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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, Hyunwoo Ahn, Seongho Lee and Kwang-Geun Lee*

Abstract

In this study, 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 (HWE). 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 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.

Keywords: Black bean, Mung bean, Soybean, Extraction methods, Volatiles, GC–MS

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, soybean, black bean, and mung bean are the main legume species used in the traditional Korean diet. Moreover, Koreans commonly consume fermented products, such as doenjang and cheongkukjang using soybean, black bean, and mung bean [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

*Correspondence: kwglee@dongguk.edu

Department of Food Science and Biotechnology, Dongguk University–Seoul, 32, Dongguk-ro, Ilsandong-gu, Goyang, Gyeonggi-do 410-820, Republic of Korea

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 have reported that variation of volatile compounds is dependent on various extraction conditions, such as 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, 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].

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) [19]. 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 [20, 21]. 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 [22].

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 (HWE) [23] followed by HS-SPME were used to profile the volatile compounds of various dry beans (black bean, mung bean, and soybean).

Materials and methods

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 using a sieve shaker (CG-211–8; Chunggye Co., Korea). The selection of the sieve size was based on the previous report [22]. 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 μm 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%), γ -hexalactone (>99.0%), 1-nonanol (>99.0%), and benzyl alcohol (>99.0%) were obtained from Tokyo Chemical Industry Co. (Tokyo, Japan).

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 [24]. 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 μL of methyl cinnamate as an internal standard (100 $\mu\text{g}/\text{mL}$) and 20 μL of n-alkane standard (100 $\mu\text{g}/\text{mL}$). The mixture was steam-distilled at designated temperatures (50 $^{\circ}\text{C}$, 60 $^{\circ}\text{C}$, and 70 $^{\circ}\text{C}$) under reduced pressures (50 mmHg) until the volume of the distillate reached 200 mL. The distillate (200 mL) was extracted with 200 mL of dichloromethane (DCM) using continuous LLE for 6 h at 60 $^{\circ}\text{C}$. 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 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.

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 [25]. 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 $^{\circ}\text{C}$), 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 μL of methyl cinnamate as an internal standard (10 $\mu\text{g}/\text{mL}$) and 20 μL of n-alkane standard (100 $\mu\text{g}/\text{mL}$). The headspace vial was incubated for 10 min in a beaker filled with distilled water maintained at a designated temperature (70 $^{\circ}\text{C}$, 80 $^{\circ}\text{C}$, and 90 $^{\circ}\text{C}$) by a hot plate. After equilibrium, the SPME fiber, divinylbenzene/carboxen/poly-dimethylsiloxane (DVB/CAR/PDMS) for 40 min at 70 $^{\circ}\text{C}$, 80 $^{\circ}\text{C}$, and 90 $^{\circ}\text{C}$, respectively. After absorption, the SPME fibre was inserted into the injection port of GC at 230 $^{\circ}\text{C}$ for 10 min for desorption of volatile compounds.

Instrumental analysis

Analysis of volatile compounds in beans (black bean, mung bean, soy bean) was performed 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 μm 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 $^{\circ}\text{C}$. The oven temperature was set to 44 $^{\circ}\text{C}$ for 5 min. Subsequently, the temperature was raised to 170 $^{\circ}\text{C}$ at 3 $^{\circ}\text{C}/\text{min}$ and held for 10 min, and finally raised to 240 $^{\circ}\text{C}$ at 8 $^{\circ}\text{C}/\text{min}$ for 5 min. The ionization energy was 70 eV and scan range was 50–550 m/z.

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 (methyl cinnamate).

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).

