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# Effects of the absorbent types on changes in benzo[a]pyrene and volatile compounds in sesame oil

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## Abstract

The effects of different absorbent types on changes in benzo[a]pyrene (BaP) and volatiles in sesame oil during filtration processes were investigated using gas chromatography–mass spectrometry analysis. The BaP content was greatly reduced after filtration using powdered activated carbons (activated carbon made from peat: PP, activated carbon made from coconut shell: PC, activated carbon made from wood: PW) in comparison to granular activated carbons (activated carbon made from coconut shell: GC, activated carbon made from wood: GW). The BaP content in sesame oil was also considerably reduced when filtrated with a PW–acid clay mixture in comparison to PW–kaolin and PW–celite mixtures. Most volatile compounds were also greatly reduced after filtration using powdered activated carbons (PP, PC, and PW) in comparison to granular activated carbons (GC and GW). These results might be due to the relatively ionic structure on the surface and internal surface area of absorbent pores.

**Keywords:** Absorbents, Activated carbon, Benzo[a]pyrene, Sesame oil, Volatile compounds

## Introduction

Sesame oil obtained from roasted sesame seeds is widely used as a seasoning ingredient, salad oil, and cooking oil in various Asian countries due to its distinctive flavor and pleasant taste [1]. Sesame oil also has a long shelf life, high oxidative stability, and beneficial health functions such as anti-inflammation and antimutagenic activity [2, 3].

The manufacturing processes for sesame oil involve the selection, cleaning, roasting, grinding, cooking, and pressing of sesame seeds, and additional processes such as refining and filtering [4, 5]. The roasting process is a particularly important step in sesame oil production as its flavor characteristics and quality could be influenced by the conditions used, such as temperature and time [6]. The roasting process at a high temperature (220–260 °C)

can lead to the generation of benzo[a]pyrene (BaP), which can be a key harmful compound due to its carcinogenicity and mutagenicity, as well as other various volatile compounds [7, 8].

Recently, several studies have been conducted to reduce BaP in sesame oil using absorbents during the refining and filtration processes [9, 10]. Various adsorbents, such as activated carbons, clay materials, agricultural wastes, and siliceous materials, were applied to remove the hazardous compounds [11]. Choi et al. [10] reported that activated carbons were more efficient in removing the BaP in sesame oil compared with other adsorbents such as kaolin, perlite, acid clay, and celite. Activated carbons had the highest absorption capacity regarding polycyclic aromatic hydrocarbons (PAHs) such as BaP, due to their well-developed porous properties and wide range of polarity derived from functional groups on the surface of activated carbons [12].

The manufacturing process can also change the flavor characteristics of sesame oil, and it has been used to improve the organoleptic quality of sesame oil. In

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particular, the overall flavor of sesame oil considerably depends on the roasting process, and the effects of the roasting process on these flavor characteristics of sesame oil have been studied [6]. Nakamura et al. [13] identified 221 compounds including pyrazines, pyrroles, pyridines, furans, and sulfur-containing compounds in the oil produced from roasted sesame seeds. Among these, pyrazines (e.g., acetyl pyrazine and alkyl pyrazines) and sulfur-containing compounds (e.g., 2-mercaptoacetaldehyde and hydrogen sulfide) seemed to significantly affect the flavor characteristics of the oil produced from roasted sesame seeds [13]. In addition, volatile compounds such as pyrazines, pyrroles, furans, and sulfur-containing compounds can be produced through various reactions such as Strecker degradation, the Maillard reaction, and lipid oxidation in sesame oil [14, 15]. Lee et al. [16] suggested that various pyrazines can be considered important contributors to the organoleptic properties of roasted sesame oil.

While many studies have investigated the effects of various adsorbents on BaP reduction in foods, no previous study has investigated the effects of various adsorbents such as activated carbons on the changes in volatile profiles during the filtration process. Accordingly, the present study aimed to determine the effects of various adsorbents on changes in BaP and volatile compounds in sesame oil during the filtration process.

## Materials and methods
### Materials and chemicals
Sesame oil was obtained from Chung-Yang food company (Anseong, Gyeonggi-do, Korea). Five adsorbents of activated carbons used in the present study were as follows: powdered type activated carbon made from peat (PP), granular type activated carbon made from coconut shell (GC), powdered type activated carbon made from coconut shell (PC), granular type activated carbon made from wood (GW), powdered type activated carbon made from wood (PW). PC, GW, and PW were obtained from Shin Kwang chemical industry (Yangsan, Gyeongnam, Korea). Two types (PP, GC) of activated carbons were purchased from O-Sung Company (Seoul, Korea) and Samchully Activated Carbon Company (Geumsan, Chungcheongnam-do, Korea), respectively. In addition, celite and kaolin were purchased from Sigma-Aldrich (St. Louis, MO), and acid clay was obtained from Duk-San Science (Seoul, Korea).

Dichloromethane, n-Hexane, and *N*-paraffin oil were obtained from J.T. Baker (Philipsburg, NJ). *p*-Cymene was purchased from Sigma-Aldrich (Saint Louis, MO). *N,N*-Dimethylformamide was purchased from Avantor Performance Materials (Central Valley, PA). benzo[*a*] pyrene (BaP) and benzo[*a*]pyrene-$d_{12}$ (BaP-$d_{12}$) (>99%) were purchased from Supelco (Bellefonte, PA, USA).

