

# Analysis of volatile compounds of black bean, mung bean, and soybean extracts prepared with distillation under reduced pressure-continuous liquid-liquid extraction and hot water extraction

**Eunji Han**

Dongguk University-Seoul

**Hyunwoo Ahn**

Dongguk University-Seoul

**Seongho Lee**

Dongguk University-Seoul

**Kwang-Geun Lee** (✉ [kwglee@dongguk.edu](mailto:kwglee@dongguk.edu))

Dongguk University-Seoul


---

## Research Article

**Keywords:** black bean, mung bean, soybean, extraction methods, volatiles, GC-MS

**Posted Date:** September 27th, 2022

**DOI:** <https://doi.org/10.21203/rs.3.rs-2087234/v1>

**License:**  This work is licensed under a Creative Commons Attribution 4.0 International License. [Read Full License](#)

---

## Abstract

Beans are cultivated world-widely and are well-known for being abundant in proteins (20–25%), carbohydrates (50–60%), minerals and vitamins. Several researchers studied the volatile aroma constituents of beans but have not researched the effect of particle size and extraction temperature of beans. Therefore, in our research, volatile compounds of various beans (black bean, mung bean, and soybean) were analyzed on the basis of particle sizes and extraction temperatures by two extraction methods, namely, distillation under reduced pressure-continuous liquid–liquid extraction (DRP-LLE) and hot water extraction. The experimental results confirmed the presence of 10 volatile components. The five major volatile compounds were hexanal, 2-methyl-1-butanol, 1-hexanol, 1-octen-3-ol and benzaldehyde. The highest total volatile compound concentrations in the extracts of black bean, mung bean, and soybean by using DRP-LLE were obtained at 60°C and 355–500 µm, 60°C and 500–710 µm, and 50°C and 355–500 µm, respectively. For the same particle size, the total volatile compound concentrations in the extracts of black bean, mung bean, and soybean obtained by HWE at 70°C were 2–3 times significantly higher than those obtained at 90°C. Moreover, the highest total volatile compound concentration was obtained in the black bean extract by HWE at 500–710 µm, while the lowest total volatile compound concentration in the soybean extract was obtained by HWE at 500–710 µm. The total concentrations of volatiles in the black bean and soybean extracts obtained by DRP-LLE were significantly higher than those obtained by HWE.

## 1. Introduction

Beans are widely grown throughout the world and include many species such as soybean, black bean, mung bean, and kidney bean, which show distinct characteristics. In Korea, beans are the main legume species used in the traditional Korean diet. Moreover, Koreans commonly consume fermented products of beans, such as doenjang and cheongkukjang (1). Beans are a rich source of proteins (20%-25%), carbohydrates (50%-60%), minerals, and vitamins (2). The nutritional content of beans varies greatly depending on the type of beans (3). In addition to these nutrients, beans contain various functional ingredients, such as isoflavones, saponins, anthocyanins, and tocopherol, which have been globally attracting attention as functional food materials (4). Beans have various physiological effects such as antioxidant and antimicrobial properties (5), anti-inflammatory activities (6), anti-obesity effects (7), antinociceptive activities (8), and the ability to prevent chronic degenerative diseases (9).

Volatile profiling of beans is both economically and sensorially important because it can be used to characterize off-odours as well as grade beans (10). Several previous studies have reported profiles of the volatile compounds in beans. The properties of beans can be affected by their cultivation region because each cultivation area has different local weather conditions, soil, and herbicide profiles (11–14). Several researchers are reported that variation of volatile compounds is dependent on extraction temperature, particle size, and sample species. According to the previous report, the highest volatile compounds of rice extract were detected at extraction temperatures of 60 and 70 at a particle size of 355 µm (15). Arabica espresso coffee prepared as the particle sizes range between 300 and 425 µm was shown the highest volatile compounds concentrations (16). The larger the particle sizes (125 µm), the more volatile compounds were detected in *Ligusticum chuanxiong* Hort (17). Volatile compounds have been shown to be significantly different between soybeans from different ecological cultivation regions (18). The properties of beans can also be affected by the maturity stage (19). The major volatile compounds in soybeans at the pod-fill stage are 3-hexanone, (E)-2-hexenal, 1-hexanol, and 3-octanone, while those at the physiological maturity stage are hexanal, (E)-2-heptenal, (E)-2-octenal, ethanol, 1-hexanol, 1-octen-3-ol, maltol, phenylethyl alcohol, and  $\gamma$ -butyrolactone (20, 21). The major volatile compounds in mung beans are hexanol, benzyl alcohol,  $\gamma$ -butyrolactone, 2-methyl-2-propenal, and pentanol (22, 23).

Several extraction and analysis methods have been used to evaluate the volatile compounds of beans. The volatile compounds of kidney beans and soybeans were isolated with the model mouth system and analysed with gas chromatography mass spectrometry (GC-MS) (24). The volatile compounds of black beans were isolated with simultaneous steam distillation extraction (SDE) and headspace solid phase microextraction (HS-SPME) and also analysed with GC-MS (25, 26). However, the SDE method can cause degradation or loss of volatiles since it is performed at high distillation temperatures. To prevent such degradation or loss of volatiles, soybeans and mung beans were extracted by steam distillation under reduced pressure (DRP) and continuous liquid–liquid extraction (LLE) and analysed with GC-MS (20).

Although several studies have assessed the volatile aroma constituents of beans, to our knowledge, the effects of milling and extraction temperatures on the volatile compounds in beans have not been reported to date. This study aimed to evaluate the effects of particle size and extraction temperature on volatile compounds of different kinds of beans. To this end, the volatile compound profiles in bean extracts were analysed in relation to their particle size and extraction temperature. Steam DRP followed by continuous LLE with 30% ethanol and hot water extraction (27) followed by HS-SPME were used to profile the volatile compounds of various dry beans (black bean, mung bean, and soybean).

## 2. Materials And Methods

### 2.1. Chemical reagents and materials

Dried black beans [*Phaseolus vulgaris* L.], mung beans [*Vigna radiata* (L.) R. Wilczek], and soybeans [*Glycine max* (L.) Merr.] were harvested and dried at Dongguk University Farm located in Goyang, Republic of Korea, in 2020. Beans were sown in June and harvested in October. The growing conditions of beans followed the farm's protocol. Beans were dried using a convection oven (Jinsung Co., Seoul, Korea) at 40°C for 48 h. The variety of plants was identified in the department of biological science of Dongguk University, Goyang, Republic of Korea. The beans were ground for 20 s by a grinder (SMKANB-4000; Poongnyun Co., Ltd., Korea), and the ground beans were passed through three different standard testing sieves with sizes of 355 µm, 500 µm, and 710 µm for 2 min by using a sieve shaker (CG-211-8; Chunggye Co., Korea). The selection of the sieve size was based on the previous report (20). The moisture contents of mung bean, soy bean, and black bean are 12, 8.5, and 8%, respectively. All beans were stored at room temperature until the analysis.

Ethanol (30% v/v) was purchased from J. T. Baker (Center Valley, PA, USA), and stored at room temperature until the analysis. HPLC-grade water, methanol (> 99.8%), hexane (> 97.0%), and dichloromethane (DCM, > 99.8%) were purchased from J. T. Baker (Center Valley, PA, USA). Anhydrous sodium sulphate (> 95%) was purchased from Junsei Chemical (Tokyo, Japan). Sodium chloride (99.0%) was purchased from Samchun Co. (Seoul, Korea).

Divinylbenzene/carboxen/polydimethylsiloxane (50  $\mu$ m DVB/CAR/PDMS) solid-phase microextraction (SPME) fibre was purchased from Supelco Inc. (Bellefonte, PA, USA). Methyl cinnamate (> 99.0%) and an alkane standard (C7-C30, > 98.0%) were purchased from Sigma-Aldrich Chemical Co. (St. Louis, Mo, USA). Seventeen standard compounds were purchased from different suppliers as follows: hexanal (> 95.0%), 2-methyl-1-butanol (> 98.0%), 2-heptanone (> 99.0%), 3-octanone (> 98.0%), 1-hexanol (> 99.9%), 3-octanol (> 99.5%), benzaldehyde (> 99.5%), phenethyl alcohol (> 98.0%) were purchased from Sigma-Aldrich Chemical Co. (St. Louis, Mo, USA), while cis-3-hexen-1-ol (> 97.0%), 1-pentanol (> 99.0%), octanol (> 98.0%), nonanal (> 95.0%), 1-octen-3-ol (> 98.0%), 1-octanol (> 98%),  $\gamma$ -hexalactone (> 99.0%), 1-nonanol (> 99.0%), and benzyl alcohol (> 99.0%) were obtained from Tokyo Chemical Industry Co, (Tokyo, Japan).

## 2.2. Isolation of volatile compounds by DRP and continuous LLE

Volatile compounds from beans (black bean, mung bean, and soybean) were extracted using a slight modification of a previously reported DRP method (28). The extraction of beans was carried out triplicate replication for the analysis.

In this method, 100 g of beans, 400 mL of 30% ethanol, a magnetic stirrer bar, and boiling chips were filled into a 1L round-bottomed flask. Next, the solution was spiked with 50  $\mu$ L of methyl cinnamate as an internal standard (100  $\mu$ g/mL) and 20  $\mu$ L of n-alkane standard (100  $\mu$ g/mL). The mixture was steam-distilled at designated temperatures (50, 60, and 70°C) under reduced pressures (50 mmHg). The distillate (200 mL) was extracted with 200 mL of dichloromethane (DCM) using continuous LLE for 6 h at 60°C. After extraction, anhydrous sodium sulphate was added to the collected DCM for dehydration, and the solution was stored in a deep freezer overnight. Subsequently, anhydrous sodium sulphate was separated from the solution by using filter paper (No. 1, 110 mm, Whatman). The solution was concentrated with a rotary evaporator to 1 mL, and the residual solution was further concentrated under a purified nitrogen steam to 0.4 mL. This extract sample was used for gas chromatography-mass spectrometer detector (GC-MSD) analysis.

