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Author	Hojun Seo[1], Haeng Ran Kim[2], In Hee Cho[1,*]
Affiliation	1 Wonkwang University, Iksan, Republic of Korean 2 Rural Development Administration (RDA), Wanju, Republic of Korea
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ORCID (All authors must have ORCID) https://orcid.org	Hojun Seo (https://orcid.org/0000-0001-6992-6854) Haeng Ran Kim (https://orcid.org/0000-0001-8151-4350) In Hee Cho ((https://orcid.org/0000-0002-1970-5348)
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CORRESPONDING AUTHOR CONTACT INFORMATION

For the corresponding author (responsible for correspondence, proofreading, and reprints)	**Fill in information in each box below**
First name, middle initial, last name	In Hee Cho
Email address – this is where your proofs will be sent	inheecho@wku.ac.kr
Secondary Email address	2lesiles@gmail.com
Postal address	Department of Food Science and Biotechnology, Wonkwang University, Iksan, Jeonbuk 54875, Republic of Korea
Cell phone number	+82-10-5416-6472
Office phone number	+82-63-850-6680
Fax number	+82-63-850-7308

Abstract

This study compared aroma compositions and sensory aroma attributes of raw and **cooked** *Tenebrio molitor* larvae (mealworms). Main sensory **aroma attributes** of raw mealworms were strong wet-soil-like, and less-intense oily, shrimp-like and sweet-corn-like. Quantitatively, the major aroma components of raw mealworms were hydrocarbons and aldehydes. As **cooking** proceeded, sweet-corn-like, roasted, and fried-oil-like sensory attributes were increasingly perceived with steaming, roasting, and frying, respectively. Some pyrazines, pyrrolidines, and carbonyls increased or appeared in roasted and fried mealworms. Partial least squares regression also showed differences in raw and **cooked** mealworms based on aroma components and their sensory attributes. Unlike raw mealworms, steamed mealworms had a relatively strong sweet-corn-like **aroma attribute**, which was related to 2,4,6-trimethyl-heptane, 2,4-dimethyl-dodecane, and 3,5-dihydroxy-6-methyl-2,3-dihydropyran-4-one. In comparison, roasted and fried mealworms exhibited roasted, shrimp-like, and fried-oil-like **aroma attributes**, which were associated with intermediates of the Maillard reaction and lipid oxidation, such as pyrazines, alcohols, and aldehydes. **This result during thermal reactions was very similar to those of meat and/or seafood. The use of mealworms as a savory-type flavor enhancer can be expected.**

Keywords: *Tenebrio molitor* larvae (mealworms), aroma compositions, sensory **aroma attributes**, **cooking** methods, partial least squares regression

Introduction

Entomophagy, the consumption or application of insects as food by humans, is increasing due to the need for protein combined with increasing animal protein costs, food and feed insecurity, environmental issues, population growth, etc. (Looy and Dunkel, 2014). More than 1,900 species, including beetles, caterpillars (Lepidoptera), bees/wasps/ants (Hymenoptera), grasshoppers/locusts/crickets (Orthoptera), cicadas/leafhoppers/planthoppers/scale insects/true bugs (Hemiptera), termites (Isoptera), dragonflies (Odonata), and flies (Diptera), have traditionally been used as food sources in some parts of the world (van Huis et al., 2013). Insects are a valuable sustainable food with ample energy content and high levels of protein, beneficial amino acids (leucine, isoleucine, and lysine), and unsaturated fatty acids (oleic acid, linoleic acid, and palmitic acids), minerals (copper, iron, magnesium, manganese, phosphorous, selenium, and zinc), and essential vitamins (vitamins A, B complex, and C) (Bukkens, 1997; Finke, 2002; Tang et al., 2018; Murefu et al., 2019). It was recently estimated that at least 2 billion people consume insects on a regular basis (van Huis et al., 2013) because of their nutritive value and their characteristic flavor.

Tenebrio molitor, whose larvae are known as mealworms, is a species of darkling beetle popularly consumed in Asia (Pal and Roy, 2014; Zhang and Zhao, 2019). It was recently accepted as a food ingredient by the Food and Drug Administration in Korea, following promulgation of the Act on Fosterage and Support of the Insect Industry in 2010 (Yun and Hwang, 2016). Numerous studies have examined **the nutritional components of mealworms**, and their safety, functionality, and utilization (Borremans et al, 2018; Parodi et al., 2018; Baek et al., 2019; Francis et al., 2019; Murefu et al., 2019). However, the use of mealworms, like most edible insects, as a food source remains somewhat limited because many people are averse to their physical appearance. Therefore, most insects have been used in crushed or powdered forms as food additives (Seo and Cho, 2018). They can also be used as the main ingredients of seasoning or flavoring materials after suitable reaction processes, because they are rich reservoirs with suitable **aroma** characteristics (Finke, 2002; Parodi et al., 2018). Therefore, basic information on their **aroma** profiles is necessary before their application. Recently, Kröncke et al. (2019) compared the nutrient qualities and volatiles of mealworms using different drying methods. However, there is still little information on their aroma profiles either raw or after **cooking**.