### Sample preparation
Sesame oil was stored at room temperature in a dark place before analysis of volatiles. Each BaP (5 µg/kg) was added in sesame oil, and then all samples were stored in the dark for 18 h at room temperature. After that, each sesame oil sample (100 g) containing BaP were filtrated with 0.5% (w/w) of activated carbons such as PP, GC, PC, GW, and PW for analyses of benzo[*a*]pyrene and volatile compounds.

Other adsorbents such as celite, kaolin, and acid clay were used to investigate the effects of the mixtures of activated carbon and other adsorbents on the changes of BaP and volatile profiles. All sesame oil samples were stirred at 600 rpm for 30 min at 70 °C, and then treated with the mixtures of activated carbon and other adsorbents at the ratio of 1:1 (*w/w*). All experiments were conducted in triplicate.

### Ananlysis of benzo[*a*]pyrene in Sesame oil
Sesame oil sample (10 g) and 200 µL of BaP-d$_{12}$ [200 µg/L (w/v) in dichloromethane] as an internal standard were added thoroughly mixed in n-hexane solution (100 mL). The mixtures were extracted twice with 25 mL of *N,N*-dimethylformamide (*N,N*-DMF): water (9:1, v/v) solution. After that, the extract [*N,N*-DMF: water (9:1, v/v) phase] was added in 100 mL of 1% sodium sulfate solution and 50 mL of n-hexane solution. The extracts were centrifuged at 4 °C and 3500 rpm for 10 min (UNION 32R PLUS; Hanil Science Industrial, Incheon, Korea). After separation, the hexane phase was washed two times with 50 mL water. The organic extract was treated with sodium sulfate to eliminate the remaining moisture, and then concentrated to 2 mL using a rotary evaporator (SB-1000; EYELA Co, Tokyo, Japan). The extracts (2 mL) were purified using bonded phase silica SPE cartridges (Sep-Pak silica, 500 mg stationary phase, 6 mL capacity; Agilent Technologies, Palo Alto, CA). Cartridges were conditioned with 10 mL of dichloromethane and 20 mL of n-hexane, and then the concentrated samples were loaded. Then the cartridges were washed with 5 mL of n-hexane before BaP was eluted with 15 mL of n-hexane: dichloromethane (3:1, v/v). The elution of BaP was concentrated using a gentle flow of nitrogen gas, and then the residue was dissolved with 200 µL of dichloromethane.

The analysis of BaP compounds was carried out using a 6890 N gas chromatograph connected to a 5975 mass spectrometer (Agilent Technologies, Santa Clara, CA, USA). A DB-5MS capillary column (30 m length × 0.25 mm I.D., 0.25 µm film thickness, J&W Scientific, Folsom, CA, USA) was used with helium gas

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as carrier gas at a constant flow rate of 1.5 mL min − 1. The injection volume was 1 μL in splitless mode at 320 °C injector temperature. The oven temperature was initially at 100 °C and held for 10 min, increased to 280 °C at 60 °C/min, and then raised to 310 °C at 60 °C/min and held for 10 min. The transfer line temperature was 280 °C, the mass spectrometer scanned from m/z 35 to 500 in electron impact (EI) mode at 70 eV. All samples were analyzed in triplicate.

For identification and quantification of BaP, selected ion monitoring (SIM) detection by GC–MS was used. BaP were identified using authentic standard compounds by matching their retention times and mass spectral data. BaP was detected at $m/z$ 252, 253, and 250, whereas BaP-$d_{12}$ was detected at $m/z$ 264, 265, and 260. For quantification of BaP, the $m/z$ 252 and 264 were used for BaP and BaP-$d_{12}$, respectively. The quantitative analysis was carried out on the base of calibration curve. The calibration curve was applied by various concentrations of BaP solutions in the range of 0.05 – 10 μg/L in dichloromethane ($w/v$), added with a fixed amount (10 μg/L) of BaP-$d_{12}$ as an internal standard. The calibration curve was determined using the ratios of the area of BaP versus the area of BaP-$d_{12}$. The linearity value of $R^2 = 0.9992$ were obtained.

### Analysis of volatile compounds in Sesame oil

20 mL of sesame oil and 0.3 mL of *p*-cymene [1 mg/mL (w/v) in paraffin oil] as an internal standard were put into 50 mL screw vial with a screw cap and kept at 30 °C for 1 h. After the equilibrium, volatiles in headspace were adsorbed onto SPME fiber coated with 50–30 μm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) purchased from Supelco (Bellefonte, PA, USA) at 30 °C for 10 min. Then, desorption was performed at 250 °C for 5 min in a GC injector. All experiments were performed in triplicate.

Analysis of volatile compounds in sesame oil was performed by 6890 N GC system coupled to 5975 mass selective detector equipped with a DB-5MS column (30 m × 0.25 mm, 0.25 μm film thickness, J & W Scientific). The oven temperature was initially held at 40 °C for 6 min, and then increased to 140 °C at 3 °C/min, and then to 200 °C at 60 °C/min. Other GC–MS conditions were based on the previous method of Lee et al. (2016).

Volatile compounds in sesame oil were tentatively identified using Wiley 7n mass spectral database and linear retention indices (RI) values. Volatile compounds were quantified by comparing their peak areas with that of *p*-cymene as internal standard.

### Statistical analysis

Analysis of variance (ANOVA) was performed using SPSS (version 12.0, Chicago, IL, U.S.A.) by Duncan's multi range test to evaluate significant differences ($p < 0.05$) in samples.