## 2.3. Isolation of volatile compounds by HWE and HS-SPME

Volatile compounds from beans (black bean, mung bean, and soybean) were extracted using a slightly modified version of a method described in a previous study (29). HWE and HS-SPME followed by GC-MSD analysis were performed to assess the volatile compounds of beans. For HWE, 30 g of beans and 150 mL of distilled water were filled into a 250 mL Duran laboratory bottle. The mixture was extracted overnight in a water bath (70°C), followed by filtration using filter paper (No. 1, 110 mm, Whatman).

For HS-SPME, 10 mL of the extracted solution, 1 g of sodium chloride, a magnetic stirrer bar, and boiling chips were filled into a 20 mL headspace vial. The solution was spiked with 3  $\mu$ L of methyl cinnamate as an internal standard (10  $\mu$ g/mL) and 20  $\mu$ L of n-alkane standard (100  $\mu$ g/mL). The headspace vial was incubated for 10 min in a beaker filled with distilled water maintained at a designated temperature (70, 80, and 90°C) by a hot plate. After equilibrium, the SPME fiber, divinylbenzene/carboxen/poly-dimethylsiloxane (DVB/CAR/PDMS) for 40 min at 70, 80, and 90°C, respectively. After absorption, the SPME fibre was inserted into the injection port of GC at 230°C for 10 min for desorption of volatile compounds.

## 2.4. Instrumental analysis

Analysis of volatile compounds in beans (black bean, mung bean, soy bean) was performed by using GC-MSD (Agilent 7820A gas chromatography with Agilent 5977E mass spectrometry detector). A DB-WAX column (60 m  $\times$  0.32 mm I.D.  $\times$  0.5  $\mu$ m film thickness, J&W Scientific, Folsom, CA) was used to separate volatile compounds. The helium carrier gas flow was maintained at 1.0 mL/min with a constant flow. The injector was used in the splitless mode at 230°C. The oven temperature was set to 44°C for 5 min. Subsequently, the temperature was raised to 170°C at 3°C/min and held for 10 min, and finally raised to 240°C at 8°C/min for 5 min. The ionization energy was 70 eV and scan range was 50–550 m/z.

## 2.5. Identification and quantification of volatile compounds

Volatile compounds of beans (black bean, mung bean, and soybean) were analysed in relation to various particle sizes and extraction temperatures. Bean extracts were analysed by two types of extraction methods: (a) DRP and continuous LLE, (b) HWE and HS-SPME.

Volatile compounds of bean extracts were analysed qualitatively and quantitatively, and the 10 most abundant volatile compounds in each extract were identified. Identification of volatile compounds was performed on the basis of three parameters: (A) mass spectrum (MS) in Wiley Library, (B) Kovats retention index (KI) on the DB-WAX column in the NIST database, and (C) co-injection (CO). For quantification of volatile compounds, the samples were run in triplicate. The peak area ratio (PAR) was calculated by dividing the integrated areas based on the total ion chromatograms with the areas of the internal standard.

## 2.6. Statistical analysis

Each experiment was repeated three times and the mean  $\pm$  standard deviation (SD) were presented. To compare significant differences, data were analysed with one-way ANOVA and Duncan's multiple-range tests ( $P < 0.05$ ). Statistical analysis was performed by IBM SPS Statistics 25 (IBM, Chicago, USA).

## 3. Results And Discussion

### 3.1. Isolation of volatile compounds by distillation under reduced pressure (DRP) and continuous liquid-liquid extraction (LLE)

The volatile compounds of each bean (black bean, mung bean, soybean) extracted by DRP-LLE were analysed qualitatively, and the major volatile compounds were selected. A total of two aldehydes, seven alcohols, and one ketone were identified in black bean extracts (Table 1). The results of quantitative analysis of

the volatile compounds in black bean extracts with various particle sizes and extraction temperatures are shown in Table 1. Bean samples have significantly different volatile components depending on the cultivation area and environment, but certain components exist in common (18). Among the 3 groups of volatile compounds, alcohols were detected the most, followed by aldehydes and ketones. Aldehydes extracted from black bean by DRP-LLE included hexanal and nonanal. Hexanal is characterized by a grassy and fatty odour, and nonanal had a floral and waxy odour (20). Compared with SDE method (26), a loss of volatiles was reduced in current DRP-LLE method.

Table 1  
Concentrations of volatile compounds identified in black bean (*Phaseolus vulgaris* L.) extracts with DRP-LLE

Compounds	KI	KI (Ref.)	Identification	Peak area ratio <sup>1)</sup>								
				Distillation at 50°C			Distillation at 60°C			Distillation at 70°C		
				355– 500 μ	500– 710 μ	Whole size	355– 500 μ	500– 710 μ	Whole size	355– 500 μ	500– 710 μ	Whole size
<b>Aldehydes</b>												
Hexanal	1069	1084	MS, KI, Co	1.012 ± 0.031 <sup>a,A</sup>	0.868 ± 0.027 <sup>a,B</sup>	0.214 ± 0.007 <sup>a,B</sup>	3.241 ± 0.098 <sup>b,A</sup>	0.927 ± 0.028 <sup>a,B</sup>	0.323 ± 0.010 <sup>a,C</sup>	3.877 ± 0.371 <sup>b,A</sup>	3.239 ± 0.098 <sup>b,A</sup>	0.829 ± 0.026 <sup>b,B</sup>
Nonanal	1408	1395	MS, KI, Co	0.279 ± 0.008 <sup>a,A</sup>	0.207 ± 0.004 <sup>a,B</sup>	0.223 ± 0.006 <sup>a,B</sup>	0.376 ± 0.012 <sup>b,A</sup>	0.355 ± 0.011 <sup>b,A</sup>	0.206 ± 0.006 <sup>a,B</sup>	0.437 ± 0.053 <sup>c,A</sup>	0.467 ± 0.015 <sup>c,A</sup>	0.320 ± 0.009 <sup>b,B</sup>
<b>Total aldehydes</b>				1.291 ± 0.039	1.075 ± 0.030	0.437 ± 0.013	3.617 ± 0.110	1.283 ± 0.039	0.529 ± 0.016	3.475 ± 0.424	3.706 ± 0.113	1.149 ± 0.035
<b>Alcohols</b>												
2-Methyl-1-butanol	1194	1208	MS, KI, Co	1.939 ± 0.060 <sup>a,A</sup>	2.238 ± 0.069 <sup>a,B</sup>	1.456 ± 0.044 <sup>b,B</sup>	5.659 ± 0.172 <sup>b,A</sup>	1.767 ± 0.053 <sup>b,B</sup>	1.437 ± 0.044 <sup>a,C</sup>	2.327 ± 0.081 <sup>c,A</sup>	2.318 ± 0.070 <sup>a,A</sup>	2.217 ± 0.064 <sup>b,A</sup>
1-Hexanol	1365	1360	MS, KI, Co	0.876 ± 0.027 <sup>a,A</sup>	0.752 ± 0.021 <sup>a,B</sup>	0.514 ± 0.016 <sup>a,C</sup>	1.300 ± 0.039 <sup>b,A</sup>	1.087 ± 0.033 <sup>b,B</sup>	0.542 ± 0.016 <sup>a,C</sup>	0.777 ± 0.004 <sup>c,AB</sup>	0.805 ± 0.024 <sup>c,A</sup>	0.720 ± 0.021 <sup>b,C</sup>
1-Pentanol	1230	1212	MS, KI, Co	6.741 ± 0.205 <sup>a,A</sup>	6.412 ± 0.198 <sup>a,B</sup>	4.256 ± 0.131 <sup>a,C</sup>	9.139 ± 0.279 <sup>b,A</sup>	6.688 ± 0.206 <sup>ab,B</sup>	4.210 ± 0.127 <sup>a,C</sup>	5.114 ± 0.050 <sup>c,A</sup>	5.264 ± 0.165 <sup>c,A</sup>	5.790 ± 0.179 <sup>b,B</sup>
3-Octanol	1404	1394	MS, KI, Co	0.579 ± 0.018 <sup>a,A</sup>	0.410 ± 0.012 <sup>a,B</sup>	0.227 ± 0.007 <sup>a,C</sup>	0.619 ± 0.019 <sup>b,A</sup>	0.464 ± 0.014 <sup>b,B</sup>	0.209 ± 0.006 <sup>a,C</sup>	0.394 ± 0.015 <sup>c,A</sup>	0.400 ± 0.012 <sup>a,A</sup>	0.371 ± 0.010 <sup>b,B</sup>
1-Octen-3-ol	1460	1445	MS, KI, Co	10.146 ± 0.309 <sup>a,A</sup>	9.189 ± 0.280 <sup>a,B</sup>	11.611 ± 0.354 <sup>a,C</sup>	12.023 ± 0.367 <sup>b,A</sup>	10.616 ± 0.326 <sup>b,B</sup>	8.151 ± 0.252 <sup>b,C</sup>	8.057 ± 0.409 <sup>c,A</sup>	8.249 ± 0.250 <sup>c,A</sup>	8.523 ± 0.262 <sup>b,A</sup>
Benzyl alcohol	1900	1880	MS, KI, Co	1.210 ± 0.037 <sup>a,A</sup>	0.903 ± 0.029 <sup>a,B</sup>	0.855 ± 0.026 <sup>a,B</sup>	1.238 ± 0.038 <sup>a,A</sup>	1.725 ± 0.054 <sup>b,B</sup>	0.518 ± 0.016 <sup>b,C</sup>	0.612 ± 0.024 <sup>b,A</sup>	0.621 ± 0.019 <sup>c,A</sup>	0.770 ± 0.023 <sup>c,B</sup>
Phenethyl-alcohol	1935	1929	MS, KI, Co	0.268 ± 0.009 <sup>a,A</sup>	0.367 ± 0.012 <sup>a,B</sup>	0.207 ± 0.007 <sup>a,C</sup>	0.354 ± 0.011 <sup>b,A</sup>	0.584 ± 0.015 <sup>b,B</sup>	0.117 ± 0.004 <sup>b,C</sup>	0.248 ± 0.008 <sup>c,A</sup>	0.251 ± 0.007 <sup>c,A</sup>	0.074 ± 0.002 <sup>c,B</sup>
<b>Total alcohols</b>				21.758 ± 0.665	20.271 ± 0.620	19.125 ± 0.585	30.332 ± 0.925	22.931 ± 0.701	15.183 ± 0.464	71.529 ± 0.530	17.906 ± 0.548	18.465 ± 0.561
<b>Ketones</b>												
γ-Hexalactone	1702	1724	MS, KI, Co	0.095 ± 0.003 <sup>a,A</sup>	0.138 ± 0.005 <sup>a,B</sup>	0.033 ± 0.001 <sup>a,C</sup>	0.138 ± 0.004 <sup>b,A</sup>	0.212 ± 0.005 <sup>b,B</sup>	0.020 ± 0.001 <sup>b,C</sup>	0.059 ± 0.007 <sup>c,A</sup>	0.063 ± 0.002 <sup>c,A</sup>	N.D.
<b>Total ketones</b>			MS, KI, Co	0.095 ± 0.003	0.138 ± 0.005	0.033 ± 0.001	0.138 ± 0.004	0.212 ± 0.005	0.020 ± 0.001	0.059 ± 0.007	0.063 ± 0.002	N.D.
<b>Total volatiles</b>				23.144 ± 0.707 <sup>a,A</sup>	21.483 ± 0.656 <sup>a,B</sup>	19.595 ± 0.599 <sup>a,C</sup>	34.087 ± 1.039 <sup>b,A</sup>	24.425 ± 0.746 <sup>b,B</sup>	15.733 ± 0.481 <sup>b,C</sup>	21.063 ± 0.923 <sup>c,A</sup>	21.675 ± 0.663 <sup>a,A</sup>	19.614 ± 0.596 <sup>a,B</sup>