Therefore, this study profiled the aroma components and sensory attributes of mealworms, comparing the aroma characteristics of raw and **cooked** samples, and examined the differences in their aroma properties according to **cooking** methods (*i.e.*, raw, steamed, roasted, and fried).

Materials and Methods

Sample preparation

Live mealworms **at the 9th larval stage (under non-fasting condition)** were purchased from a local market (MG-Natural Co., Damyang-gun, Republic of Korea), separated from wheat bran, and stored in a deep-freezer at -70℃ until use. Raw and **cooked** mealworms were prepared (*i.e.*, steamed in boiling water for 30 min, roasted in a convection oven (**EDF 213 XPT, ESCO Co., ESKİŞEHİR, Turkey**) at 180℃ for 5 min, or deep-fried (**DKR-113, Delki Co., Gyeonggi-do, Korea**) in **soybean** oil at 180℃ for 3 min) and then placed in a mortar, frozen in liquid nitrogen, and ground to powder.

Extraction of aroma components

Ground mealworms (30 g) were mixed with 0.1 mL of benzyl acetate (200 ppm in diethyl ether) as an internal standard and extracted with 150 mL of re-distilled diethyl ether at 300 rpm for 30 min. They were separated using a centrifuge (Combi-514R, Hanil Co., Republic of Korea) at 3,000 rpm and 4℃ for 10 min, and the supernatants were filtered under a vacuum. Aroma components were then separated from the non-volatiles by solvent-assisted flavor evaporation (SAFE) with an operating vacuum that was typically below 5×10^{-5} Torr. The extract was dehydrated over anhydrous sodium sulfate, evaporated on a Vigreux column in a water bath at 40±2℃, and then concentrated under a slow stream of nitrogen gas to obtain a final volume of 0.1 mL.

Gas chromatography-mass spectrometry (GC-MS)

GC-MS analysis was performed using an Agilent 7980B gas chromatography-5977B mass selective detector (Agilent Technologies, Palo Alto, CA, USA) equipped with an HP-5MS column (30 m length×0.25 mm i.d.×0.25 mm film thickness). The carrier gas was helium at a constant flow rate of 0.8 mL/min. One microliter of the extract was injected in the split ratio (10:1) mode. The oven was started at 40℃ for 5 min, increased to 200℃ at a rate of 4℃ /min, and held at 200℃ for 10 min. The injector and detector temperatures were 250 and 230℃, respectively. The mass detector was operated in electron ionization mode with an ionization energy of 70 eV and a scan range of 50 and 550 a.m.u.

Identification and quantification of aroma components

Aroma components were identified based on comparison of their mass spectra with those of the NIST 17 (ver. 2.2) and Wiley 7.0 databases or by manual interpretation. Retention index (RI) values were compared with those reported previously (Adams, 2007;

Bianchi et al., 2007; Babushok et al., 2011; Kang et al., 2016). The RIs of volatiles were calculated using *n*-alkanes (C_7-C_{22}) as external references. Semiquantitative analysis of aroma components was performed by comparing their peak areas to that of the internal standard compound (0.1 mL of 200 ppm benzyl acetate in diethyl ether, v/v) on the GC-MS total ion chromatogram.

Sensory evaluation

To evaluate sensory aroma attributes perceived in raw and **cooked** mealworms, sensory aroma profiling modified from flavor profiling (Stampanoni, 1994) was performed (**IRB No. WKIRB-201903-HR-014**). Ten subjects (female, 21-25 years of age) who previously participated in descriptive analyses in the Department of Food Science and Biotechnology at Wonkwang University were selected and trained until they could reliably discriminate among aroma attributes: shrimp-like (aroma associated with shrimp snacks), wet-soil-like (aroma associated with damp soil), oily (aroma associated with fresh olive oil), roasted (aroma associated with baked cookies), sweet-corn-like (aroma associated with sweet corn), and fried-oil-like (aroma associated with oil fried once or twice). Each subject was given 5 g of each sample in a porcelain container (8 cm i.d.×4.5 cm height). All samples were coded with random three-digit numbers. The intensities of attributes perceived in each sample were evaluated on a 9-point intensity scale, ranging from “weak” (score of 1) to “strong” (score of 9).