Results and discussion

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

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 ¹	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
Aldehydes													
Hexanal	1069	1084	MS ² , KI ³ , Co ⁴	1.012 ± 0.031 ^{a,A}	0.868 ± 0.027 ^{aB}	0.214 ± 0.007 ^{aB}	3.241 ± 0.098 ^{bA}	0.927 ± 0.028 ^{aB}	0.323 ± 0.010 ^{aC}	3.877 ± 0.371 ^{b,A}	3.239 ± 0.098 ^{bA}	0.829 ± 0.026 ^{b,B}	
Nonanal	1408	1395	MS, KI, Co	0.279 ± 0.008 ^{a,A}	0.207 ± 0.004 ^{aB}	0.223 ± 0.006 ^{aB}	0.376 ± 0.012 ^{bA}	0.355 ± 0.011 ^{bA}	0.206 ± 0.006 ^{aB}	0.437 ± 0.053 ^{cA}	0.467 ± 0.015 ^{cA}	0.320 ± 0.009 ^{b,B}	
Total aldehydes				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	
Alcohols													
2-Methyl-1-butanol	1194	1208	MS, KI, Co	1.939 ± 0.060 ^{a,A}	2.238 ± 0.069 ^{aB}	1.456 ± 0.044 ^{bB}	5.659 ± 0.172 ^{bA}	1.767 ± 0.053 ^{bB}	1.437 ± 0.044 ^{aC}	2.327 ± 0.081 ^{cA}	2.318 ± 0.070 ^{aA}	2.217 ± 0.064 ^{bA}	
1-Hexanol	1365	1360	MS, KI, Co	0.876 ± 0.027 ^{a,A}	0.752 ± 0.021 ^{aB}	0.514 ± 0.016 ^{aC}	1.300 ± 0.039 ^{bA}	1.087 ± 0.033 ^{bB}	0.542 ± 0.016 ^{aC}	0.777 ± 0.004 ^{cAB}	0.805 ± 0.024 ^{cA}	0.720 ± 0.021 ^{b,C}	
1-Pentanol	1230	1212	MS, KI, Co	6.741 ± 0.205 ^{a,A}	6.412 ± 0.198 ^{aB}	4.256 ± 0.131 ^{aC}	9.139 ± 0.279 ^{bA}	6.688 ± 0.206 ^{abB}	4.210 ± 0.127 ^{aC}	5.114 ± 0.050 ^{cA}	5.264 ± 0.165 ^{cA}	5.790 ± 0.179 ^{b,B}	
3-Octanol	1404	1394	MS, KI, Co	0.579 ± 0.018 ^{a,A}	0.410 ± 0.012 ^{aB}	0.227 ± 0.007 ^{aC}	0.619 ± 0.019 ^{bA}	0.464 ± 0.014 ^{bB}	0.209 ± 0.006 ^{aC}	0.394 ± 0.015 ^{cA}	0.400 ± 0.012 ^{aA}	0.371 ± 0.010 ^{b,B}	
1-Octen-3-ol	1460	1445	MS, KI, Co	10.146 ± 0.309 ^{a,A}	9.189 ± 0.280 ^{aB}	11.611 ± 0.354 ^{aC}	12.023 ± 0.367 ^{bA}	10.616 ± 0.326 ^{bB}	8.151 ± 0.252 ^{bC}	8.057 ± 0.409 ^{cA}	8.249 ± 0.250 ^{cA}	8.523 ± 0.262 ^{bA}	
Benzyl alcohol	1900	1880	MS, KI, Co	1.210 ± 0.037 ^{a,A}	0.903 ± 0.029 ^{aB}	0.855 ± 0.026 ^{aB}	1.238 ± 0.038 ^{aA}	1.725 ± 0.054 ^{bB}	0.518 ± 0.016 ^{bC}	0.612 ± 0.024 ^{bA}	0.621 ± 0.019 ^{cA}	0.770 ± 0.023 ^{cB}	
Phenethyl-alcohol	1935	1929	MS, KI, Co	0.268 ± 0.009 ^{a,A}	0.367 ± 0.012 ^{aB}	0.207 ± 0.007 ^{aC}	0.354 ± 0.011 ^{bA}	0.584 ± 0.015 ^{bB}	0.117 ± 0.004 ^{bC}	0.248 ± 0.008 ^{cA}	0.251 ± 0.007 ^{cA}	0.074 ± 0.002 ^{cB}	
Total alcohols				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	
Ketones													
γ-Hexalactone	1702	1724	MS, KI, Co	0.095 ± 0.003 ^{a,A}	0.138 ± 0.005 ^{aB}	0.033 ± 0.001 ^{aC}	0.138 ± 0.004 ^{bA}	0.212 ± 0.005 ^{bB}	0.020 ± 0.001 ^{bC}	0.059 ± 0.007 ^{cA}	0.063 ± 0.002 ^{cA}	NID	
Total ketones				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	NID	
Total volatiles				23.144 ± 0.707 ^{a,A}	21.483 ± 0.656 ^{aB}	19.595 ± 0.599 ^{aC}	34.087 ± 1.039 ^{bA}	24.425 ± 0.746 ^{bB}	15.733 ± 0.481 ^{bC}	21.063 ± 0.923 ^{cA}	21.675 ± 0.663 ^{aA}	19.614 ± 0.596 ^{aB}	

¹ All concentration values are presented as mean ± standard deviation. ²MS, identification of mass spectrum with reference to the NIST mass spectrum library. ³Kovats retention index on DB-WAX; KI (Ref), Retention index on DB-WAX in the NIST database. ⁴Co-injection with authentic chemicals 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

DRP–LLE included hexanal and nonanal. Hexanal is characterized by a grassy and fatty odour, and nonanal had a floral and waxy odour [22]. Compared with SDE method [21], a loss of volatiles was reduced in current DRP–LLE method. At 50 °C, the hexanal content was greater than the nonanal 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 [26]. The ketone compound was γ -hexalactone, which had a waxy and creamy odour [27]. 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. In black beans total volatiles were reversely proportional to particle size in all distillation temperature. 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 [28].

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 γ -butyrolactone [29, 30]. Octanal is characterized by a fatty odour and benzaldehyde had a bitter almond odour [24]. 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 μm size and at 70 °C for the 355–500 μm 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 μm size. The highest concentration of volatile compounds in mung bean was extracted at 60 °C with a particle size of 500–710 μm , while the lowest concentration of volatile compounds was extracted at 70 °C with a particle size of 355–500 μm . This shows the same tendency as the result that when fine particles (355–510 μm) 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 [31]. In addition, 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 [32].