<sup>1)</sup> All concentration values are presented as mean ± standard deviation. <sup>2)</sup> MS, identification of mass spectrum with reference to the NIST mass spectrum library, <sup>3)</sup> Kovats retention index on DB-WAX; KI (Ref), Retention index on DB-WAX in the NIST database. Mean values with different letters (a, b, c – extraction temperature/A, B, C – particle size) in the same horizontal line indicate significant differences according to Duncan's test (p < 0.05). N.D.: not detected

At 50°C, the nonanal content was greater than the hexanal content. Alcohol compounds included 2-methyl-1-butanol, 1-pentanol, 1-hexanol, 3-octanol, 1-octen-3-ol, benzyl alcohol, and phenethyl alcohol. These alcohol compounds are characterized by sweet, oily, and floral odours. Among alcohol compounds, 1-octen-3-ol showed the highest concentration, and it had a mushroom-like and fatty odour (30). The ketone compound was γ-hexalactone, which had a waxy and creamy odour (31). However, at 70°C, γ-hexalactone was not detected. The sums of the peak area ratios of nine samples are presented in Table 1. The highest concentration of volatile compounds in black bean was extracted at 60°C with a particle size of 355–500 μm, while the lowest concentration of volatile compounds was extracted at 60°C with whole size. These results are consistent with the results of previous studies in which the smaller the coffee bean particles, the more soluble and volatile compounds were extracted (32).

A total of three aldehydes and seven alcohols were identified in mung bean extracts (Table 2), with alcohols being predominantly detected. The aldehyde compounds extracted from mung bean by DRP-LLE included octanal, nonanal, and benzaldehyde. According to the previous report, the major volatiles in mung beans are hexanol, benzyl alcohol, and  $\gamma$ -butyrolactone (22, 23).

Table 2  
Concentrations of volatile compounds identified in mung bean (*Vigna radiata L.*) extracts with DRP-LLE

Compounds	KI	KI (Ref.)	Identification	Peak area ratio <sup>1)</sup>								
				Distillation at 50°C			Distillation at 60°C			Distillation at 70°C		
				355–500 $\mu$	500–710 $\mu$	Whole size	355–500 $\mu$	500–710 $\mu$	Whole size	355–500 $\mu$	500–710 $\mu$	Whole size
<b>Aldehydes</b>												
Octanal	1282	1264	MS, KI, Co	0.020 $\pm$ 0.001 <sup>a,A</sup>	N.D.	0.016 $\pm$ 0.002 <sup>a,B</sup>	0.006 $\pm$ 0.001 <sup>b,A</sup>	0.015 $\pm$ 0.001 <sup>a,BC</sup>	0.015 $\pm$ 0.001 <sup>ab,BC</sup>	N.D.	0.019 $\pm$ 0.002 <sup>b,A</sup>	0.000 $\pm$ 0.000
Nonanal	1401	1395	MS, KI, Co	0.418 $\pm$ 0.025 <sup>a,A</sup>	0.123 $\pm$ 0.007 <sup>a,B</sup>	0.255 $\pm$ 0.028 <sup>a,C</sup>	0.361 $\pm$ 0.045 <sup>ab,AB</sup>	0.283 $\pm$ 0.014 <sup>b,C</sup>	0.302 $\pm$ 0.032 <sup>ab,AC</sup>	0.163 $\pm$ 0.079 <sup>c,A</sup>	0.279 $\pm$ 0.023 <sup>b,B</sup>	0.200 $\pm$ 0.000
Benzaldehyde	1539	1527	MS, KI, Co	0.292 $\pm$ 0.018 <sup>a,A</sup>	0.335 $\pm$ 0.017 <sup>a,A</sup>	0.219 $\pm$ 0.024 <sup>a,A</sup>	0.252 $\pm$ 0.031 <sup>a,A</sup>	0.694 $\pm$ 0.035 <sup>b,B</sup>	0.215 $\pm$ 0.023 <sup>a,A</sup>	0.428 $\pm$ 0.108 <sup>ab,AB</sup>	0.256 $\pm$ 0.022 <sup>a,A</sup>	4.600 $\pm$ 0.100
<b>Total aldehydes</b>				0.507 $\pm$ 0.031	0.525 $\pm$ 0.026	0.324 $\pm$ 0.035	0.406 $\pm$ 0.051	0.990 $\pm$ 0.050	0.319 $\pm$ 0.034	0.577 $\pm$ 0.144	0.429 $\pm$ 0.036	4.500 $\pm$ 0.400
<b>Alcohols</b>												
2-Methyl-1-butanol	1205	1208	MS, KI, Co	4.529 $\pm$ 0.273 <sup>a,A</sup>	4.957 $\pm$ 0.253 <sup>a,A</sup>	1.242 $\pm$ 0.134 <sup>a,B</sup>	3.668 $\pm$ 0.453 <sup>b,A</sup>	6.140 $\pm$ 0.313 <sup>b,B</sup>	1.254 $\pm$ 0.135 <sup>a,C</sup>	1.138 $\pm$ 0.065 <sup>c,A</sup>	4.037 $\pm$ 0.333 <sup>c,A</sup>	0.800 $\pm$ 0.000
1-Pentanol	1235	1212	MS, KI, Co	0.433 $\pm$ 0.026 <sup>a,A</sup>	0.447 $\pm$ 0.023 <sup>a,A</sup>	0.223 $\pm$ 0.025 <sup>a,B</sup>	0.322 $\pm$ 0.040 <sup>b,A</sup>	0.615 $\pm$ 0.031 <sup>b,B</sup>	0.247 $\pm$ 0.027 <sup>a,C</sup>	0.217 $\pm$ 0.097 <sup>c,A</sup>	0.343 $\pm$ 0.032 <sup>c,A</sup>	0.200 $\pm$ 0.000
1-Hexanol	1377	1360	MS, KI, Co	1.467 $\pm$ 0.088 <sup>a,A</sup>	1.084 $\pm$ 0.055 <sup>a,B</sup>	0.678 $\pm$ 0.073 <sup>a,C</sup>	1.049 $\pm$ 0.129 <sup>b,A</sup>	1.644 $\pm$ 0.084 <sup>b,B</sup>	0.680 $\pm$ 0.073 <sup>a,C</sup>	0.250 $\pm$ 0.030 <sup>c,A</sup>	0.873 $\pm$ 0.073 <sup>c,B</sup>	0.400 $\pm$ 0.000
1-Octanol	1577	1563	MS, KI, Co	0.195 $\pm$ 0.012 <sup>a,A</sup>	0.190 $\pm$ 0.009 <sup>a,A</sup>	0.089 $\pm$ 0.010 <sup>a,B</sup>	0.148 $\pm$ 0.019 <sup>b,A</sup>	0.281 $\pm$ 0.014 <sup>b,B</sup>	0.089 $\pm$ 0.010 <sup>a,C</sup>	0.149 $\pm$ 0.037 <sup>b,A</sup>	0.150 $\pm$ 0.013 <sup>c,A</sup>	0.000 $\pm$ 0.000
1-Nonanol	1714	1724	MS, KI, Co	0.432 $\pm$ 0.026 <sup>a,A</sup>	0.384 $\pm$ 0.020 <sup>a,B</sup>	0.173 $\pm$ 0.019 <sup>a,C</sup>	0.224 $\pm$ 0.028 <sup>b,A</sup>	0.594 $\pm$ 0.030 <sup>b,B</sup>	0.172 $\pm$ 0.019 <sup>a,C</sup>	0.224 $\pm$ 0.045 <sup>b,A</sup>	0.293 $\pm$ 0.025 <sup>c,B</sup>	0.100 $\pm$ 0.000
Benzyl alcohol	1900	1880	MS, KI, Co	1.120 $\pm$ 0.067 <sup>a,A</sup>	2.168 $\pm$ 0.111 <sup>a,B</sup>	0.966 $\pm$ 0.104 <sup>ab,AC</sup>	0.466 $\pm$ 0.057 <sup>b,A</sup>	3.170 $\pm$ 0.161 <sup>b,B</sup>	1.114 $\pm$ 0.120 <sup>b,C</sup>	0.299 $\pm$ 0.097 <sup>b,A</sup>	1.112 $\pm$ 0.093 <sup>c,B</sup>	0.700 $\pm$ 0.000
Phenethyl alcohol	1930	1929	MS, KI, Co	1.010 $\pm$ 0.061 <sup>a,A</sup>	2.399 $\pm$ 0.122 <sup>a,B</sup>	1.150 $\pm$ 0.124 <sup>a,A</sup>	0.199 $\pm$ 0.025 <sup>b,A</sup>	2.604 $\pm$ 0.135 <sup>b,B</sup>	1.148 $\pm$ 0.124 <sup>a,C</sup>	N.D.	0.835 $\pm$ 0.070 <sup>c,A</sup>	0.400 $\pm$ 0.000
<b>Total alcohols</b>				9.184 $\pm$ 0.553	11.628 $\pm$ 0.592	4.527 $\pm$ 0.487	6.076 $\pm$ 0.750	15.048 $\pm$ 0.768	4.705 $\pm$ 0.507	2.277 $\pm$ 0.096	7.643 $\pm$ 0.638	3.000 $\pm$ 0.300
<b>Total volatiles</b>				9.915 $\pm$ 0.597 <sup>a,A</sup>	12.093 $\pm$ 0.616 <sup>a,B</sup>	5.017 $\pm$ 0.540 <sup>a,C</sup>	6.695 $\pm$ 0.827 <sup>b,A</sup>	16.039 $\pm$ 0.818 <sup>b,B</sup>	5.237 $\pm$ 0.564 <sup>a,C</sup>	2.869 $\pm$ 0.242 <sup>c,A</sup>	8.201 $\pm$ 0.684 <sup>c,B</sup>	7.700 $\pm$ 0.800