Statistical analysis

Analysis of variance (ANOVA) using SPSS software (ver. 24.0; IBM Corp., Armonk, NY, USA) was performed for statistical evaluation of the differences in the aroma compositions of raw mealworms and those treated with different **cooking** methods. The results of Duncan's multi-range test were evaluated at $p<0.05$ significance level. The values of aroma components are presented as the average ± standard deviation of three replicates. Partial least squares regression (PLSR) was used to determine the relationship between instrumental and sensory data sets, performed with SIMCA-P (ver. 11.0; Umetrics, Umeå, Sweden).

Results and Discussion

Aroma components of raw and cooked mealworms

The aroma components of raw and **cooked** mealworms were extracted using SAFE and then analyzed by GC-MS. Table 1 lists the aroma components identified in mealworms, their relative peak areas, and their RIs on the HP-5ms column. In total, 46 aroma components were found in the mealworms, including four pyrazines, three pyrrolidines, three aldehydes, six ketones, five alcohols, three acids and esters, and 22 hydrocarbons. The aromas of raw mealworms were primarily composed of hydrocarbons (50.11%) and aldehydes (37.14%). In particular, 4-methylbenzaldehyde (no. 10) was the most prevalent individual component in raw mealworms and increased in steamed and roasted mealworms. Pyrazines, pyrrolidines, and carbonyls were increased or newly appeared in **cooked** mealworms (especially with roasting and frying), although there were some quantitative and qualitative differences among **cooking** methods. These components can be generated through a complex series of thermal reactions, including Maillard or non-enzymatic browning reactions between reducing sugars and amino acids, thermal degradation of lipids, and decomposition of sugars, and their interactions, which then produce the characteristic flavors differentiated from those of raw mealworms (Amrani-Hemaimi et al., 1995; Schenker et al., 2002).

Sensory aroma attributes of raw and cooked mealworms

In this study, ten trained panelists described the sensory aroma attributes of raw and **cooked** mealworms. Six sensory aroma attributes were perceived in raw and **cooked** mealworms: oily, shrimp-like, wet-soil-like, roasted, sweet-corn-like, and fried-oil-like **aroma attributes**; the mean intensities of each attribute are shown in Fig. 1. The intensities of the attributes differed significantly among mealworms (**$p<0.05$**). Raw mealworms had a strong wet-soil-like odor and less-intense oily, shrimp-like, and sweet-corn-like **aroma attributes**. In comparison, steamed mealworms had a strong sweet-corn-like **aroma attribute**, and weak shrimp-like and wet-soil-like **aroma attributes**. The intensities of two sensory attributes (e.g., roasted and shrimp-like) were noticeably increased in roasted mealworms, whereas strong fried-oil-like and roasted **aroma attributes** were major in fried mealworms.

Relationship between aroma compositions and sensory aroma attributes of raw and cooked mealworms

Multivariate statistical techniques can be used to extract, organize, and visualize statistically interpretable and reliable data information. In particular, PLSR can be used to establish the relationship between two datasets by predicting one (X) from the other (Y) (Dijksterhuis, 1994). In this study, the PLSR model calculated the cross-validation based on 46 aroma components (x variable) and six sensory aroma attributes (y variable) analyzed in raw and **cooked** mealworms and then visualized the differences among raw and **cooked** mealworms (Fig. 2). It is also possible to understand which variables carry class-separating information and which variables are mainly associated with samples (Ledauphin et al., 2010; Zhang et al., 2013). Fig. 2 shows the distributions of raw and

cooked mealworms based on the first and second PLS components of the PLSR plot. Raw and steamed mealworms were located on the negative PC 1 axis, whereas roasted and fried ones were on the positive PC 1 axis. Fried *vs.* roasted and raw *vs.* steamed mealworms were further separated along the PC 2 axis. Fried and raw mealworms were located on the positive PC 2, whereas steamed and roasted ones were on the negative PC 2 axis. The first PLS component was mainly defined by the aroma descriptors and contrasted shrimp-like, fried-oil-like, roasted, and oily **aroma attributes** on the positive dimension and wet soil-like and sweet-corn-like **aroma attributes** on the negative dimension. In particular, fried and roasted mealworms were evaluated as possessing strong shrimp-like, roasted, and fried-oily like attributes. By contrast, wet soil-like and sweet-corn-like attributes were strongly correlated with raw and steamed mealworms, respectively. In addition, 2,3-dimethyl-heptane (no. 28), bis(2-methylpropyl)-hexanedioate (no. 22), 2-butoxy-ethanol (no. 18), and ethyl-benzene (no. 29), were related to wet-soil-like aroma attributes, which were close to raw mealworms. In comparison, 2,4,6-trimethyl-heptane (no. 32), 2,4-dimethyl-dodecane (no. 42), and 3,5-dihydroxy-6-methyl-2,3-dihydropyran-4-one (no. 14) were associated with sweet-corn-like odor notes. 2,6-Dimethyl-pyrazine (no. 1), heptan-2-one (no. 11), 2,3,5-trimethyl-pyrazine (no. 4), and 1-butyl-pyrrolidine (no. 5) were correlated with the sensory attribute of shrimp-like odor, whereas 1-(1-pentenyl)-pyrrolidine (no. 7), pentan-1-ol (no. 17), oct-2-ene (no. 26), 1-(2-methyl-1-butenyl)-pyrrolidine (no. 6), 2,6,11-trimethyl-dodecane (no. 45) were responsible for fried-oil-like aroma characteristics. 2,5-Dimethyl-pyrazine (no. 2), oct-1-one-3-ol (no. 18), 2-ethyl-5-metyl-pyrazine (no. 3), and benzaldehyde (no. 8) were associated with roasted sensory attributes. **In general, raw meat (or seafood) has little aroma and only blood-like (or fresh fish-like) taste. However, it has lots of nonvolatile precursors of muscle flavor including free amino acids, peptides,**