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.

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 [23, 33]. The sums of the peak area ratios of nine samples are presented in Table 4. The lower the SPME-HS incubation temperature, the higher the concentration of volatile compounds.

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

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 ¹	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
Aldehydes													
Octanal	1282	1264	MS ² , K ¹ , Co ⁴	0.020 ± 0.001 ^{aA}	ND	0.016 ± 0.002 ^{aB}	0.006 ± 0.001 ^{bA}	0.015 ± 0.001 ^{aBC}	0.015 ± 0.001 ^{aBC}	0.015 ± 0.001 ^{aBC}	0.015 ± 0.002 ^{bA}	0.019 ± 0.002 ^{bA}	0.014 ± 0.002 ^{bB}
Nonanal	1401	1395	MS, KI, Co	0.418 ± 0.025 ^{aA}	0.123 ± 0.007 ^{aB}	0.255 ± 0.028 ^{aC}	0.361 ± 0.045 ^{abAB}	0.283 ± 0.014 ^{bC}	0.302 ± 0.032 ^{abAC}	0.302 ± 0.032 ^{abAC}	0.163 ± 0.079 ^{cA}	0.279 ± 0.023 ^{bB}	0.260 ± 0.028 ^{aB}
Benzaldehyde	1539	1527	MS, KI, Co	0.292 ± 0.018 ^{aA}	0.335 ± 0.017 ^{aA}	0.219 ± 0.024 ^{aA}	0.252 ± 0.031 ^{aA}	0.694 ± 0.035 ^{bb}	0.215 ± 0.023 ^{aA}	0.215 ± 0.023 ^{aA}	0.428 ± 0.108 ^{abAB}	0.256 ± 0.022 ^{aA}	4.666 ± 0.122 ^{bC}
Total aldehydes				0.507 ± 0.031	0.525 ± 0.026	0.324 ± 0.035	0.406 ± 0.051	0.990 ± 0.050	0.319 ± 0.034	0.319 ± 0.034	0.577 ± 0.144	0.429 ± 0.036	4.501 ± 0.484
Alcohols													
2-Methyl-1-butanol	1205	1208	MS, KI, Co	4.529 ± 0.273 ^{aA}	4.957 ± 0.253 ^{aA}	1.242 ± 0.134 ^{aB}	3.668 ± 0.453 ^{bA}	6.140 ± 0.313 ^{bb}	1.254 ± 0.135 ^{aC}	1.254 ± 0.135 ^{aC}	1.138 ± 0.065 ^{cA}	4.037 ± 0.333 ^{cA}	0.842 ± 0.090 ^{aA}
1-Pentanol	1235	1212	MS, KI, Co	0.433 ± 0.026 ^{aA}	0.447 ± 0.023 ^{aA}	0.223 ± 0.025 ^{aB}	0.322 ± 0.040 ^{bA}	0.615 ± 0.031 ^{bb}	0.247 ± 0.027 ^{aC}	0.247 ± 0.027 ^{aC}	0.217 ± 0.097 ^{cA}	0.343 ± 0.032 ^{cA}	0.232 ± 0.025 ^{aA}
1-Hexanol	1377	1360	MS, KI, Co	1.467 ± 0.088 ^{aA}	1.084 ± 0.055 ^{aB}	0.678 ± 0.073 ^{aC}	1.049 ± 0.129 ^{bA}	1.644 ± 0.084 ^{bb}	0.680 ± 0.073 ^{aC}	0.680 ± 0.073 ^{aC}	0.250 ± 0.030 ^{cA}	0.873 ± 0.073 ^{cb}	0.483 ± 0.052 ^{bC}
1-Octanol	1577	1563	MS, KI, Co	0.195 ± 0.012 ^{aA}	0.190 ± 0.009 ^{aA}	0.089 ± 0.010 ^{aB}	0.148 ± 0.019 ^{aA}	0.281 ± 0.014 ^{bb}	0.089 ± 0.010 ^{aC}	0.089 ± 0.010 ^{aC}	0.149 ± 0.037 ^{bA}	0.150 ± 0.013 ^{cA}	0.090 ± 0.010 ^{aB}
1-Nonanol	1714	1724	MS, KI, Co	0.432 ± 0.026 ^{aA}	0.384 ± 0.020 ^{aB}	0.173 ± 0.019 ^{aC}	0.224 ± 0.028 ^{bA}	0.594 ± 0.030 ^{bb}	0.172 ± 0.019 ^{aC}	0.172 ± 0.019 ^{aC}	0.224 ± 0.0450 ^{bA}	0.293 ± 0.025 ^{cb}	0.159 ± 0.017 ^{aC}
Benzyl alcohol	1900	1880	MS, KI, Co	1.120 ± 0.067 ^{aA}	2.168 ± 0.111 ^{aB}	0.966 ± 0.104 ^{abAC}	0.466 ± 0.057 ^{bA}	3.170 ± 0.161 ^{bb}	1.114 ± 0.120 ^{bC}	1.114 ± 0.120 ^{bC}	0.299 ± 0.097 ^{bA}	1.112 ± 0.093 ^{cb}	0.798 ± 0.086 ^{aC}
Phenethyl alcohol	1930	1929	MS, KI, Co	1.010 ± 0.061 ^{aA}	2.399 ± 0.122 ^{aB}	1.150 ± 0.124 ^{aA}	0.199 ± 0.025 ^{bA}	2.604 ± 0.135 ^{bb}	1.148 ± 0.124 ^{aC}	1.148 ± 0.124 ^{aC}	N.D.	0.835 ± 0.070 ^{cA}	0.438 ± 0.048 ^{bb}
Total alcohols				9.184 ± 0.553	11.628 ± 0.592	4.527 ± 0.487	6.076 ± 0.750	15.048 ± 0.768	4.705 ± 0.507	4.705 ± 0.507	2.277 ± 0.096	7.643 ± 0.638	3.041 ± 0.328
Total volatiles				9.915 ± 0.597 ^{aA}	12.093 ± 0.616 ^{ab}	5.017 ± 0.540 ^{aC}	6.695 ± 0.827 ^{bA}	16.039 ± 0.818 ^{bb}	5.237 ± 0.564 ^{aC}	5.237 ± 0.564 ^{aC}	2.869 ± 0.242 ^{cA}	8.201 ± 0.684 ^{cb}	7.712 ± 0.831 ^{bBC}