<sup>1)</sup> All concentration values are presented as mean  $\pm$  standard deviation. <sup>2)</sup> MS, identification of mass spectrum with reference to the NIST mass spectrum library; Kovats retention index on DB-WAX; KI (Ref), Retention index on DB-WAX in the NIST database. Mean values with different letters (a, b, c – extraction temperature/A, B, C – particle size) in the same horizontal line indicate significant differences according to Duncan's test ( $p < 0.05$ ). N.D.: not detected

Octanal is characterized by a fatty odour and benzaldehyde had a bitter almond odour (28). Among the aldehyde compounds, benzaldehyde was usually detected more often than the other compounds. Octanal was not detected at 50°C for the 500–700  $\mu$ m size and at 70°C for the 355–500  $\mu$ m size. The alcohol compounds included 2-methyl-1-butanol, 1-pentanol, 1-hexanol, 1-octanol, 1-nonanol, benzyl alcohol, and phenethyl alcohol. Among the alcohol compounds, 2-methyl-1-butanol was predominant. Phenethyl alcohol was not detected at 70°C for the 355–500  $\mu$ m size. The highest concentration of volatile compounds in mung bean was extracted at 60°C with a particle size of 500–710  $\mu$ m, while the lowest concentration of volatile compounds was extracted at 70°C with a particle size of 355–500  $\mu$ m. This shows the same tendency as the result that when fine particles (355–510  $\mu$ m) are used in the espresso method, the penetration rate may be slow because the gaps between the ground coffee powder are small and the porosity is low (33). Also, a previous study has suggested that this is because the cellular structure of coffee beans opens up, allowing higher levels of residual levels of volatile compounds to be released (34).

A total of three aldehydes and seven alcohols were identified in soybean extracts (Table 3), with alcohols being predominantly detected. The aldehyde compounds extracted from soybean by DRP-LLE included hexanal, nonanal, and benzaldehyde. Among these aldehyde compounds, hexanal was usually detected more than the other compounds. Benzaldehyde was not detected at 70°C for whole size. The alcohol compounds detected included 2-methyl-1-butanol, 1-hexanol, cis-3-hexen-1-ol, 3-octanol, 1-nonanol, benzyl alcohol, and phenethyl alcohol. Among the alcohol compounds, 1-hexanol was usually

detected more than the other compounds. 1-Nonanol was not detected at 50°C for the 500–710 µm size. The sums of the peak area ratios of nine samples are presented in Table 3. The highest concentration of volatile compounds in soybean was extracted at 50°C with a particle size of 355–500 µm. However, the lowest concentration of volatile compounds in soybean was extracted at 60°C with whole size. This result is similar to the tendency for black bean samples.

Table 3  
Concentrations of volatile compounds identified in soybean (*Glycine max L.*) extracts with DRP-LLE

Compounds	KI	KI (Ref.)	Identification	Peak area ratio <sup>1)</sup>								
				Distillation at 50°C			Distillation at 60°C			Distillation at 70°C		
				355–500 µm	500–710 µm	Whole size	355–500 µm	500–710 µm	Whole size	355–500 µm	500–710 µm	Whole size
<b>Aldehydes</b>												
Hexanal	1074	1084	MS, KI, Co	2.127 ± 0.065a,A	1.004 ± 0.030a,B	0.289 ± 0.009a,C	1.255 ± 0.037b,A	1.495 ± 0.044b,B	0.148 ± 0.004b,C	1.147 ± 0.035c,A	2.003 ± 0.062c,B	
Nonanal	1412	1395	MS, KI, Co	0.414 ± 0.012a,A	0.190 ± 0.006a,B	0.218 ± 0.007a,C	0.214 ± 0.007b,A	0.204 ± 0.006bc,AB	0.233 ± 0.007b,C	0.445 ± 0.013c,A	0.191 ± 0.006ab,BC	
Benzaldehyde	1521	1527	MS, KI, Co	0.678 ± 0.020a,A	0.646 ± 0.020a,A	0.201 ± 0.006a,B	0.599 ± 0.019b,A	1.271 ± 0.040b,B	0.144 ± 0.004b,C	0.606 ± 0.018b,A	0.881 ± 0.026c,B	
<b>Total aldehydes</b>				3.219 ± 0.098	1.840 ± 0.055	0.708 ± 0.022	2.067 ± 0.063	2.969 ± 0.090	0.525 ± 0.016	2.199 ± 0.067	3.075 ± 0.093	
<b>Alcohols</b>												
2-Methyl-1-butanol	1194	1208	MS, KI, Co	5.259 ± 0.161a,A	1.963 ± 0.060a,B	1.361 ± 0.042a,C	0.982 ± 0.030b,A	1.716 ± 0.053b,B	0.512 ± 0.016b,C	1.183 ± 0.036c,A	1.020 ± 0.031c,B	
1-Hexanol	1368	1360	MS, KI, Co	4.112 ± 0.127a,A	3.000 ± 0.092a,B	1.606 ± 0.049a,C	3.266 ± 0.100b,A	4.612 ± 0.141b,B	0.982 ± 0.030b,C	4.070 ± 0.125a,A	3.503 ± 0.106c,B	
cis-3-Hexen-1-ol	1401	1391	MS, KI, Co	0.778 ± 0.024a,A	0.251 ± 0.008a,B	0.109 ± 0.003a,C	0.398 ± 0.012b,A	0.333 ± 0.011ab,AB	0.026 ± 0.001a,C	0.585 ± 0.018c,A	0.299 ± 0.010ab,B	
3-Octanol	1417	1394	MS, KI, Co	0.718 ± 0.022a,A	0.653 ± 0.020a,B	0.479 ± 0.015a,C	0.400 ± 0.012b,A	0.845 ± 0.026b,B	0.334 ± 0.010b,C	0.471 ± 0.014c,A	0.538 ± 0.016c,B	
1-Nonanol	1672	1666	MS, KI, Co	0.255 ± 0.008a,A	N.D.	0.106 ± 0.003a,C	0.138 ± 0.004b,A	0.246 ± 0.007a,C	0.175 ± 0.005b,B	0.175 ± 0.005c,A	0.174 ± 0.005b,A	
Benzyl alcohol	1891	1880	MS, KI, Co	1.529 ± 0.047a,A	1.052 ± 0.033a,B	0.556 ± 0.017a,C	1.141 ± 0.034b,A	1.508 ± 0.045b,B	0.678 ± 0.021b,C	1.062 ± 0.033c,A	0.741 ± 0.023c,B	
Phenethyl-alcohol	1930	1929	MS, KI, Co	0.239 ± 0.007a,A	0.229 ± 0.007a,B	0.053 ± 0.002a,C	0.189 ± 0.006b,A	0.288 ± 0.009b,B	0.041 ± 0.001b,C	0.118 ± 0.004c,A	0.123 ± 0.004c,A	
<b>Total alcohols</b>				12.890 ± 0.394	7.148 ± 0.219	4.269 ± 0.130	6.514 ± 0.199	9.547 ± 0.291	2.747 ± 0.084	7.665 ± 0.234	6.397 ± 0.195	
<b>Total volatiles</b>				16.109 ± 0.492a,A	8.988 ± 0.275ab,B	4.977 ± 0.152a,C	8.581 ± 0.261b,A	12.517 ± 0.381c,B	3.271 ± 0.099b,C	9.864 ± 0.301c,A	9.472 ± 0.289b,AB	