reducing sugars, vitamins, and nucleotides. The interaction of nonvolatile precursors with one another and/or their degradation products, *via* the Maillard reaction and lipid degradation during thermal processing, produces a large number of aroma components which contribute to the development of desirable aroma of cooked meat/or seafood (Shahidi, 1998). In particular, pyrazines, pyridines, thiophenes, thiazoles, thiazolines, and Strecker aldehydes which contributing to the roasted aroma characteristics derive from the Maillard reaction, whereas those responsible for species aroma characteristics are formed from lipid degradation (alcohol, aldehyde, ketones, and furans) (Amrani-Hemaimi et al., 1995; Shahidi, 1998; Schenker et al., 2002). In our study, pyrazines, pyrrolidines, and aldehydes were produced in cooked mealworms. They have been known as major savory-type (e.g., meaty, roasted, baked, popcorn-like, and so on) aroma components (Amrani-Hemaimi et al., 1995; Shahidi, 1998; Schenker et al., 2002). However, pyrazines were dominant only in roasted and fried mealworms, but not detected in boiled ones. According to previous study, the substituted pyrazines were main products from thermal reactions (especially Maillard reaction), and decreased as the water content increase (Eichner and Karel, 1972; Lu et al., 2005). In addition, Mottram (1994) reported that high temperatures for cooking lead to high formation rate of the rapid oxidation of unsaturated fatty acids. The major lipid oxidation components (e.g., pentan-1-ol and oct-1en-3-ol) in our study were also more found in roasted and deep-fried ones.

Conclusion

The sensory **aroma attributes** of raw mealworms had strong wet-soil-like notes and less intense oily, shrimp-like, and sweet-corn-like notes. Hydrocarbons with solvent-like aroma note and aldehydes described as sweet and roasted aroma characteristic were the major aroma components in raw mealworms. The cook-treatments (e.g., steaming, roasting, and frying) increased the intensities of roasted, fried-oil-like, and sweet-corn-like **aroma attributes** of mealworms. Specifically, nitrogen-containing heterocyclic components (e.g., pyrazines and pyrrolidines) and carbonyls contributed to those aroma attributes and were associated with their differences. Raw mealworms are rich in Maillard reaction precursors and their changes in the aroma compositions during thermal processing were similar to those of meat or seafood. Therefore, basic information on their aroma profiles should be valuable in the application of flavoring materials as main ingredients.

References

Adams RP. 2007. Identification of essential oil components by gas chromatography/mass spectrometry. 4th ed. Allured publishing Corporation, Carol Stream, IL, USA.

Amrani-Hemaimi M, Cerny C, Fay LB. 1995. Mechanisms of formation of alkylpyrazines in the maillard reaction. J Agric Food Chem 43:2818-2822.

Babushok VI, Linstrom PJ, Zenkevich IG. 2011. Retention indices for frequently reported compounds of plant essential oils. J Phys Chem Ref Data 40:04310101-04310147.

Baek M, Kim MA, Kwon YS, Hwang JS, Goo TW, Jun M, Yun EY. 2019. Effects of processing methods on nutritional composition and antioxidant activity of mealworm (*Tenebrio molitor*) larvae. Entomol Res. 49:285-294.

Bianchi F, Careri M, Mangia A, Musci M. 2007. Retention indices in the analysis of food aroma volatile compounds in temperature-programmed gas chromatography: Database creation and evaluated of precision and robustness. J Sep Sci 30:563-572.