¹ All concentration values are presented as mean ± standard deviation. ²MS, identification of mass spectrum with reference to the NIST mass spectrum library. ³Kovats retention index on DB-WAX; KI (Ref), Retention index on DB-WAX in the NIST database. ⁴Co-injection with authentic chemicals 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

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

Compounds	KI	KI (Ref.)	Identification	Peak area ratio ¹	Distillation at 50 °C			Distillation at 60 °C			Distillation at 70 °C			Whole size
					355–500 µm	500–710 µm	Whole size	355–500 µm	500–710 µm	Whole size	355–500 µm	500–710 µm	Whole size	
Aldehydes														
Hexanal	1074	1084	MS ² , KI ³ , Co ⁴	2.127 ± 0.065aA	1.004 ± 0.030aB	0.289 ± 0.009aC	1.255 ± 0.037bA	1.495 ± 0.044bB	0.148 ± 0.004bC	1.147 ± 0.035cA	2.003 ± 0.062cB	0.202 ± 0.006bC		
Nonanal	1412	1395	MS, KI, Co	0.414 ± 0.012aA	0.190 ± 0.006aB	0.218 ± 0.007aC	0.214 ± 0.007bA	0.204 ± 0.006bcAB	0.233 ± 0.007bC	0.445 ± 0.013cA	0.191 ± 0.006abBC	0.188 ± 0.005cC		
Benzaldehyde	1521	1527	MS, KI, Co	0.678 ± 0.020aA	0.646 ± 0.020aA	0.201 ± 0.006aB	0.599 ± 0.019bA	1.271 ± 0.040bB	0.144 ± 0.004bC	0.606 ± 0.018bA	0.881 ± 0.026cB	N.D.		
Total aldehydes				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	0.390 ± 0.011		
Alcohols														
2-Methyl-1-butanol	1194	1208	MS, KI, Co	5.259 ± 0.161aA	1.963 ± 0.060aB	1.361 ± 0.042aC	0.982 ± 0.030bA	1.716 ± 0.053bB	0.512 ± 0.016bC	1.183 ± 0.036cA	1.020 ± 0.031cB	1.098 ± 0.034cAB		
1-Hexanol	1368	1360	MS, KI, Co	4.112 ± 0.127aA	3.000 ± 0.092aB	1.606 ± 0.049aC	3.266 ± 0.100bA	4.612 ± 0.141bB	0.982 ± 0.030bC	4.070 ± 0.125aA	3.503 ± 0.106cB	1.069 ± 0.033bC		
cis-3-Hexen-1-ol	1401	1391	MS, KI, Co	0.778 ± 0.024aA	0.251 ± 0.008aB	0.109 ± 0.003aC	0.398 ± 0.012bA	0.333 ± 0.011abAB	0.026 ± 0.001aC	0.585 ± 0.018cA	0.299 ± 0.010abB	5.772 ± 0.176bC		
3-Octanol	1417	1394	MS, KI, Co	0.718 ± 0.022aA	0.653 ± 0.020aB	0.479 ± 0.015aC	0.400 ± 0.012bA	0.845 ± 0.026bB	0.334 ± 0.010bC	0.471 ± 0.014cA	0.538 ± 0.016cB	0.319 ± 0.009bC		
1-Nonanol	1672	1666	MS, KI, Co	0.255 ± 0.008aA	N.D.	0.106 ± 0.003aC	0.138 ± 0.004bA	0.246 ± 0.007aC	0.175 ± 0.005bB	0.175 ± 0.005cA	0.174 ± 0.005bA	0.061 ± 0.002cC		
Benzyl alcohol	1891	1880	MS, KI, Co	1.529 ± 0.047aA	1.052 ± 0.033aB	0.556 ± 0.017aC	1.141 ± 0.034bA	1.508 ± 0.045bB	0.678 ± 0.021bC	1.062 ± 0.033cA	0.741 ± 0.023cB	0.493 ± 0.015cC		
Phenethyl alcohol	1930	1929	MS, KI, Co	0.239 ± 0.007aA	0.229 ± 0.007aB	0.053 ± 0.002aC	0.189 ± 0.006bA	0.288 ± 0.009bB	0.041 ± 0.001bC	0.118 ± 0.004cA	0.123 ± 0.004cA	0.043 ± 0.001bB		
Total alcohols				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	8.853 ± 0.271		
Total volatile				16.109 ± 0.492aA	8.988 ± 0.275abB	4.977 ± 0.152aC	8.581 ± 0.261bA	12.517 ± 0.381cB	3.271 ± 0.099bC	9.864 ± 0.301cA	9.472 ± 0.289bAB	9.243 ± 0.283cB		