<sup>1)</sup> All concentration values are presented as mean ± standard deviation. <sup>2)</sup> MS, identification of mass spectrum with reference to the NIST mass spectrum library; Kovats retention index on DB-WAX; KI (Ref), Retention index on DB-WAX in the NIST database. Mean values with different letters (a, b, c – extraction temperature; A – B – particle size) in the same horizontal line indicate significant differences according to Duncan's test (p < 0.05). N.D.: not detected

### 3.2. Isolation of volatile compounds by HWE and HS-SPME

A total of three aldehydes, five alcohols, and two ketones were identified in black bean extracts by HWE (Table 4). The 10 major volatile compounds were selected and analysed. Among the three groups of volatile compounds, alcohols were detected the most, followed by aldehydes and ketones. Aldehyde compounds detected by HS-SPME in black bean extracts included hexanal, nonanal, and benzaldehyde. Among the aldehyde compounds, hexanal was detected more often than the other compounds. The alcohol compounds included 1-hexanol, 3-octanol, 1-octen-3-ol, benzyl alcohol, and phenethyl alcohol, of which 1-octen-3-ol was predominantly detected. The ketone compounds detected were 2-heptanone and 3-octanone. 2-Heptanone had a soapy odour while 3-octanone had a herbaceous and buttery odour (27, 35). The sums of the peak area ratios of nine samples are presented in Table 4. The lower the incubation temperature, the higher the concentration of volatile compounds.

Table 4  
Concentrations of volatile compounds identified in black bean (*Phaseolus vulgaris* L.) extracts with HWE

Compounds	KI	KI (Ref.)	Identification	Peak area ratio <sup>1)</sup>								
				Distillation at 70°C			Distillation at 80°C			Distillation at 90°C		
				355– 500 μ	500– 710 μ	Whole size	355– 500 μ	500– 710 μ	Whole size	355– 500 μ	500– 710 μ	Whole size
<b>Aldehydes</b>												
Hexanal	1074	1084	MS, KI, Co	2.173 ± 0.003 <sup>a,A</sup>	4.292 ± 0.004 <sup>a,B</sup>	0.461 ± 0.003 <sup>a,C</sup>	1.125 ± 0.003 <sup>b,A</sup>	2.354 ± 0.001 <sup>b,B</sup>	0.656 ± 0.003 <sup>b,C</sup>	0.659 ± 0.002 <sup>c,A</sup>	1.119 ± 0.002 <sup>c,B</sup>	0.314 ± 0.001 <sup>c,C</sup>
Nonanal	1412	1395	MS, KI, Co	0.223 ± 0.001 <sup>a,A</sup>	0.308 ± 0.001 <sup>a,B</sup>	0.036 ± 0.001 <sup>a,C</sup>	0.152 ± 0.001 <sup>b,A</sup>	0.198 ± 0.002 <sup>b,B</sup>	0.366 ± 0.001 <sup>b,C</sup>	0.102 ± 0.002 <sup>c,A</sup>	0.152 ± 0.001 <sup>c,B</sup>	0.078 ± 0.002 <sup>c,C</sup>
Benzaldehyde	1521	1527	MS, KI, Co	1.633 ± 0.006 <sup>a,A</sup>	1.937 ± 0.003 <sup>a,B</sup>	0.375 ± 0.003 <sup>a,C</sup>	1.291 ± 0.004 <sup>b,A</sup>	1.749 ± 0.002 <sup>b,B</sup>	0.494 ± 0.002 <sup>b,C</sup>	1.171 ± 0.008 <sup>c,A</sup>	1.271 ± 0.004 <sup>c,B</sup>	0.687 ± 0.002 <sup>c,C</sup>
<b>Total aldehydes</b>				4.029 ± 0.006	6.538 ± 0.004	0.871 ± 0.005	2.569 ± 0.003	4.298 ± 0.002	1.186 ± 0.002	1.932 ± 0.001	2.543 ± 0.005	1.076 ± 0.005
<b>Alcohols</b>												
1-Hexanol	1363	1360	MS, KI, Co	1.583 ± 0.001 <sup>a,A</sup>	1.784 ± 0.002 <sup>a,B</sup>	0.545 ± 0.004 <sup>a,C</sup>	0.459 ± 0.001 <sup>b,A</sup>	0.998 ± 0.003 <sup>b,B</sup>	0.416 ± 0.005 <sup>b,C</sup>	0.485 ± 0.002 <sup>c,A</sup>	0.458 ± 0.005 <sup>c,B</sup>	0.194 ± 0.004 <sup>c,C</sup>
3-Octanol	1400	1394	MS, KI, Co	0.052 ± 0.003 <sup>a,A</sup>	0.111 ± 0.002 <sup>a,B</sup>	0.106 ± 0.001 <sup>a,C</sup>	0.041 ± 0.002 <sup>b,A</sup>	0.077 ± 0.004 <sup>b,B</sup>	0.076 ± 0.004 <sup>b,C</sup>	0.019 ± 0.001 <sup>c,A</sup>	0.041 ± 0.003 <sup>c,B</sup>	0.044 ± 0.002 <sup>c,C</sup>
1-Octen-3-ol	1458	1445	MS, KI, Co	7.966 ± 0.002 <sup>a,A</sup>	12.145 ± 0.008 <sup>a,B</sup>	7.025 ± 0.010 <sup>a,C</sup>	3.781 ± 0.002 <sup>b,A</sup>	7.308 ± 0.004 <sup>b,B</sup>	5.664 ± 0.008 <sup>b,C</sup>	2.713 ± 0.004 <sup>c,A</sup>	3.573 ± 0.003 <sup>c,B</sup>	3.321 ± 0.004 <sup>c,C</sup>
Benzyl alcohol	1886	1880	MS, KI, Co	0.498 ± 0.004 <sup>a,A</sup>	0.171 ± 0.003 <sup>a,B</sup>	0.016 ± 0.001 <sup>a,C</sup>	0.106 ± 0.004 <sup>b,A</sup>	0.149 ± 0.001 <sup>b,B</sup>	0.012 ± 0.001 <sup>b,C</sup>	0.142 ± 0.003 <sup>c,A</sup>	0.110 ± 0.004 <sup>c,B</sup>	0.016 ± 0.002 <sup>c,C</sup>
Phenethyl-alcohol	1912	1929	MS, KI, Co	0.014 ± 0.004 <sup>a,A</sup>	0.019 ± 0.001 <sup>a,B</sup>	0.120 ± 0.002 <sup>a,C</sup>	0.015 ± 0.004 <sup>b,A</sup>	0.021 ± 0.001 <sup>b,B</sup>	0.076 ± 0.002 <sup>b,C</sup>	0.010 ± 0.002 <sup>c,A</sup>	0.016 ± 0.004 <sup>c,B</sup>	0.067 ± 0.001 <sup>c,C</sup>
<b>Total alcohols</b>				10.113 ± ± 0.003	14.226 ± ± 0.004	7.813 ± 0.007	4.401 ± 0.003	8.552 ± 0.005	6.245 ± 0.009	3.368 ± 0.007	4.198 ± 0.003	3.643 ± 0.005
<b>Ketones</b>												
2-Heptanone	1193	1189	MS, KI, Co	0.284 ± 0.001 <sup>a,A</sup>	0.474 ± 0.001 <sup>a,B</sup>	0.024 ± 0.001 <sup>a,C</sup>	0.228 ± 0.002 <sup>b,A</sup>	0.363 ± 0.006 <sup>b,B</sup>	0.085 ± 0.002 <sup>b,C</sup>	0.130 ± 0.001 <sup>c,A</sup>	0.226 ± 0.003 <sup>c,B</sup>	0.044 ± 0.007 <sup>c,C</sup>
3-Octanone	1266	1261	MS, KI, Co	0.207 ± 0.001 <sup>a,A</sup>	0.667 ± 0.003 <sup>a,B</sup>	0.087 ± 0.002 <sup>a,C</sup>	0.131 ± 0.003 <sup>b,A</sup>	0.327 ± 0.001 <sup>b,B</sup>	0.052 ± 0.004 <sup>b,C</sup>	0.070 ± 0.003 <sup>c,A</sup>	0.128 ± 0.001 <sup>c,B</sup>	0.024 ± 0.002 <sup>c,C</sup>
<b>Total ketones</b>				0.493 ± 0.001	1.141 ± 0.001	0.111 ± 0.001	0.358 ± 0.001	0.689 ± 0.008	0.137 ± 0.003	0.199 ± 0.003	0.355 ± 0.001	0.068 ± 0.009
<b>Total volatiles</b>				14.632 ± 0.004 <sup>a,A</sup>	21.904 ± 0.004 <sup>a,B</sup>	8.795 ± 0.001 <sup>a,C</sup>	7.328 ± 0.006 <sup>b,A</sup>	13.539 ± 0.004 <sup>b,B</sup>	7.568 ± 0.006 <sup>b,C</sup>	5.503 ± 0.004 <sup>c,A</sup>	7.095 ± 0.004 <sup>c,B</sup>	4.786 ± 0.004 <sup>c,C</sup>

<sup>1)</sup> All concentration values are presented as mean ± standard deviation. <sup>2)</sup> MS, identification of mass spectrum with reference to the NIST mass spectrum library, <sup>3)</sup> Kovats retention index on DB-WAX; KI (Ref), Retention index on DB-WAX in the NIST database. Mean values with different letters (a, b, c – extraction temperature/A, B, C – particle size) in the same horizontal line indicate significant differences according to Duncan's test (p < 0.05). N.D.: not detected

A total of three aldehydes, six alcohols, and one ketone were identified in mung bean extracts (Table 5). Among the three groups of volatile compounds, alcohols were detected the most, followed by aldehydes and ketones. The aldehyde compounds detected in mung bean extracts by HS-SPME included hexanal, nonanal, and benzaldehyde, with benzaldehyde being detected more than the other compounds. The alcohol compounds included 1-hexanol, cis-3-hexen-1-ol, 1-octen-3-ol, 1-octanol, benzyl alcohol, and phenethyl alcohol, with 1-hexanol being detected in the highest concentration. The ketone compound detected was 2-heptanone. The sums of the peak area ratios of nine samples are presented in Table 5. The lower incubation temperature, the higher the concentration of volatile compounds, similar to the tendency for black bean.