Borremans A, Lenaerts S, Crauwels S, Lievens B, Campenhout LV. 2018. Marination and fermentation of yellow mealworm larvae (*Tenebrio molitor*). Food Control 92:47-52.

Bukkens SGF. 1997. The nutritional value of edible insects. Ecol Food Nutr 36:287-319.

Dijksterhuis GB. 1994. Procrusters analysis in studying sensory-instrumental relationships. Food Qual Pref 5:115-120.

Eichner K, Karel M. 1972. Influence of water content and water activity on the sugar-amino browning reaction in model systems under various conditions. J Agric Food Chem 20:218-223.

Finke MD. 2002. Complete nutrient composition of commercially raised invertebrates used as food for insectivores. Zoo Biol 21:269-285.

Francis F, Doyen V, Debaugnies F, Mazzucchelli G, Caparros R, Alabi T, Blecker C, Haubruge E, Corazza F. 2019. Limited cross reactivity among arginine kinase allergens from mealworm and cricket edible insects. Food Chem 276:714-718.

Kang HR, Hwang HJ, Lee JE, Kim HR. 2016. Quantitative analysis of volatile flavor components in Korean alcoholic beverage and Japanese sake using SPME-GC/MS. Food Sci Biotechnol 25:979-985.

Kröncke N, Grebenteuch S, Keil C, Demtröder S, Kroh L, Thünemann AF, Benning R, Haase H. 2019. Effect of different drying methods on nutrient quality of the yellow mealworm (*Tenebrio molitor* L.). Insects 10:84-96.

Ledauphin J, Le Milbeau C, Barillier D, Hennequin D. 2010. Differences in the volatile compositions of French labeled brandies (Armagnac, Calvados, Cognac, and Mirabelle) using GC-MS and PLS-DA. J Agric Food Chem 58:7782-7793.

Looy H, Dunkel FV. 2014. How then shall we eat? Insect-eating attitudes and sustainable foodways. Agric Hum Values 31:131-141.

Lu C-Y, Hao Z, Payne R, Ho C-T. 2005. Effects of water content on volatile generation and peptide degradation in the Maillard reaction of glycine, diglycine, and triglycine. J Agric Food Chem 53:6443-6447.

Mottram, D. 1994. Flavor compounds formed during Maillard reaction. In Thermal generated flavors, Maillard, microwave and extrusion processes. Parliament TH, Morello MJ, McGorrin RJ (ed). pp 105-126. American Chemical Society, Washington, DC, USA.

Murefu TR, Macheka L, Musundire R, Manditsera FA. 2019. Safety of wild harvested and reared edible insects: A review. Food Control 101:209-224.

Pal P, Roy S. 2014. Edible insects: Future of human food – A review. Int Letters Nat Sci 21:1-11.

Parodi A, Leip A, DoBoer IJM, Slegers PM, Ziegler F, Temme EHM, Herrero M, Tuomisto H, Valin H, VanMiddelaar CE, VanLoon JJA, VanZanten HHE. 2018. The potential of future foods for sustainable and healthy diets. Nat Sustain 1:782-789.

Schenker S, Heinemann C, Huber M, Pompizzi R, Perren R, Escher F. 2002. Impact of roasting conditions on the formation of aroma compounds in coffee beans. J Food Sci 67:60-66.

Seo H, Cho IH. 2018. Physico-chemical and affective properties of bagel made with mealworm (*Tenebrio molitor* L.) powder. Korean J Food Cook Sci 34:569-575.

Shahidi F. 1998. Flavour of muscle food. In Flavor of meat, meat products and Seafoods. 2nd ed. pp 1-4. Chapman & Hall, London, United Kingdom.

Stampanoni CR. 1994. The use of standardized flavor languages and quantitative flavor profiling technique for flavored dairy products. J Sens Stud 9:383-400.

Tang Y, Debnath T, Choi EJ, Kim YW, Ryu JP, Jang S, Chung SU, Choi YJ, Kim EK. 2018. Changes in the amino acid profiles and free radical scavenging activities of *Tenebrio molitor* larvae following enzymatic hydrolysis. PLoS ONE 13:e0196218-0196223.

van Huis A, Itterbeeck JV, Klunder H, Mertens E, Halloran A, Muir G, Vantomme P. 2013. Edible insects: future prospects for food and feed security. FAO Forestry paper 9-16.

Yun EY, Hwang JS. 2016. Status and prospect for development of insect foods. Food Sci Ind 49:31-39.

Zhang L, Zeng Z, Zhao C, Kong H, Lu X, Xu G. 2013. A comparative study of volatile components in green, oolong and black teas by using comprehensive two-dimensional gas chromatography–time-of-flight mass spectrometry and multivariate data analysis. J Chromatogr A 1313:245-252.