¹ All concentration values are presented as mean ± standard deviation. ²MS, identification of mass spectrum with reference to the NIST mass spectrum library. ³Kovats retention index on DB-WAX; KI (Ref). Retention index on DB-WAX in the NIST database. ⁴Co-injection with authentic chemicals 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

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

Compounds	KI	KI (Ref.)	Identification	Peak area ratio ¹								
				70°C ²			80°C			90°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
Aldehydes												
Hexanal	1074	1084	MS ³ , KI ⁴ , Co ⁵	2.173 ± 0.003 ^{aA}	4.292 ± 0.004 ^{AB}	0.461 ± 0.003 ^{AC}	1.125 ± 0.003 ^{BA}	2.354 ± 0.001 ^{BB}	0.656 ± 0.003 ^{BC}	0.659 ± 0.002 ^{CA}	1.119 ± 0.002 ^{CB}	0.314 ± 0.001 ^{CC}
Nonanal	1412	1395	MS, KI, Co	0.223 ± 0.001 ^{aA}	0.308 ± 0.001 ^{aB}	0.035 ± 0.001 ^{aC}	0.152 ± 0.001 ^{bA}	0.198 ± 0.002 ^{bB}	0.366 ± 0.001 ^{bC}	0.102 ± 0.002 ^{cA}	0.152 ± 0.001 ^{cB}	0.078 ± 0.002 ^{cC}
Benzaldehyde	1521	1527	MS, KI, Co	1.633 ± 0.006 ^{aA}	1.937 ± 0.003 ^{aB}	0.375 ± 0.003 ^{aC}	1.291 ± 0.004 ^{bA}	1.749 ± 0.002 ^{bB}	0.494 ± 0.002 ^{bC}	1.171 ± 0.008 ^{cA}	1.271 ± 0.004 ^{cB}	0.687 ± 0.002 ^{cC}
Total aldehydes												
				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
Alcohols												
1-Hexanol	1363	1360	MS, KI, Co	1.583 ± 0.001 ^{aA}	1.784 ± 0.002 ^{aB}	0.545 ± 0.004 ^{aC}	0.459 ± 0.001 ^{bA}	0.998 ± 0.003 ^{bB}	0.416 ± 0.005 ^{bC}	0.485 ± 0.002 ^{cA}	0.458 ± 0.005 ^{cB}	0.194 ± 0.004 ^{cC}
3-Octanol	1400	1394	MS, KI, Co	0.052 ± 0.003 ^{aA}	0.111 ± 0.002 ^{aB}	0.106 ± 0.001 ^{aC}	0.041 ± 0.002 ^{bA}	0.077 ± 0.004 ^{bB}	0.076 ± 0.004 ^{bB}	0.019 ± 0.001 ^{cA}	0.041 ± 0.003 ^{cB}	0.044 ± 0.002 ^{cC}
1-Octen-3-ol	1458	1445	MS, KI, Co	7.966 ± 0.002 ^{aA}	12.145 ± 0.008 ^{aB}	7.025 ± 0.010 ^{aC}	3.781 ± 0.002 ^{bA}	7.308 ± 0.004 ^{bB}	5.664 ± 0.008 ^{bC}	2.713 ± 0.004 ^{cA}	3.573 ± 0.003 ^{cB}	3.321 ± 0.004 ^{cC}
Benzyl alcohol	1886	1880	MS, KI, Co	0.498 ± 0.004 ^{aA}	0.171 ± 0.003 ^{aB}	0.016 ± 0.001 ^{aC}	0.106 ± 0.004 ^{bA}	0.149 ± 0.001 ^{bB}	0.012 ± 0.001 ^{bC}	0.142 ± 0.003 ^{cA}	0.110 ± 0.004 ^{cB}	0.016 ± 0.002 ^{cC}
Phenethylalcohol	1912	1929	MS, KI, Co	0.014 ± 0.004 ^{aA}	0.019 ± 0.001 ^{aB}	0.120 ± 0.002 ^{aC}	0.015 ± 0.004 ^{bA}	0.021 ± 0.001 ^{bB}	0.076 ± 0.002 ^{bC}	0.010 ± 0.002 ^{cA}	0.016 ± 0.004 ^{cB}	0.067 ± 0.001 ^{cC}
Total alcohols												
				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
Ketones												
2-Heptanone	1193	1189	MS, KI, Co	0.284 ± 0.001 ^{aA}	0.474 ± 0.001 ^{aB}	0.024 ± 0.001 ^{aC}	0.228 ± 0.002 ^{bA}	0.363 ± 0.006 ^{bB}	0.085 ± 0.002 ^{bC}	0.130 ± 0.001 ^{cA}	0.226 ± 0.003 ^{cB}	0.044 ± 0.007 ^{cC}
3-Octanone	1266	1261	MS, KI, Co	0.207 ± 0.001 ^{aA}	0.667 ± 0.003 ^{aB}	0.087 ± 0.002 ^{aC}	0.131 ± 0.003 ^{bA}	0.327 ± 0.001 ^{bB}	0.052 ± 0.004 ^{bC}	0.070 ± 0.003 ^{cA}	0.128 ± 0.001 ^{cB}	0.024 ± 0.002 ^{cC}
Total ketones				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
Total volatiles				14.632 ± 0.004 ^{aA}	21.904 ± 0.004 ^{aB}	8.795 ± 0.001 ^{aC}	7.328 ± 0.006 ^{bA}	13.539 ± 0.004 ^{bB}	7.568 ± 0.006 ^{bC}	5.503 ± 0.004 ^{cA}	7.095 ± 0.004 ^{cB}	4.786 ± 0.004 ^{cC}