## Results and discussion

### Effects of different types of activated carbons on BaP reduction

Sesame oil samples with additional BaP (5 μg/kg) were filtrated with 0.5% (w/w) activated carbons, such as PP, GC, PC, GW, and PW. Figure 1 shows the BaP contents in sesame oil samples filtrated by different types of activated carbons. PW significantly reduced the BaP level in sesame oil compared with that of the control (4.55 μg/kg) to 1.58 μg/kg, followed by PP (1.68 μg/kg), PC (2.08 μg/kg), GW (2.29 μg/kg), and GC (2.47 μg/kg) ($p < 0.05$).

Among the activated carbons, PW and PP were adsorbents that effectively reduced the BaP level of the sesame oil compared with PC, GW, and GC, indicating that polarity of activated carbon could affect BaP absorption in sesame oil. The internal surface areas of mesopores with 1.7—10.0 nm diameter in activated carbons ranged from 21.14 to 406.75 m2/g. PW had the largest internal surface area (406.75 m2/g) of mesopores with 1.7–10.0 nm diameter in activated carbons, followed by GW (305.70 m$^2$/g), PC (203.50 m$^2$/g), PP (137.15 m$^2$/g), and GC (21.14 m$^2$/g). Sesame oil sample filtrated with GC showed the smallest reduction ratio of BaP in sesame oil possibly due to the smallest value of internal surface area of mesopores with 1.7–10.0 nm diameter. In the case of
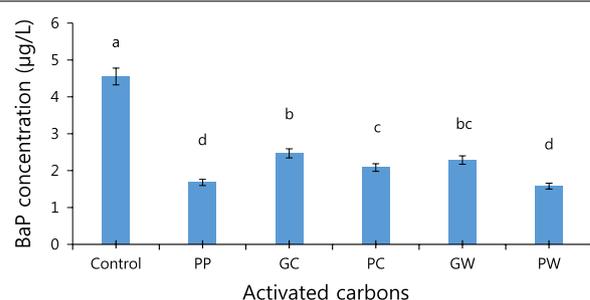


**Fig. 1** Effects of different types of activated carbons on the reduction of benzo[a]pyrene in sesame oil. The symbols of the samples are as follows: Control, sesame oil sample with no treatment; PP, sesame oil sample filtrated with powdered activated carbon made from peat; GC, sesame oil sample filtrated with granular activated carbon made from coconut shell; PC, sesame oil sample filtrated with powdered activated carbon made from coconut shell; GW, sesame oil sample filtrated with granular activated carbon made from wood; PW, sesame oil sample filtrated with powdered activated carbon made from wood. Data are expressed as mean ± SEM (n = 3). The presence of different letters (a–d) indicates a statistical difference at $P < 0.05$ between bars

PW, it showed the highest reduction ratio of BaP in sesame oil samples due to the highest internal surface area of mesopores with 1.7−10.0 nm diameter. On the other hands, PP and PW had the higher carbon/oxygen ratio of internal surface on particles compared to that of other granular types of activated carbons (GC and GW) (data not shown). Accordingly, the reduction of BaP in sesame oil samples could be affected by the internal surface area of mesopores with 1.7−10.0 nm diameter and carbon/oxygen ratio of internal surface on particles including internal surface of pores in activated carbons. Therefore, these activated carbons, particularly, powdered activated carbons, could be used to reduce BaP content in sesame oil products, minimizing their quality change.

## Effects of combining activated carbon and other adsorbents on BaP reduction

Figure 2 shows the contents of BaP in sesame oil filtrated with a mixture (at a 1:1 ratio) of PW and other adsorbents, such as acid clay, kaolin, and celite, at a 600-rpm agitation speed. PW was the most effective adsorbents for the reduction of BaP levels in sesame oil (1.02 μg/kg), followed by a mixture acid of PW and clay (1.26 μg/kg), a mixture acid of PW and kaolin (2.04 μg/kg), and a mixture acid of PW and celite (2.13 μg/kg) ($p < 0.05$). BaP reductions in sesame oil filtrated with a mixture of PW and other adsorbents such as kaolin and celite were smaller than that filtrated with only PW. On the other hand, BaP level in sesame oil filtrated with a mixture of acid clay and PW was reduced more considerably than those treated with PW–kaolin and PW–celite mixtures
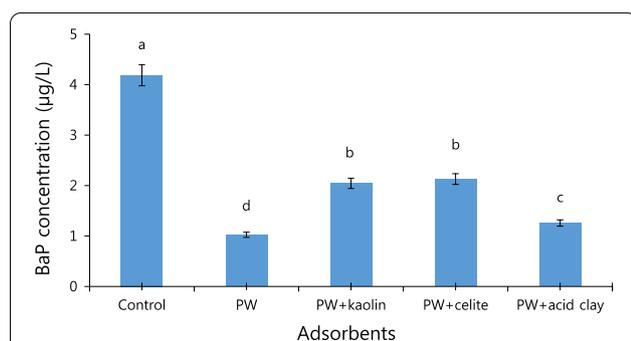