Zhang YH, Zhao YY. 2019. The complete mitochondrial genome of dark mealworm *Tenebrio obscurus* (Coleoptera: Tenebrionidae). Mitochondr DNA Part B 4:2560-

2561.

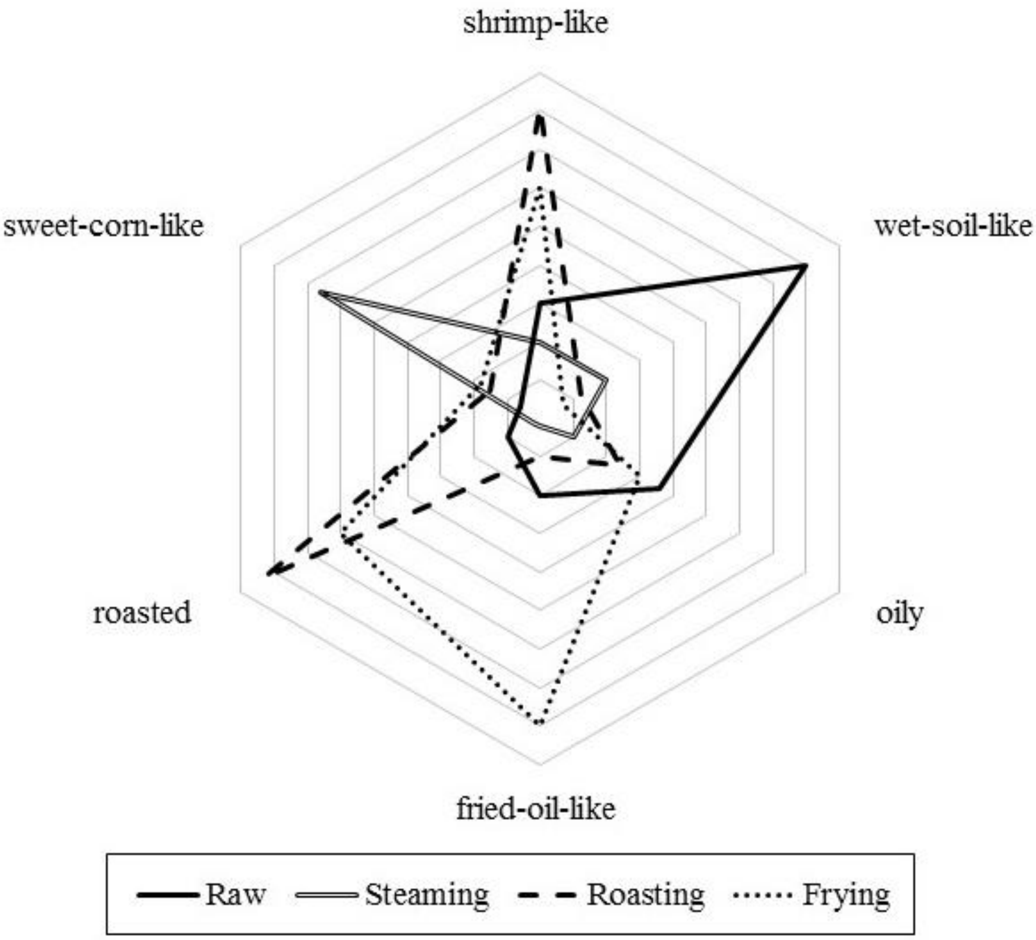

Fig. 1. Sensory aroma profiles of mealworms by different cooking methods.