Table 5  
Concentrations of volatile compounds identified in mung bean (*Vigna radiata* L.) extracts with HWE

Compounds	KI	KI (Ref.)	Identification	Peak area ratio <sup>1)</sup>								
				Distillation at 70°C			Distillation at 80°C			Distillation at 90°C		
				355– 500 $\mu$	500– 710 $\mu$	Whole size	355– 500 $\mu$	500– 710 $\mu$	Whole size	355– 500 $\mu$	500– 710 $\mu$	Whole size
<b>Aldehydes</b>												
Hexanal	1092	1084	MS, KI, Co	1.876 ± 0.008 <sup>a,A</sup>	4.746 ± 0.001 <sup>a,B</sup>	0.597 ± 0.001 <sup>a,C</sup>	0.472 ± 0.001 <sup>b,A</sup>	1.918 ± 0.009 <sup>b,B</sup>	0.406 ± 0.001 <sup>b,A</sup>	0.437 ± 0.001 <sup>c,A</sup>	1.091 ± 0.020 <sup>c,B</sup>	0.383 ± 0.002 <sup>c,A</sup>
Nonanal	1404	1395	MS, KI, Co	0.712 ± 0.001 <sup>a,A</sup>	2.189 ± 0.003 <sup>a,B</sup>	0.471 ± 0.002 <sup>a,C</sup>	0.222 ± 0.001 <sup>b,A</sup>	1.671 ± 0.065 <sup>b,B</sup>	0.491 ± 0.002 <sup>a,C</sup>	0.153 ± 0.002 <sup>c,A</sup>	1.822 ± 0.080 <sup>c,B</sup>	0.245 ± 0.001 <sup>b,C</sup>
Benzaldehyde	1540	1527	MS, KI, Co	3.416 ± 0.004 <sup>a,A</sup>	4.548 ± 0.003 <sup>a,B</sup>	1.706 ± 0.002 <sup>a,C</sup>	1.196 ± 0.003 <sup>b,A</sup>	3.159 ± 0.055 <sup>b,B</sup>	2.155 ± 0.001 <sup>b,C</sup>	1.062 ± 0.002 <sup>c,A</sup>	3.017 ± 0.094 <sup>c,B</sup>	4.834 ± 0.001 <sup>c,C</sup>
<b>Total aldehydes</b>				6.003 ± 0.011	11.483 ± 0.001	2.773 ± 0.002	1.891 ± 0.006	6.747 ± 0.085	3.051 ± 0.001	1.650 ± 0.004	5.930 ± 0.038	5.461 ± 0.002
<b>Alcohols</b>												
1-Hexanol	1363	1360	MS, KI, Co	3.723 ± 0.005 <sup>a,A</sup>	2.383 ± 0.001 <sup>a,B</sup>	3.298 ± 0.001 <sup>a,C</sup>	0.817 ± 0.003 <sup>b,A</sup>	0.976 ± 0.034 <sup>b,B</sup>	2.034 ± 0.005 <sup>b,C</sup>	0.554 ± 0.005 <sup>c,A</sup>	0.547 ± 0.003 <sup>c,A</sup>	0.913 ± 0.003 <sup>c,B</sup>
cis-3-Hexen-1-ol	1393	1391	MS, KI, Co	0.932 ± 0.001 <sup>a,A</sup>	0.435 ± 0.001 <sup>a,B</sup>	0.346 ± 0.001 <sup>a,C</sup>	0.182 ± 0.002 <sup>b,A</sup>	0.186 ± 0.003 <sup>b,B</sup>	0.246 ± 0.001 <sup>b,C</sup>	0.145 ± 0.002 <sup>c,A</sup>	0.102 ± 0.004 <sup>c,B</sup>	0.081 ± 0.001 <sup>c,C</sup>
1-Octen-3-ol	1459	1445	MS, KI, Co	0.910 ± 0.002 <sup>a,A</sup>	2.234 ± 0.002 <sup>a,B</sup>	2.092 ± 0.006 <sup>a,C</sup>	0.284 ± 0.004 <sup>b,A</sup>	1.095 ± 0.010 <sup>b,B</sup>	1.703 ± 0.002 <sup>b,C</sup>	0.227 ± 0.002 <sup>c,A</sup>	0.661 ± 0.033 <sup>c,B</sup>	0.984 ± 0.001 <sup>c,C</sup>
1-Octanol	1569	1563	MS, KI, Co	0.747 ± 0.001 <sup>a,A</sup>	1.507 ± 0.001 <sup>a,B</sup>	0.522 ± 0.001 <sup>a,C</sup>	0.214 ± 0.002 <sup>b,A</sup>	1.107 ± 0.045 <sup>b,B</sup>	0.457 ± 0.002 <sup>b,C</sup>	0.186 ± 0.003 <sup>b,A</sup>	0.788 ± 0.020 <sup>c,B</sup>	0.635 ± 0.002 <sup>c,C</sup>
Benzyl alcohol	1887	1880	MS, KI, Co	1.683 ± 0.003 <sup>a,A</sup>	1.224 ± 0.005 <sup>a,B</sup>	1.136 ± 0.001 <sup>a,C</sup>	0.503 ± 0.001 <sup>b,A</sup>	0.787 ± 0.049 <sup>b,B</sup>	1.245 ± 0.001 <sup>b,C</sup>	0.396 ± 0.004 <sup>c,A</sup>	0.619 ± 0.013 <sup>c,B</sup>	1.620 ± 0.001 <sup>c,C</sup>
Phenethyl-alcohol	1913	1929	MS, KI, Co	0.232 ± 0.001 <sup>a,A</sup>	0.162 ± 0.001 <sup>a,B</sup>	2.073 ± 0.006 <sup>a,C</sup>	0.084 ± 0.005 <sup>b,A</sup>	0.124 ± 0.006 <sup>b,B</sup>	2.132 ± 0.006 <sup>b,C</sup>	0.105 ± 0.002 <sup>c,A</sup>	0.122 ± 0.012 <sup>b,B</sup>	0.747 ± 0.002 <sup>c,C</sup>
<b>Total alcohols</b>				8.227 ± 0.004	7.944 ± 0.006	9.468 ± 0.008	2.083 ± 0.009	4.275 ± 0.041	7.816 ± 0.010	1.613 ± 0.001	2.839 ± 0.054	4.962 ± 0.004
<b>Ketones</b>												
2-Heptanone	1193	1189	MS, KI, Co	0.323 ± 0.004 <sup>a,A</sup>	2.425 ± 0.002 <sup>a,B</sup>	0.987 ± 0.001 <sup>a,C</sup>	0.147 ± 0.001 <sup>b,A</sup>	1.134 ± 0.050 <sup>b,B</sup>	0.554 ± 0.001 <sup>b,C</sup>	0.137 ± 0.001 <sup>b,A</sup>	0.764 ± 0.012 <sup>c,B</sup>	0.192 ± 0.001 <sup>c,C</sup>
<b>Total ketones</b>				0.323 ± 0.004 <sup>a,A</sup>	2.425 ± 0.002 <sup>a,B</sup>	0.987 ± 0.001 <sup>a,C</sup>	0.147 ± 0.001 <sup>b,A</sup>	1.134 ± 0.050 <sup>b,B</sup>	0.554 ± 0.001 <sup>b,C</sup>	0.137 ± 0.001 <sup>b,A</sup>	0.764 ± 0.012 <sup>c,B</sup>	0.192 ± 0.001 <sup>c,C</sup>
<b>Total volatiles</b>				14.554 ± 0.003 <sup>a,A</sup>	21.851 ± 0.009 <sup>a,B</sup>	13.228 ± 0.009 <sup>a,C</sup>	4.121 ± 0.006 <sup>b,A</sup>	12.156 ± 0.013 <sup>b,B</sup>	11.421 ± 0.010 <sup>b,C</sup>	3.400 ± 0.004 <sup>c,A</sup>	9.533 ± 0.009 <sup>c,B</sup>	10.616 ± 0.005 <sup>c,C</sup>

<sup>1)</sup> All concentration values are presented as mean ± standard deviation. <sup>2)</sup> MS, identification of mass spectrum with reference to the NIST mass spectrum library, <sup>3)</sup> Kovats retention index on DB-WAX; KI (Ref), Retention index on DB-WAX in the NIST database. Mean values with different letters (a, b, c – extraction temperature/A, B, C – particle size) in the same horizontal line indicate significant differences according to Duncan's test (p < 0.05). N.D.: not detected

A total of two aldehydes and eight alcohols were identified in soybean (Table 6). Among the two groups of volatile compounds, alcohols were detected the most, followed by aldehydes. The aldehyde compounds detected by HS-SPME included hexanal and benzaldehyde. Among aldehyde compounds, hexanal was detected more than benzaldehyde. The alcohol compounds included 2-methyl-1-butanol, 1-hexanol, cis-3-hexen-1-ol, 3-octanol, 1-octen-3-ol, 1-octanol, benzyl alcohol and phenethyl alcohol, with 1-octen-3-ol being detected in the highest concentration. The sums of the peak area ratios of nine samples are presented in Table 6. The lower the incubation temperature, the higher the concentration of volatile compounds, similar to the tendency for black bean and mung bean. Previous studies have demonstrated that HS-SPME at a low incubation temperature is the most efficient method when analyzing the volatile compounds of soymilk made from soy beans (36).