**Fig. 2** Effects of the combination of activated carbon and other adsorbents on the reduction of benzo[a]pyrene in sesame oil. The symbols of each samples are as follows: Control, sesame oil sample with no treatment; PW, sesame oil samples filtrated with powdered activated carbon made from wood; PW + kaolin, sesame oil samples filtrated with a mixture of PW and kaolin at a ratio 1:1; PW + celite, sesame oil samples filtrated with a mixture of PW and celite at a ratio 1:1; PW + acid clay, sesame oil samples filtrated with a mixture of PW and acid clay at a ratio 1:1. Data are expressed as mean ± SEM (n = 3). The presence of different letters (a–d) indicates a statistical difference at P < 0.05 between bars

($p < 0.05$). Choi et al. [10] investigated how various adsorbents such as activated carbon, diatomaceous earth, kaolin, perlite, silicate, celite, and acid clay affected the reduction of BaP in sesame oil. They reported that activated carbon was the most effective adsorbent for reducing BaP, which was consistent with the results of the present study. This might be explained by the ability of nonpolar adsorbents with low oxygen contents to efficiently adsorb PAHs such as BaP [17]. In addition, there was little difference between polar adsorbents such as celite, kaolin, and acid clay, possibly due to their relatively polar structure. Previous studies indicated that celite has a silica surface mostly composed of $SiO_2$ (72%), and is covered by hydroxyl groups and oxygen bridges [18, 19]. In addition, clays such as kaolin and acid clay have the capability to adsorb positively charged compounds because the clay structures have a net negative charge [11]; however, they are used to remove PAHs due to their low cost.

## Effects of activated carbons on changes in volatile compounds

Table 1 lists the effects of different adsorbents such as activated carbons on changes in the volatile profiles of sesame oil. In the sesame oil filtrated with absorbents, 61 volatile compounds including 18 pyrazines, 12 furan and furfurals, 13 thiazoles, 2 pyridines, 5 pyrroles, and 11 sulfur-containing compounds were identified.

Among the various volatile compounds, pyrazines, which are mostly produced by the Maillard reaction that occurs during thermal food processing [2], were predominant in the sesame oil samples in the present study. Pyrazines are major volatile compounds that contribute to the roasted and nutty odor notes in food products processed at high temperatures [20]. Some studies have found that levels of monoalkylpyrazines in deep-roasted sesame oil were relatively decreased, while there were more di- and trialkylpyrazines in deep-roasted sesame oil than in light-roasted sesame oil [5, 21, 22]. In particular, 2-ethyl-6-methylpyrazine, 2-ethyl-5-methylpyrazine, and 3-ethyl-2,5-dimethylpyrazine were important contributors to the flavor characteristics of sesame oil [15]. The present study found no significant differences in pyrazine content in the sesame oil samples filtrated with five activated carbons ($p > 0.05$). However, pyrazines such as 2-methylpyrazine, 2-ethenylpyrazine, 2-ethyl-3-methylpyrazine, 2-ethenyl-6-methylpyrazine, 2-acetylpyrazine, and 2-acetyl-3-methylpyrazine were significantly reduced when sesame oil was filtrated with acid clay combined with PW ($p < 0.05$). This might be explained by the relatively polar surface structure of acid clay and its negative net charge [11].

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**Table 1** Volatile compounds in sesame oil filtrated with different type of adsorbents