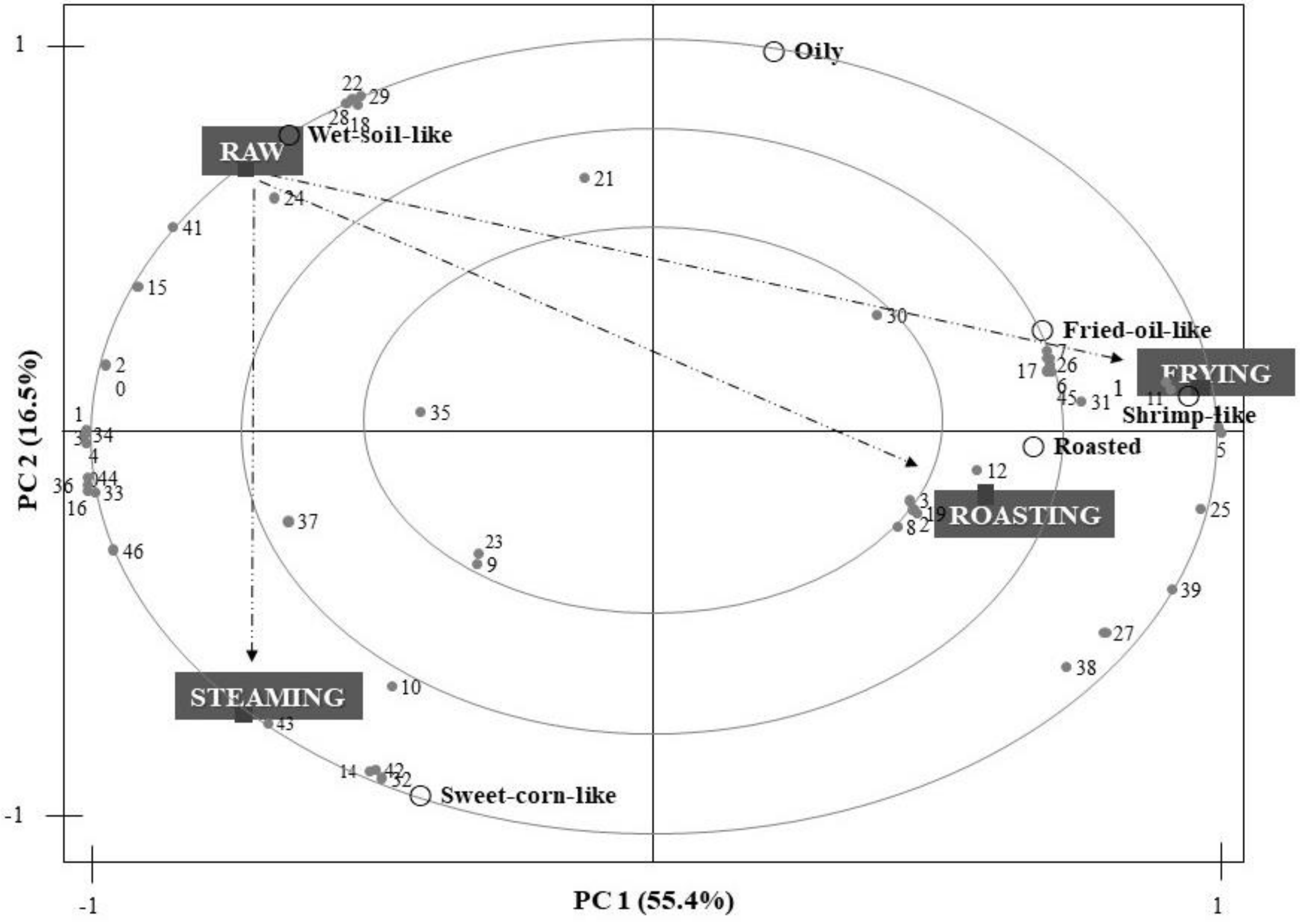

Fig. 2. The PLSR model of raw and **cooked** mealworms based on aroma components and sensory aroma attributes (■ samples, ○ sensory aroma attributes, and ● numbers correspond to aroma components in Table 1).

Table 1 Aroma components of mealworms be different cooking methods

No.	RI[1]	Aroma components	Cas #	Relative peak area (mean ± SD)[2]				ID
				Raw	Steaming	Roasting	Frying	
		PYRAZINES						
1	910	2,6-Dimethyl-pyrazine	108-50-9	ND[3]b[4]	NDb	NDb	0.283 ± 0.029a	MS/RI
2	911	2,5-Dimethyl-pyrazine	123-32-0	NDb	NDb	0.675 ± 0.027a	NDb	MS/RI
3	1001	2-Ethyl-5-methyl-pyrazine	13360-64-0	NDb	NDb	0.196 ± 0.032a	NDb	MS/RI
4	1002	2,3,5-Trimethyl-pyrazine	14667-55-1	NDc	NDc	0.157 ± 0.015b	0.238 ± 0.008a	MS/RI
		Sum of pyrazines		0 (0 %)[5]	0 (0 %)	1.028 (6.07 %)	0.521 (3.44 %)	
		PYRROLIDINES						
5	895	1-Butyl-pyrrolidine	767-10-2	NDc	NDc	0.220 ± 0.019b	0.296 ± 0.042a	MS
6	1082	1-(2-methyl-1-butenyl)-pyrrolidine	14091-87-3	NDb	NDb	NDb	1.083 ± 0.142a	MS/RI
7	1128	1-(1-pentenyl)-pyrrolidine	13937-90-1	NDb	NDb	NDb	0.071 ± 0.012a	MS
		Sum of pyrrolidines		0	0	0.220 (1.30 %)	1.450 (9.57 %)	
		ALDEHYDES						
8	957	Benzaldehyde	100-52-7	NDb	0.012 ± 0.001b	0.292 ± 0.021a	NDb	MS/RI
9	1041	2-Phenylacetaldehyde	122-78-1	0.075 ± 0.015b	0.090 ± 0.001b	0.125 ± 0.012a	NDc	MS/RI
10	1078	4-Methyl-benzaldehyde	104-87-0	5.496 ± 0.754b	6.788 ± 0.496a	6.350 ± 0.081ab	4.289 ± 0.805c	MS/RI
		Sum of aldehydes		5.571 (37.14 %)	6.890 (39.37 %)	6.767 (39.93 %)	4.289 (28.28 %)	
		KETONES						
11	892	Heptan-2-one	110-43-0	NDc	NDc	0.077 ± 0.013b	0.188 ± 0.021a	MS/RI
12	923	Methylsulfonylmethane	67-71-0	0.033 ± 0.005bc	0.012 ± 0.001c	0.255 ± 0.028a	0.051 ± 0.001b	MS/RI
13	939	4-Methylheptan-2-one	6137-06-0	0.071 ± 0.003a	0.019 ± 0.003a	NDb	NDb	MS/RI
14	1139	3,5-Dihydroxy-6-methyl-2,3-dihydropyran-4-one	28564-83-2	NDb	0.069 ± 0.010a	NDb	NDb	MS/RI