Table 6  
Concentrations of volatile compounds identified in soybean (*Glycine max L.*) extracts with HWE

Compounds	KI	KI (Ref.)	Identification	Peak area ratio <sup>1)</sup>								
				Distillation at 70°C			Distillation at 80°C			Distillation at 90°C		
				355– 500 μ	500– 710 μ	Whole size	355– 500 μ	500– 710 μ	Whole size	355– 500 μ	500– 710 μ	Whole size
<b>Aldehydes</b>												
Hexanal	1092	1084	MS, KI, Co	3.583 ± 0.002 <sup>a,A</sup>	2.177 ± 0.001 <sup>a,B</sup>	1.267 ± 0.001 <sup>a,C</sup>	1.533 ± 0.001 <sup>b,A</sup>	1.366 ± 0.007 <sup>b,B</sup>	0.144 ± 0.000 <sup>b,C</sup>	0.845 ± 0.002 <sup>c,A</sup>	0.778 ± 0.016 <sup>c,B</sup>	0.133 ± 0.003 <sup>c,C</sup>
Benzaldehyde	1540	1527	MS, KI, Co	1.863 ± 0.024 <sup>a,A</sup>	0.847 ± 0.002 <sup>a,B</sup>	0.979 ± 0.001 <sup>a,C</sup>	1.550 ± 0.014 <sup>b,A</sup>	0.658 ± 0.030 <sup>b,B</sup>	0.340 ± 0.001 <sup>b,C</sup>	1.162 ± 0.004 <sup>c,A</sup>	0.393 ± 0.003 <sup>c,B</sup>	0.531 ± 0.002 <sup>c,C</sup>
<b>Total aldehydes</b>				5.446 ± 0.022	3.023 ± 0.003	2.246 ± 0.001	3.083 ± 0.013	2.025 ± 0.010	0.484 ± 0.002	2.008 ± 0.003	1.170 ± 0.016	0.665 ± 0.004
<b>Alcohols</b>												
2-Methyl-1-butanol	1215	1208	MS, KI, Co	0.033 ± 0.001 <sup>a,A</sup>	0.021 ± 0.002 <sup>a,B</sup>	0.066 ± 0.001 <sup>a,C</sup>	0.020 ± 0.001 <sup>b,A</sup>	0.012 ± 0.000 <sup>b,B</sup>	0.071 ± 0.001 <sup>b,C</sup>	0.011 ± 0.004 <sup>c,A</sup>	0.007 ± 0.002 <sup>c,B</sup>	0.052 ± 0.002 <sup>c,C</sup>
1-Hexanol	1363	1360	MS, KI, Co	1.975 ± 0.007 <sup>a,A</sup>	0.233 ± 0.003 <sup>a,B</sup>	1.148 ± 0.001 <sup>a,C</sup>	0.989 ± 0.002 <sup>b,A</sup>	0.146 ± 0.002 <sup>b,B</sup>	0.926 ± 0.004 <sup>b,C</sup>	0.531 ± 0.001 <sup>c,A</sup>	0.110 ± 0.002 <sup>c,B</sup>	0.588 ± 0.004 <sup>c,C</sup>
cis-3-Hexen-1-ol	1393	1391	MS, KI, Co	0.039 ± 0.001 <sup>a,A</sup>	0.011 ± 0.003 <sup>a,B</sup>	0.083 ± 0.001 <sup>a,C</sup>	0.019 ± 0.001 <sup>b,A</sup>	0.007 ± 0.001 <sup>b,B</sup>	0.070 ± 0.002 <sup>b,C</sup>	0.009 ± 0.000 <sup>c,A</sup>	0.003 ± 0.000 <sup>c,B</sup>	0.048 ± 0.002 <sup>c,C</sup>
3-Octanol	1401	1394	MS, KI, Co	0.042 ± 0.001 <sup>a,A</sup>	0.022 ± 0.001 <sup>a,B</sup>	0.297 ± 0.001 <sup>a,C</sup>	0.045 ± 0.003 <sup>b,A</sup>	0.028 ± 0.001 <sup>b,B</sup>	0.249 ± 0.001 <sup>b,C</sup>	0.032 ± 0.001 <sup>c,A</sup>	0.016 ± 0.001 <sup>c,B</sup>	0.189 ± 0.003 <sup>c,C</sup>
1-Octen-3-ol	1459	1445	MS, KI, Co	4.225 ± 0.035 <sup>a,A</sup>	2.241 ± 0.010 <sup>a,B</sup>	8.894 ± 0.002 <sup>a,C</sup>	2.062 ± 0.015 <sup>b,A</sup>	1.305 ± 0.017 <sup>b,B</sup>	5.835 ± 0.007 <sup>b,C</sup>	1.398 ± 0.005 <sup>c,A</sup>	0.767 ± 0.003 <sup>c,B</sup>	4.213 ± 0.007 <sup>c,C</sup>
1-Octanol	1569	1563	MS, KI, Co	0.249 ± 0.007 <sup>a,A</sup>	0.274 ± 0.002 <sup>a,B</sup>	0.107 ± 0.002 <sup>a,C</sup>	0.158 ± 0.002 <sup>b,A</sup>	0.174 ± 0.004 <sup>b,B</sup>	0.060 ± 0.003 <sup>b,C</sup>	0.109 ± 0.001 <sup>c,A</sup>	0.093 ± 0.002 <sup>c,B</sup>	0.050 ± 0.001 <sup>c,C</sup>
Benzyl alcohol	1887	1880	MS, KI, Co	0.438 ± 0.003 <sup>a,A</sup>	0.049 ± 0.002 <sup>a,B</sup>	0.036 ± 0.001 <sup>a,C</sup>	0.423 ± 0.022 <sup>b,A</sup>	0.028 ± 0.001 <sup>b,B</sup>	0.044 ± 0.002 <sup>b,C</sup>	0.269 ± 0.002 <sup>c,A</sup>	0.026 ± 0.002 <sup>c,B</sup>	0.046 ± 0.001 <sup>c,C</sup>
Phenethyl-alcohol	1913	1895	MS, KI, Co	0.035 ± 0.002 <sup>a,A</sup>	0.010 ± 0.001 <sup>a,B</sup>	0.057 ± 0.001 <sup>a,C</sup>	0.039 ± 0.010 <sup>b,A</sup>	0.008 ± 0.003 <sup>b,B</sup>	0.098 ± 0.003 <sup>b,C</sup>	0.022 ± 0.002 <sup>c,A</sup>	0.007 ± 0.000 <sup>c,B</sup>	0.091 ± 0.004 <sup>c,C</sup>
<b>Total alcohols</b>				7.034 ± 0.021	2.838 ± 0.009	10.687 ± 0.002	3.753 ± 0.006	1.695 ± 0.014	7.354 ± 0.007	2.381 ± 0.005	1.021 ± 0.003	5.276 ± 0.001
<b>Total volatiles</b>				12.481 ± 0.005 <sup>a,A</sup>	5.883 ± 0.007 <sup>a,B</sup>	12.933 ± 0.003 <sup>a,C</sup>	6.836 ± 0.007 <sup>b,A</sup>	3.732 ± 0.005 <sup>b,B</sup>	7.838 ± 0.006 <sup>b,C</sup>	4.388 ± 0.002 <sup>c,A</sup>	2.197 ± 0.011 <sup>c,B</sup>	5.941 ± 0.005 <sup>c,C</sup>

<sup>1)</sup> All concentration values are presented as mean ± standard deviation. <sup>2)</sup> MS, identification of mass spectrum with reference to the NIST mass spectrum library, <sup>3)</sup> Kovats retention index on DB-WAX; KI (Ref), Retention index on DB-WAX in the NIST database. Mean values with different letters (a, b, c – extraction temperature/A, B, C – particle size) in the same horizontal line indicate significant differences according to Duncan's test (p < 0.05). N.D.: not detect

Among the total volatile compounds of extracts of various beans detected by DRP-LLE, black bean showed the highest concentration of volatile compounds, followed by soybean and mung bean. However, mung bean showed the highest concentration of volatile compounds in HS-SPME. The total volatile compound contents of black bean and soybean extracts detected by DRP-LLE were more than those detected by HS-SPME. However, the volatile compound content in mung bean extracts detected by DRP-LLE was less than that detected by HS-SPME. In conclusion, DRP-LLE for black beans and soybeans and HWE for mung beans were the highest concentration of volatile compounds. The difference in the concentration of volatile compounds detected in different species may be due to differences in the internal structure of beans and the distribution of compound components (34).

#### 4. Concluding Remarks

This study measured the volatile compounds in extracts of various beans (black bean, mung bean, soybean). The amounts of volatile compounds obtained by two extraction and analysis methods were assessed in relation to the particle size and temperature. The main volatile compounds in 54 bean extracts were hexanal, 2-methyl-1-butanol, 1-hexanol, 1-octen-3-ol, and benzaldehyde. The total volatile compound content of black bean extracts was the highest when extraction was performed with DRP at 60°C with a particle size of 355–500 μm. The total volatile compound content of mung bean extracts was the highest when extraction was performed with HWE at 50°C with a particle size of 500–710 μm. The total volatile compound content of soybean was the highest when extraction was performed with DRP at 70°C with a particle size of 355–500 μm. The total volatile compound contents of black bean and soybean extracts obtained with DRP were higher than those obtained with HWE, while the reverse was true for the mung bean extract. On the basis of these results, optimal

conditions for particle size, extraction temperature, and extraction method can be used to maximize the volatile compound content and to produce foods with various bean flavourings.