| No | RI[a] | Volatile compounds | Relative peak area (Mean ± SD)[b] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | Control[c] | PP[d] | GC[e] | PC[f] | GW[g] | PW[h] | PW + Acid clay[i] |
| Pyrazines | | | | | | | | | |
| a1 | 726 | Pyrazine | 0.452 ± 0.036 a[j] | 0.357 ± 0.085 a | 0.398 ± 0.006 a | 0.368 ± 0.111 a | 0.371 ± 0.076 a | 0.337 ± 0.022 a | 0.348 ± 0.081 a |
| a2 | 820 | 2-Methylpyrazine | 5.377 ± 0.614 a | 4.129 ± 0.760 bc | 5.169 ± 0.179 ab | 4.269 ± 0.822 abc | 4.731 ± 0.823 ab | 4.845 ± 0.360 ab | 3.340 ± 0.289 c |
| a3 | 908 | 2,5-Dimethylpyrazine | 1.222 ± 0.144 a | 1.024 ± 0.187 a | 1.131 ± 0.139 a | 1.092 ± 0.218 a | 1.164 ± 0.171 a | 1.008 ± 0.075 a | 1.201 ± 0.270 a |
| a4 | 909 | 2,6-Dimethylpyrazine | 0.552 ± 0.081 a | 0.455 ± 0.058 a | 0.470 ± 0.051 a | 0.493 ± 0.085 a | 0.484 ± 0.074 a | 0.501 ± 0.025 a | 0.766 ± 0.053 a |
| a5 | 912 | Ethylpyrazine | 0.420 ± 0.060 a | 0.367 ± 0.075 c | 0.357 ± 0.018 a | 0.361 ± 0.086 a | 0.344 ± 0.049 a | 0.314 ± 0.020 a | 0.352 ± 0.031 a |
| a6 | 913 | 2,4-Dimethylpyrazine | 0.104 ± 0.013 a | 0.073 ± 0.012 c | 0.100 ± 0.001 ab | 0.085 ± 0.016 bc | 0.099 ± 0.008 ab | 0.084 ± 0.007 bc | N.D d |
| a7 | 915 | 2,3-Dimethylpyrazine | 0.136 ± 0.014 a | 0.120 ± 0.018 a | 0.131 ± 0.009 a | 0.126 ± 0.021 a | 0.116 ± 0.012 a | 0.117 ± 0.005 a | 0.164 ± 0.016 a |
| a8 | 927 | Ethenylpyrazine | 0.036 ± 0.004 a | 0.031 ± 0.002 b | 0.033 ± 0.002 ab | 0.033 ± 0.007 ab | 0.046 ± 0.015 a | 0.028 ± 0.001 b | 0.002 ± 0.003 c |
| a9 | 995 | 2-Ethyl-6-methylpyrazine | 0.131 ± 0.015 a | 0.113 ± 0.015 a | 0.133 ± 0.013 a | 0.120 ± 0.022 a | 0.122 ± 0.016 a | 0.116 ± 0.009 a | 0.124 ± 0.012 a |
| a10 | 998 | 2-Ethyl-5-methylpyrazine | 0.105 ± 0.006 a | 0.086 ± 0.015 a | 0.101 ± 0.010 a | 0.100 ± 0.018 a | 0.093 ± 0.015 a | 0.087 ± 0.007 a | 0.108 ± 0.014 a |
| a11 | 999 | Trimethylpyrazine | 0.136 ± 0.009 a | 0.124 ± 0.023 a | 0.140 ± 0.002 a | 0.135 ± 0.021 a | 0.125 ± 0.019 a | 0.118 ± 0.006 a | 0.184 ± 0.02 a |
| a12 | 1002 | 2-Ethyl-3-methylpyrazine | 0.066 ± 0.002 a | 0.061 ± 0.008 ab | 0.061 ± 0.003 ab | 0.064 ± 0.012 ab | 0.057 ± 0.008 ab | 0.053 ± 0.003 bc | N.D c |
| a13 | 1014 | 2-Ethenyl-6-methylpyrazine | 0.014 ± 0.003 abc | 0.011 ± 0.003 abc | 0.010 ± 0.000 bc | 0.008 ± 0.001 c | 0.014 ± 0.003 a | 0.010 ± 0.001 bc | 0.001 ± 0.002 d |
| a14 | 1019 | 2-Acetylpyrazine | 0.053 ± 0.001 ab | 0.044 ± 0.003 b | 0.054 ± 0.004 ab | 0.052 ± 0.009 ab | 0.058 ± 0.008 a | 0.052 ± 0.004 ab | 0.023 ± 0.003 c |
| a15 | 1076 | 3-Ethyl-2,5-dimethyl-pyrazine | 0.092 ± 0.012 a | 0.083 ± 0.015 a | 0.092 ± 0.008 a | 0.087 ± 0.011 a | 0.090 ± 0.013 a | 0.078 ± 0.005 a | 0.099 ± 0.011 a |
| a16 | 1082 | 2-Ethyl-3,5-dimethyl-pyrazine | 0.012 ± 0.000 a | 0.010 ± 0.003 b | 0.013 ± 0.001 a | 0.012 ± 0.001 a | 0.013 ± 0.001 a | 0.012 ± 0.001 a | 0.003 ± 0.003 b |
| a17 | 1084 | 2,3-Dimethyl-5-ethyl-pyrazine | 0.013 ± 0.001 a | 0.012 ± 0.002 a | 0.013 ± 0.000 a | 0.012 ± 0.002 a | 0.015 ± 0.002 a | 0.012 ± 0.001 a | 0.002 ± 0.003 b |
| a18 | 1113 | 2-Acetyl-3-methylpyra-zine | 0.021 ± 0.001 abc | 0.017 ± 0.002 ab | 0.022 ± 0.000 ab | 0.020 ± 0.004 bc | 0.024 ± 0.001 a | 0.017 ± 0.001 c | N.D d |
| Furans and furfurals | | | | | | | | | |
| b1 | 830 | 2-Methylfuran | 0.340 ± 0.007 b | 0.414 ± 0.009 a | 0.282 ± 0.005 c | 0.261 ± 0.042 c | 0.289 ± 0.017 c | 0.352 ± 0.017 b | 0.272 ± 0.011 c |
| b2 | 706 | 2,5-Dimethylfuran | 0.076 ± 0.008 a | 0.051 ± 0.002 b | 0.072 ± 0.004 a | 0.058 ± 0.010 b | 0.073 ± 0.004 a | 0.070 ± 0.006 a | 0.075 ± 0.005 a |
| b3 | 805 | Dihydro-2-methyl-3(2H)-furanone | 0.053 ± 0.002 a | 0.037 ± 0.003 c | 0.050 ± 0.003 ab | 0.041 ± 0.006 bc | 0.053 ± 0.001 a | 0.046 ± 0.003 ab | 0.052 ± 0.005 a |
| b4 | 830 | Furfural | 0.241 ± 0.028 a | 0.195 ± 0.016 b | 0.194 ± 0.004 b | 0.191 ± 0.033 b | 0.182 ± 0.006 b | 0.186 ± 0.013 b | 0.214 ± 0.018 ab |
| b5 | 851 | 2-(2-Propenyl)-furan | 0.015 ± 0.002 a | 0.011 ± 0.001 b | 0.013 ± 0.001 b | 0.011 ± 0.002 b | 0.015 ± 0.000 a | 0.013 ± 0.000 b | 0.004 ± 0.001 c |
| b6 | 856 | 2-Furanmethanol | 0.594 ± 0.072 a | 0.494 ± 0.076 ab | 0.584 ± 0.079 a | 0.526 ± 0.089 a | 0.572 ± 0.060 a | 0.531 ± 0.033 a | 0.38 ± 0.041 b |
| b7 | 884 | 5-Methylene-2(5H)-furanone | 0.024 ± 0.002 a | 0.021 ± 0.003 ab | 0.022 ± 0.002 b | 0.019 ± 0.003 ab | 0.022 ± 0.002 ab | 0.020 ± 0.002 b | N.D c |
| b8 | 708 | 5-Methyl-3(2H)-furanone | 0.017 ± 0.002 ab | 0.013 ± 0.004 b | 0.017 ± 0.002 ab | 0.018 ± 0.004 a | 0.015 ± 0.001 ab | 0.016 ± 0.001 ab | N.D c |

