dimethyl-pyrazine	N	17,5
23	2083	Octanoic acid	58,4	15,2

**LRI: Linear retention index calculated on DB-WAX capillary column; N: Not found; The results are the means of amounts (µg/l) obtained from 3 different injections.*
***SO: Sesame oil; SC: Sesame cake*

4. CONCLUSIONS

It was aimed to compare volatile compounds in cold pressed sesame oil and its cake extract. A total of 23 aroma compounds with the majority of aldehydes and alcohols were identified in samples. Hexanal was found dominantly in oil samples and was not detected in the cake. In the light of the findings, it was determined that the amount of volatile compounds in sesame oil was higher than the cake. Thus, cold-press can be recommended in obtaining the oil while preserving the volatiles. The resulting data might be useful for the pressing process, improvement of product flavor and increase of consumer acceptability.

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