¹ All concentration values are presented as mean ± standard deviation. ²SPME-HS incubation temperature. ³MS, identification of mass spectrum with reference to the NIST mass spectrum library. ⁴Kovats retention index on DB-WAX; KI (Ref). ⁵Co-injection with authentic chemicals. Kovats 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

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

Compounds	KI	KI (Ref.)	Identification	70 °C ²			80 °C			90 °C					
				Peak area ratio ¹			Whole size			Whole size			Whole size		
				355–500 μm	500–710 μm	500–710 μm	355–500 μm	500–710 μm	500–710 μm	355–500 μm	500–710 μm	500–710 μm	355–500 μm	500–710 μm	500–710 μm
Aldehydes															
Hexanal	1092	1084	MS ³ , KI ⁴ , Co ⁵	1.876 ± 0.008 ^{aA}	4.746 ± 0.001 ^{aB}	0.597 ± 0.001 ^{aC}	0.472 ± 0.001 ^{bA}	1.918 ± 0.009 ^{bB}	0.406 ± 0.001 ^{bA}	0.437 ± 0.001 ^{cA}	1.091 ± 0.020 ^{cB}	0.383 ± 0.002 ^{cA}			
Nonanal	1404	1395	MS, KI, Co	0.712 ± 0.001 ^{aA}	2.189 ± 0.003 ^{aB}	0.471 ± 0.002 ^{aC}	0.222 ± 0.001 ^{bA}	1.671 ± 0.065 ^{bB}	0.491 ± 0.002 ^{bC}	0.153 ± 0.002 ^{cA}	1.822 ± 0.080 ^{cB}	0.245 ± 0.001 ^{bC}			
Benzaldehyde	1540	1527	MS, KI, Co	3.416 ± 0.004 ^{aA}	4.548 ± 0.003 ^{aB}	1.706 ± 0.002 ^{aC}	1.196 ± 0.003 ^{bA}	3.159 ± 0.055 ^{bB}	2.155 ± 0.001 ^{bC}	1.062 ± 0.002 ^{cA}	3.017 ± 0.094 ^{cB}	4.834 ± 0.001 ^{cC}			
Total aldehydes															
				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			
Alcohols															
1-Hexanol	1363	1360	MS, KI, Co	3.723 ± 0.005 ^{aA}	2.383 ± 0.001 ^{aB}	3.298 ± 0.001 ^{aC}	0.817 ± 0.003 ^{bA}	0.976 ± 0.034 ^{bB}	2.034 ± 0.005 ^{bC}	0.554 ± 0.005 ^{cA}	0.547 ± 0.003 ^{cA}	0.913 ± 0.003 ^{cB}			
cis-3-Hexen-1-ol	1393	1391	MS, KI, Co	0.932 ± 0.001 ^{aA}	0.435 ± 0.001 ^{aB}	0.346 ± 0.001 ^{aC}	0.182 ± 0.002 ^{bA}	0.186 ± 0.003 ^{bB}	0.246 ± 0.001 ^{bC}	0.145 ± 0.002 ^{cA}	0.102 ± 0.004 ^{cB}	0.081 ± 0.001 ^{cC}			