**Table 1** (continued)

| No | RI[a] | Volatile compounds | Relative peak area (Mean ± SD)[b] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | Control[c] | Pp[d] | GC[e] | PC[f] | GW[g] | PW[h] | PW + Acid clay[i] |
| b9 | 944 | 2,5-Dimethyl-3(2H)-furanone | 0.029 ± 0.003 a | 0.023 ± 0.003 b | 0.026 ± 0.003 ab | 0.027 ± 0.005 ab | 0.026 ± 0.003 ab | 0.023 ± 0.002 b | 0.005 ± 0.001 c |
| b10 | 961 | 5-Methyl-2-furancarboxaldehyde | 0.106 ± 0.013 a | 0.082 ± 0.016 a | 0.098 ± 0.011 a | 0.092 ± 0.018 a | 0.089 ± 0.010 a | 0.088 ± 0.004 a | 0.057 ± 0.007 b |
| b11 | 991 | 2-Pentyl-furan | 0.016 ± 0.002 b | 0.013 ± 0.000 c | 0.020 ± 0.001 a | 0.012 ± 0.003 c | 0.019 ± 0.001 ab | 0.017 ± 0.002 ab | 0.017 ± 0.001 ab |
| b12 | 1032 | Dihydro-3-hydroxy-4,4-dimethyl-2(3H)-furanone | 0.017 ± 0.002 ab | 0.009 ± 0.001 c | 0.019 ± 0.002 a | 0.011 ± 0.001 c | 0.019 ± 0.002 a | 0.015 ± 0.001 b | N.D d |
| Thiazoles | | | | | | | | | |
| c1 | 735 | Thiazole | 0.292 ± 0.030 a | 0.109 ± 0.005 d | 0.157 ± 0.017 bc | 0.132 ± 0.011 cd | 0.177 ± 0.024 b | 0.158 ± 0.025 bc | 0.167 ± 0.013 bc |
| c2 | 804 | 2-Methylthiazole | 0.097 ± 0.010 a | 0.077 ± 0.007 b | 0.087 ± 0.001 ab | 0.085 ± 0.010 ab | 0.094 ± 0.008 ab | 0.086 ± 0.011 ab | 0.059 ± 0.008 c |
| c3 | 812 | 4-Methylthiazole | 0.206 ± 0.025 ab | 0.165 ± 0.005 bc | 0.173 ± 0.003 bc | 0.169 ± 0.036 bc | 0.178 ± 0.026 bc | 0.154 ± 0.027 c | 0.243 ± 0.022 a |
| c4 | 843 | 5-Methylthiazole | 0.105 ± 0.011 a | 0.076 ± 0.003 c | 0.094 ± 0.007 ab | 0.083 ± 0.012 bc | 0.099 ± 0.015 ab | 0.083 ± 0.002 bc | 0.015 ± 0.000 d |
| c5 | 870 | 4,5-Dihydro-2-methyl-thiazole | 0.022 ± 0.003 a | 0.014 ± 0.004 b | 0.013 ± 0.002 b | 0.012 ± 0.002 b | 0.020 ± 0.001 a | 0.011 ± 0.001 b | N.D c |
| c6 | 880 | 2,4-Dimethylthiazole | 0.191 ± 0.032 a | 0.154 ± 0.031 a | 0.169 ± 0.012 a | 0.174 ± 0.031 a | 0.168 ± 0.022 a | 0.156 ± 0.013 a | 0.196 ± 0.014 a |
| c7 | 898 | 2-Ethylthiazole | 0.009 ± 0.001 a | 0.005 ± 0.000 c | 0.007 ± 0.001 b | 0.007 ± 0.001 b | 0.007 ± 0.001 ab | 0.006 ± 0.001 bc | 0.007 ± 0.001 c |
| c8 | 928 | 4,5-Dimethylthiazole | 0.018 ± 0.006 a | 0.012 ± 0.002 b | 0.020 ± 0.001 a | 0.015 ± 0.002 ab | 0.019 ± 0.003 a | 0.016 ± 0.001 ab | N.D c |
| c9 | 933 | 4,5-Dihydro-2-methyl-thiazole | 0.024 ± 0.001 ab | 0.021 ± 0.002 bc | 0.026 ± 0.002 a | 0.022 ± 0.004 ab | 0.019 ± 0.003 ab | 0.017 ± 0.003 c | 0.002 ± 0.004 d |
| c10 | 969 | 2-Ethyl-4-methylthiazole | 0.017 ± 0.002 a | 0.015 ± 0.002 ab | 0.017 ± 0.001 ab | 0.017 ± 0.003 a | 0.012 ± 0.000 c | 0.014 ± 0.001 bc | 0.005 ± 0.000 d |
| c11 | 970 | 5-Ethyl-2-methylthiazole | 0.010 ± 0.000 b | 0.009 ± 0.000 bc | 0.010 ± 0.001 bc | 0.010 ± 0.002 bc | 0.008 ± 0.001 c | 0.009 ± 0.001 bc | 0.018 ± 0.002 a |
| c12 | 994 | 2,4,5-Trimethylthiazole | 0.016 ± 0.001 a | 0.009 ± 0.000 c | 0.012 ± 0.002 b | 0.009 ± 0.001 c | 0.011 ± 0.002 bc | 0.010 ± 0.001 bc | 0.018 ± 0.002 d |
| c13 | 1017 | 2-Acetylthiazole | 0.016 ± 0.002 bc | 0.018 ± 0.002 ab | 0.020 ± 0.001 ab | 0.019 ± 0.003 ab | 0.021 ± 0.003 a | 0.018 ± 0.001 abc | 0.015 ± 0.002 c |
| Pyridines | | | | | | | | | |
| d1 | 737 | Pyridine | 0.169 ± 0.040 a | N.D c | 0.019 ± 0.001 c | 0.057 ± 0.008 b | 0.028 ± 0.006 b | 0.052 ± 0.017 b | N.D c |
| d2 | 813 | 2-Methylpyridine | 0.107 ± 0.009 a | 0.046 ± 0.006 d | 0.080 ± 0.004 b | 0.044 ± 0.002 d | 0.063 ± 0.005 c | 0.063 ± 0.007 c | N.D e |
| Pyrroles | | | | | | | | | |
| e1 | 736 | 1-Methyl-1H-pyrrole | 0.010 ± 0.003 a | 0.031 ± 0.006 e | 0.060 ± 0.005 b | 0.039 ± 0.007 d | 0.044 ± 0.002 cd | 0.049 ± 0.002 c | N.D f |
| e2 | 751 | 1H-pyrroles | 0.379 ± 0.027 a | 0.276 ± 0.040 b | 0.379 ± 0.006 a | 0.298 ± 0.052 b | 0.296 ± 0.004 b | 0.271 ± 0.026 b | 0.200 ± 0.018 c |
| e3 | 838 | 3-Methyl-1H-pyrrole | 0.030 ± 0.004 a | 0.023 ± 0.003 b | 0.022 ± 0.005 b | 0.024 ± 0.007 ab | 0.013 ± 0.002 c | 0.028 ± 0.002 ab | N.D d |
| e4 | 849 | 2-Methyl-1H-pyrrole | 0.017 ± 0.003 a | 0.007 ± 0.001 d | 0.014 ± 0.002 ab | 0.009 ± 0.003 cd | 0.012 ± 0.001 bc | 0.007 ± 0.001 d | N.D e |