## Declarations

- Ethics approval and consent to participate: Not applicable
- Consent for publication: Not applicable
- Availability of data and material: The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.
- Competing interests: The authors declare that they have no conflicts of interest.
- Funding: This study was supported by the Basic Science Research Program through the National Research Foundation of Korea [grant number NRF, 2021R1A2B5B01002296] and Korea Institute of Planning and Evaluation for Technology in Food, Agriculture, Forestry and Fisheries [IPET, No. 118012033SB010].
- Authors' contributions: E. Han: Formal analysis, Investigation, Methodology; H. Ahn: Formal analysis; S. Lee: Validation, Methodology; K.-G. Lee: Supervision; Validation; Investigation; Project administration.
- Acknowledgements: The authors would like to thank the support of Dongguk University.

## References

1. Kim SH, Kim MS, Lee MS, Park YS, Lee HJ, Kang S-a, et al. Korean diet: Characteristics and historical background. *Journal of Ethnic Foods*. 2016;3(1):26-31.
2. Campos-Vega R, Reynoso-Camacho R, Pedraza-Aboytes G, Acosta-Gallegos JA, Guzman-Maldonado SH, Paredes-Lopez O, et al. Chemical Composition and In Vitro Polysaccharide Fermentation of Different Beans (*Phaseolus vulgaris* L.). *Journal of Food Science*. 2009;74(7):T59-T65.
3. Moon H-K, Moon J-N, Yoon W-J. Quality characteristics of various beans in distribution. *Journal of the East Asian Society of Dietary Life*. 2011;21(2):215-21.
4. Myung J, Hwang I. Functional components and antioxidative activities of soybean extracts. *Korea Soybean Digest*. 2008;25(1):23-9.
5. Roy M, Sarker A, Azad MAK, Shaheb MR, Hoque MM. Evaluation of antioxidant and antimicrobial properties of dark red kidney bean (*Phaseolus vulgaris*) protein hydrolysates. *Journal of Food Measurement and Characterization*. 2020;14(1):303-13.
6. Zhang X, Shang P, Qin F, Zhou Q, Gao B, Huang H, et al. Chemical composition and antioxidative and anti-inflammatory properties of ten commercial mung bean samples. *LWT-Food Science and Technology*. 2013;54(1):171-8.
7. Kitano-Okada T, Ito A, Koide A, Nakamura Y, Han KH, Shimada K, et al. Anti-obesity role of adzuki bean extract containing polyphenols: in vivo and in vitro effects. *Journal of the Science of Food and Agriculture*. 2012;92(13):2644-51.
8. Ali NM, Mohd Yusof H, Yeap S-K, Ho W-Y, Beh B-K, Long K, et al. Anti-inflammatory and antinociceptive activities of untreated, germinated, and fermented mung bean aqueous extract. *Evidence-Based Complementary and Alternative Medicine*. 2014;2014.
9. Ganesan K, Xu B. A critical review on phytochemical profile and health promoting effects of mung bean (*Vigna radiata*). *Food Science and Human Wellness*. 2018;7(1):11-33.
10. Seitz LM, Sauer DB. Off-Odors in Grains. In: Charalambous G, editor. *Developments in Food Science*. 28: Elsevier; 1992. p. 17-35.
11. Dong Y, Zhao L, Liu B, Wang Z, Jin Z, Sun H. The genetic diversity of cultivated soybean grown in China. *Theoretical and Applied Genetics*. 2004;108(5):931-6.
12. Kaplan L. What is the origin of the common bean? *Economic Botany*. 1981;35(2):240-54.
13. Namrata K, Datta J, Kumar R, Chakravarty A, Pal S. Herbicide induced changes in nutrient and antinutrient content during mung bean (*Vigna radiata* L.) seed development. *International Journal of Chemical Studies*. 2020;8(2):223-8.
14. Pongsuwan W, Fukusaki E, Bamba T, Yonetani T, Yamahara T, Kobayashi A. Prediction of Japanese green tea ranking by gas chromatography/mass spectrometry-based hydrophilic metabolite fingerprinting. *Journal of agricultural and food chemistry*. 2007;55(2):231-6.
15. Lee H, Lee G, Kim Y, Ahn H, Lee K-G. Analysis of volatile compounds and antioxidant activity in rice extracts (*Oryza sativa* L.) extracted by various conditions. *International Journal of Food Science & Technology*. 2022;57(8):5289-96.
16. De Vivo A, Tricarico MC, Sarghini F. Espresso coffee design based on non-monotonic granulometric distribution of aromatic profile. *Food Research International*. 2019;123:650-61.
17. Zhang C, Qi M, Shao Q, Zhou S, Fu R. Analysis of the volatile compounds in *Ligusticum chuanxiong* Hort. using HS-SPME-GC-MS. *Journal of Pharmaceutical and Biomedical Analysis*. 2007;44(2):464-70.
18. Kim S-Y, Kim SY, Lee SM, Lee DY, Shin BK, Kang DJ, et al. Discrimination of Cultivated Regions of Soybeans (*Glycine max*) Based on Multivariate Data Analysis of Volatile Metabolite Profiles. *Molecules*. 2020;25(3):763.
19. Fehr W, Caviness C, Burmood D, Pennington J. Stage of development descriptions for soybeans, *Glycine Max* (L.) Merrill 1. *Crop science*. 1971;11(6):929-31.
20. Lee K-G, Shibamoto T. Antioxidant properties of aroma compounds isolated from soybeans and mung beans. *Journal of Agricultural and Food Chemistry*. 2000;48(9):4290-3.

21. Boué SM, Shih BY, Carter-Wientjes CH, Cleveland TE. Identification of volatile compounds in soybean at various developmental stages using solid phase microextraction. *Journal of Agricultural and Food Chemistry*. 2003;51(17):4873-6.
22. Fischer E, Cayot N, Cachon R. Potential of Microorganisms to Decrease the “Beany” Off-Flavor: A Review. *Journal of Agricultural and Food Chemistry*. 2022;70(15):4493-508.
23. Yi C, Li Y, Zhu H, Liu Y, Quan K. Effect of *Lactobacillus plantarum* fermentation on the volatile flavors of mung beans. *LWT*. 2021;146:111434.
24. Van Ruth S, Roozen J. Influence of mastication and saliva on aroma release in a model mouth system. *Food chemistry*. 2000;71(3):339-45.
25. Barra A, Baldovini N, Loiseau A-M, Albino L, Lesecq C, Cuvelier LL. Chemical analysis of French beans (*Phaseolus vulgaris* L.) by headspace solid phase microextraction (HS-SPME) and simultaneous distillation/extraction (SDE). *Food Chemistry*. 2007;101(3):1279-84.
26. Oomah BD, Liang LS, Balasubramanian P. Volatile compounds of dry beans (*Phaseolus vulgaris* L.). *Plant Foods for Human Nutrition*. 2007;62(4):177-83.
27. Naudé Y, van Aardt M, Rohwer ER. Multi-channel open tubular traps for headspace sampling, gas chromatographic fraction collection and olfactory assessment of milk volatiles. *Journal of Chromatography A*. 2009;1216(14):2798-804.
28. Lee K-g, Shibamoto T. Determination of antioxidant potential of volatile extracts isolated from various herbs and spices. *Journal of agricultural and food chemistry*. 2002;50(17):4947-52.
29. Lee SJ, Kim MK, Lee K-G. Effect of reversed coffee grinding and roasting process on physicochemical properties including volatile compound profiles. *Innovative Food Science & Emerging Technologies*. 2017;44:97-102.
30. Phong WN, Sung B, Cao Z, Gibberd MR, Dykes GA, Payne AD, et al. Impact of different processing techniques on the key volatile profile, sensory, and consumer acceptance of black truffle (*Tuber melanosporum* Vittadini). *Journal of Food Science*. n/a(n/a).
31. Ueda S, Yamanoue M, Sirai Y, Iwamoto E. Exploring the Characteristic Aroma of Beef from Japanese Black Cattle (Japanese Wagyu) via Sensory Evaluation and Gas Chromatography-Olfactometry. *Metabolites*. 2021;11(1):56.
32. Yu J-M, Chu M, Park H, Park J, Lee K-G. Analysis of Volatile Compounds in Coffee Prepared by Various Brewing and Roasting Methods. *Foods*. 2021;10(6):1347.
33. Hyong S, Chu M, Park H, Park J, Lee K-G. Analysis of  $\alpha$ -dicarbonyl compounds and 4-methylimidazole in coffee made with various roasting and brewing conditions. *LWT*. 2021;151:112231.
34. Genovese A, De Vivo A, Aprea A, Cristina Tricarico M, Sacchi R, Sarghini F. Particle size and variety of coffee used as variables in mitigation of furan and 2-methylfuran content in espresso coffee. *Food Chemistry*. 2021;361:130037.
35. Hedges MV, Moreira MA, Steffens CA, Talamini do Amarante CV. Aromatic profile of Feijoa (*Feijoa sellowiana*) fruit in protected cultivation, at harvest and after cold storage. *Scientia Horticulturae*. 2022;293:110691.
36. Achouri A, Boye JI, Zamani Y. Identification of volatile compounds in soymilk using solid-phase microextraction-gas chromatography. *Food Chemistry*. 2006;99(4):759-66.