1-Octen-3-ol	1459	1445	MS, KI, Co	0.910 ± 0.002 ^{aA}	2.234 ± 0.002 ^{aB}	2.092 ± 0.006 ^{aC}	0.284 ± 0.004 ^{bA}	1.095 ± 0.010 ^{bB}	1.703 ± 0.002 ^{bC}	0.227 ± 0.002 ^{cA}	0.661 ± 0.033 ^{cB}	0.984 ± 0.001 ^{cC}			
1-Octanol	1569	1563	MS, KI, Co	0.747 ± 0.001 ^{aA}	1.507 ± 0.001 ^{aB}	0.522 ± 0.001 ^{aC}	0.214 ± 0.002 ^{bA}	1.107 ± 0.045 ^{bB}	0.457 ± 0.002 ^{bC}	0.186 ± 0.003 ^{bA}	0.788 ± 0.020 ^{cB}	0.635 ± 0.002 ^{cC}			
Benzyl alcohol	1887	1880	MS, KI, Co	1.683 ± 0.003 ^{aA}	1.224 ± 0.005 ^{aB}	1.136 ± 0.001 ^{aC}	0.503 ± 0.001 ^{bA}	0.787 ± 0.049 ^{bB}	1.245 ± 0.001 ^{bC}	0.396 ± 0.004 ^{cA}	0.619 ± 0.013 ^{cB}	1.620 ± 0.001 ^{cC}			
Phenethylalcohol	1913	1929	MS, KI, Co	0.232 ± 0.001 ^{aA}	0.162 ± 0.001 ^{aB}	2.073 ± 0.006 ^{aC}	0.084 ± 0.005 ^{bA}	0.124 ± 0.006 ^{bB}	2.132 ± 0.006 ^{bC}	0.105 ± 0.002 ^{cA}	0.122 ± 0.012 ^{cB}	0.747 ± 0.002 ^{cC}			
Total alcohols															
				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			
Ketones															
2-Hep-tanone	1193	1189	MS, KI, Co	0.323 ± 0.004 ^{aA}	2.425 ± 0.002 ^{aB}	0.987 ± 0.001 ^{aC}	0.147 ± 0.001 ^{bA}	1.134 ± 0.050 ^{bB}	0.554 ± 0.001 ^{bC}	0.137 ± 0.001 ^{bA}	0.764 ± 0.012 ^{cB}	0.192 ± 0.001 ^{cC}			
Total ketones															
				0.323 ± 0.004 ^{aA}	2.425 ± 0.002 ^{aB}	0.987 ± 0.001 ^{aC}	0.147 ± 0.001 ^{bA}	1.134 ± 0.050 ^{bB}	0.554 ± 0.001 ^{bC}	0.137 ± 0.001 ^{bA}	0.764 ± 0.012 ^{cB}	0.192 ± 0.001 ^{cC}			
Total volatiles															
				14.554 ± 0.003 ^{aA}	21.851 ± 0.009 ^{aB}	13.228 ± 0.009 ^{aC}	4.121 ± 0.006 ^{bA}	12.156 ± 0.013 ^{bB}	11.421 ± 0.010 ^{bC}	3.400 ± 0.004 ^{cA}	9.533 ± 0.009 ^{cB}	10.616 ± 0.005 ^{cC}			

¹ All concentration values are presented as mean ± standard deviation. ²SPME-HS incubation temperature, ³MS, identification of mass spectrum with reference to the NIST mass spectrum library, ⁴Kovats retention index on DB-WAX; KI (Ref.). ⁵Co-injection with authentic chemicals. Kovats 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 Table 6. Concentrations of volatile compounds identified in soybean (Glycine max L.) extracts with HWE