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**Table 1** (continued)

| No | RI[a] | Volatile compounds | Relative peak area (Mean ± SD)[b] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | Control[c] | PP[d] | GC[e] | PC[f] | GW[g] | PW[h] | PW + Acid clay[i] |
| e5 | 1011 | 1H-pyrrole-2-carboxaldehyde | 0.042±0.002 a | 0.026±0.004 c | 0.038±0.003 bc | 0.023±0.003 c | 0.049±0.003 b | 0.035±0.002 bc | 0.006±0.003 d |
| S-containing compounds | | | | | | | | | |
| f1 | <700 | Methanethiol | 0.038±0.014 ab | 0.020±0.004 b | 0.025±0.003 b | 0.029±0.006 ab | 0.034±0.008 ab | 0.033±0.007 ab | 0.048±0.022 a |
| f2 | <700 | 2-Methylthiophene | 0.159±0.007 a | 0.093±0.007 d | 0.128±0.012 bc | 0.106±0.021 cd | 0.144±0.016 ab | 0.156±0.009 a | 0.098±0.007 d |
| f3 | <700 | 2-Methyl-1-propanethiol | 0.034±0.001 b | 0.011±0.003 d | 0.020±0.003 a | 0.014±0.006 cd | 0.023±0.002 c | 0.013±0.002 cd | 0.013±0.006 cd |
| f4 | 738 | Dimethyl disulfide | 0.541±0.070 a | 0.381±0.051 b | 0.534±0.011 c | 0.412±0.068 b | 0.437±0.022 b | 0.438±0.006 b | 0.444±0.048 b |
| f5 | 766 | 2-Methylthiophene | 0.088±0.021 a | 0.057±0.006 c | 0.076±0.002 ab | 0.052±0.008 c | 0.060±0.006 bc | 0.065±0.000 bc | 0.065±0.011 bc |
| f6 | 775 | 3-Methylthiophene | 0.061±0.010 a | 0.026±0.001 d | 0.050±0.006 b | 0.026±0.003 d | 0.041±0.007 bc | 0.030±0.006 cd | 0.028±0.003 d |
| f7 | 783 | 2-Methyl-1-butanethiol | 0.037±0.010 a | 0.041±0.002 a | 0.025±0.001 b | 0.039±0.004 a | 0.046±0.012 a | 0.020±0.002 b | 0.016±0.004 b |
| f8 | 873 | 2,3-Dimethylthiophene | 0.009±0.001 a | 0.006±0.001 b | 0.006±0.001 b | 0.008±0.002 ab | 0.008±0.001 a | 0.008±0.001 a | N.D c |
| f9 | 946 | Dihydro-3-(2H)-thiophenone | 0.025±0.002 abc | 0.025±0.004 abc | 0.027±0.001 ab | 0.026±0.004 abc | 0.029±0.002 a | 0.023±0.003 bc | 0.021±0.004 c |
| f10 | 964 | Dimethyl trisulfide | 0.037±0.004 a | 0.030±0.005 a | 0.024±0.002 a | 0.031±0.005 a | 0.035±0.006 a | 0.033±0.001 a | N.D b |
| f11 | 984 | 4,5-dihydro-2-methyl-3(2H)-thiophenone | 0.008±0.001 b | 0.009±0.001 b | 0.009±0.000 ab | 0.010±0.002 a | 0.009±0.000 ab | 0.008±0.000 ab | N.D c |

[a] Retention indices were determined by using n-paraffins $C_7$-$C_{22}$ as external references

[b] Average of relative peak areas compared to that of the internal standard ± standard deviation

[c] Sesame oil sample with no treatment

[d] Sesame oil sample filtrated with powdered activated carbon made from peat

[e] Sesame oil sample filtrated with granular activated carbon made from coconut shell

[f] Sesame oil sample filtrated with powdered activated carbon made from coconut shell

[g] Sesame oil sample filtrated with granular activated carbon made from wood

[h] Sesame oil sample filtrated with powdered activated carbon made from wood

[i] Sesame oil sample filtrated with acid clay combined with powdered activated carbon made from wood in a proportion of 1:1

[j] There are significant differences ($p < 0.05$) among sesame oil samples filtrated with 6 different activated carbons by using Duncan's multiple comparison test between the samples having different letter in row

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Furans are also considered major flavor constituents of sesame oil, with caramel-like and roasted odor notes [23]. Furans, including alkylfurans, which can be produced at high-temperature food processing, are mostly generated by carbohydrate and lipid degradation [24]. Some furans, such as 5-methylene-2(5H)-furanone, 5-methyl-3(2H)-furanone, 2,5-dimethyl-3-(2H)-furanone, 5-methyl-2-furancarboxaldehyde, and dihydro-3-hydroxy-4,4-dimethyl-2(3H)-furanone, were significantly reduced after filtration with acid clay combined with PW. This could be due to the relatively polar structure of furan compounds, especially furanones, which have more than one oxygen molecule, and the relatively polar structure of acid clay [11, 25].

Thiazoles are major contributors to the pleasant sulfur and nutty odor notes in roasted sesame seeds [13]. Some thiazoles, such as thiazole, 4-methylthiazole, 5-methylthiazole, 4,5-dihydro-2-methylthiazole, 2-ethylthiazole, 4,5-dimethylthiazole, and 2,4,5-trimethylthiazole, detected in the present study were considerably reduced after filtration with powdered activated carbons. It might be due to the relatively large internal surface areas of the pores in activated carbons compared with those of granular activated carbons derived from coconut shells.

Pyrroles were important contributors to the burnt and earthy characteristic odor notes in sesame oil [15]. The amounts of pyrroles were greatly decreased in sesame oil samples filtrated with powdered activated carbon (PP, PC, and PW), while these compounds were slightly reduced in sesame oil samples filtrated with granular activated carbon (GC and GW). Especially, 1H-pyrrole-2-carboxaldehyde, which has sweet and medicinal odor notes, was greatly decreased after filtration with powdered activated carbons (PP, PC, and PW).

Pyridines were detected in large amounts in sesame oil samples. The amounts of pyridines, including pyridine and 2-methylpyridine, were greatly decreased by filtration with powdered activated carbons due to the large internal surface area of the pores in these carbons.

Sulfur-containing compounds were also considerably decreased by filtration with activated carbons (PP, PC, PW, GC, and GW) in this study. Among sulfur-containing compounds, thiol compounds have predominantly roasted, green, nutty, and vegetable-like odor notes in sesame seed oil [15]. Increased thiol concentration could also be associated with the undesirable flavor of sesame oil as it induces a burnt or rubbery aroma from the formation of furfurylthiol [22]. In the present study, the concentration of sulfur-containing compounds such as thiols was also greatly decreased after filtration with powdered activated carbons (PP, PC, and PW) compared with filtration with granular activated carbons (GC and GW).

Overall, volatile compounds changed considerably when filtrated with powdered activated carbons (PP, PC, and PW) compared with granular activated carbons (GC and GW). It could be explained that powdered activated carbons (PP, PC, and PW) showed higher adsorption efficiency of volatile compounds in sesame oil, possibly due to the large surface area of the internal pores in powdered activated carbons [26]. In particular, volatile compounds were greatly reduced after filtration with acid clay combined with PW at a 1:1 ratio. This might be due to the relatively ionic surface structure of acid clay [11]. Some studies explained the ionic characteristics of clays used in this study, demonstrating that clays have cation exchange capacity in addition to high specific surface area [27, 28]. In particular, their ionic characteristics could affect the adsorption and reduction of some volatiles, such as pyrazines, with positive ions depending the pH. However, it would be more critical in aqueous systems rather than sesame oil.

## Authors' contributions
Conceptualization, Y-SK; Experiments, J-YJ; Analysis, SML and J-GL; Investigation, Y-SK and J-GL; Data Curation, J-YJ and SML; Writing-Original Draft Preparation, SML and J-YJ; Writing-Review and Editing, Y-SK and SML; Supervision, Y-SK. All authors read and approved the final manuscript.

## Declarations

### Competing interests
The authors declare that they have no competing interests.

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