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

Compounds	KI	KI (Ref.)	Identification	Peak area ratio ¹								
				70 °C		80 °C		90 °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
Aldehydes												
Hexanal	1092	1084	MS ³ , KI ¹ , Co ⁵	3.583 ± 0.002 ^{aA}	2.177 ± 0.001 ^{aB}	1.267 ± 0.001 ^{aC}	1.533 ± 0.001 ^{bA}	1.366 ± 0.007 ^{bB}	0.144 ± 0.000 ^{bC}	0.845 ± 0.002 ^{cA}	0.778 ± 0.016 ^{cB}	0.133 ± 0.003 ^{cC}
Benzaldehyde	1540	1527	MS, KI, Co	1.863 ± 0.024 ^{aA}	0.847 ± 0.002 ^{aB}	0.979 ± 0.001 ^{aC}	1.550 ± 0.014 ^{bA}	0.658 ± 0.030 ^{bB}	0.340 ± 0.001 ^{bC}	1.162 ± 0.004 ^{cA}	0.393 ± 0.003 ^{cB}	0.531 ± 0.002 ^{cC}
Total aldehydes				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
Alcohols												
2-Methyl-1-butanol	1215	1208	MS, KI, Co	0.033 ± 0.001 ^{aA}	0.021 ± 0.002 ^{aB}	0.066 ± 0.001 ^{aC}	0.020 ± 0.001 ^{bA}	0.012 ± 0.000 ^{bB}	0.071 ± 0.001 ^{bC}	0.011 ± 0.004 ^{cA}	0.007 ± 0.002 ^{cB}	0.052 ± 0.002 ^{cC}
1-Hexanol	1363	1360	MS, KI, Co	1.975 ± 0.007 ^{aA}	0.233 ± 0.003 ^{aB}	1.148 ± 0.001 ^{aC}	0.989 ± 0.002 ^{bA}	0.146 ± 0.002 ^{bB}	0.926 ± 0.004 ^{bC}	0.531 ± 0.001 ^{cA}	0.110 ± 0.002 ^{cB}	0.588 ± 0.004 ^{cC}
cis-3-Hexen-1-ol	1393	1391	MS, KI, Co	0.039 ± 0.001 ^{aA}	0.011 ± 0.003 ^{aB}	0.083 ± 0.001 ^{aC}	0.019 ± 0.001 ^{bA}	0.007 ± 0.001 ^{bB}	0.070 ± 0.002 ^{bC}	0.009 ± 0.000 ^{cA}	0.003 ± 0.000 ^{cB}	0.048 ± 0.002 ^{cC}
3-Octanol	1401	1394	MS, KI, Co	0.042 ± 0.001 ^{aA}	0.022 ± 0.001 ^{aB}	0.297 ± 0.001 ^{aC}	0.045 ± 0.003 ^{bA}	0.028 ± 0.001 ^{bB}	0.249 ± 0.001 ^{bC}	0.032 ± 0.001 ^{cA}	0.016 ± 0.001 ^{cB}	0.189 ± 0.003 ^{cC}
1-Octen-3-ol	1459	1445	MS, KI, Co	4.225 ± 0.035 ^{aA}	2.241 ± 0.010 ^{aB}	8.894 ± 0.002 ^{aC}	2.062 ± 0.015 ^{bA}	1.305 ± 0.017 ^{bB}	5.835 ± 0.007 ^{bC}	1.398 ± 0.005 ^{cA}	0.767 ± 0.003 ^{cB}	4.213 ± 0.007 ^{cC}
1-Octanol	1569	1563	MS, KI, Co	0.249 ± 0.007 ^{aA}	0.274 ± 0.002 ^{aB}	0.107 ± 0.002 ^{aC}	0.158 ± 0.002 ^{bA}	0.174 ± 0.004 ^{bB}	0.060 ± 0.003 ^{bC}	0.109 ± 0.001 ^{cA}	0.093 ± 0.002 ^{cB}	0.050 ± 0.001 ^{cC}
Benzyl alcohol	1887	1880	MS, KI, Co	0.438 ± 0.003 ^{aA}	0.049 ± 0.002 ^{aB}	0.036 ± 0.001 ^{aC}	0.423 ± 0.022 ^{bA}	0.028 ± 0.001 ^{bB}	0.044 ± 0.002 ^{bC}	0.269 ± 0.002 ^{cA}	0.026 ± 0.002 ^{cB}	0.046 ± 0.001 ^{cC}
Phenethyl alcohol	1913	1895	MS, KI, Co	0.035 ± 0.002 ^{aA}	0.010 ± 0.001 ^{aB}	0.057 ± 0.001 ^{aC}	0.039 ± 0.010 ^{bA}	0.008 ± 0.003 ^{bB}	0.098 ± 0.003 ^{bC}	0.022 ± 0.002 ^{cA}	0.007 ± 0.000 ^{cB}	0.091 ± 0.004 ^{cC}
Total alcohols				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
Total volatiles				12.481 ± 0.005 ^{aA}	5.883 ± 0.007 ^{aB}	12.933 ± 0.003 ^{aC}	6.836 ± 0.007 ^{bA}	3.732 ± 0.005 ^{bB}	7.838 ± 0.006 ^{bC}	4.388 ± 0.002 ^{cA}	2.197 ± 0.011 ^{cB}	5.941 ± 0.005 ^{cC}

¹All concentration values are presented as mean ± standard deviation. ²SPME-HS incubation temperature, ³MS, identification of mass spectrum with reference to the NIST mass spectrum library, ⁴Kovats retention index on DB-WAX; KI (Ref.), ⁵Co-injection with authentic chemicals. Kovats 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

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.

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 [34].

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 [32].

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.

Acknowledgements

The authors would like to thank the support of Dongguk University.

Author contributions

EH: formal analysis, investigation, and methodology; HA: formal analysis; SL: validation and methodology; K-GL: supervision; validation; investigation; and project administration. All authors read and approved the final manuscript.

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. 118012033S8010].

Availability of data and materials

The data sets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare that they have no conflicts of interest.

Received: 21 September 2022 Accepted: 4 December 2022

Published online: 09 December 2022

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