15	1431	1-(4-acetylphenyl)-Ethanone	1009-61-6	0.985 ± 0.172a	0.770 ± 0.152a	0.510 ± 0.059b	0.303 ± 0.058b	MS/RI
16	1466	2,6-Di*tert*-butylcyclohexa-2,5-diene-1,4-dione	719-22-2	0.181 ± 0.028b	0.237 ± 0.045a	NDc	NDc	MS/RI
		Sum of ketones		1.270 (8.11 %)	1.107 (6.32 %)	0.842 (4.97 %)	0.542 (3.57 %)	
		ALCOHOLS						
17	759	Pentan-1-ol	71-41-0	NDb	NDb	NDb	0.193 ± 0.019a	MS/RI
18	907	2-Butoxyethanol	111-76-2	0.010 ± 0.002a	NDb	NDb	NDb	MS/RI
19	980	Oct-1-en-3-ol	3391-86-4	NDb	NDb	0.102 ± 0.015a	NDb	MS/RI
20	1511	2,4-Bis(1,1-dimethylethyl)-phenol	96-76-4	0.580 ± 0.109a	0.487 ± 0.093a	0.153 ± 0.024b	0.100 ± 0.017b	MS/RI
21	1655	Cadin-4-en-10-ol	481-34-5	0.025 ± 0.005a	NDb	0.024 ± 0.003a	NDb	MS/RI
		Sum of alcohols		0.615 (4.10 %)	0.487 (2.78 %)	0.279 (1.65 %)	0.293 (1.93 %)	
		ESTERS & ACID						
22	1684	Bis(2-methylpropyl)-hexanedioate	141-04-8	0.027 ± 0.004a	NDb	NDb	NDb	MS/RI
23	1947	Hexadecanoic acid	57-10-3	0.013 ± 0.002b	0.017 ± 0.003b	0.023 ± 0.002a	NDc	MS/RI
24	1960	Dibutly-benzene-1,2-dicarboxylate	84-74-2	0.041 ± 0.011a	0.015 ± 0.002b	0.019 ± 0.002b	NDc	MS/RI
		Sum of acid and esters		0.081 (0.55 %)	0.032 (0.18 %)	0.042 (0.25 %)	0 (0 %)	
		HYDROCARBONS						
25	797	Octane	111-65-9	0.263 ± 0.046c	0.341 ± 0.029c	0.481 ± 0.081b	0.602 ± 0.079a	MS/RI
26	805	Oct-2-ene	111-67-1	NDb	NDb	NDb	0.063 ± 0.009a	MS/RI
27	838	2,4-Dimethyl-hept-1-ene	19549-87-2	0.948 ± 0.164b	1.073 ± 0.076ab	1.301 ± 0.223a	1.175 ± 0.200ab	MS/RI
28	852	2,3-Dimethyl-heptane	3074-71-3	0.016 ± 0.003a	NDb	NDb	NDb	MS/RI
29	857	Ethyl-benzene	100-41-4	0.015 ± 0.001a	NDb	NDb	NDb	MS/RI
30	861	4-Methyl-octane	2216-34-4	0.110 ± 0.013b	0.080 ± 0.005b	0.168 ± 0.022a	0.103 ± 0.018b	MS/RI
31	865	1,4-Xylene	106-42-3	0.034 ± 0.004b	0.034 ± 0.005b	0.037 ± 0.004b	0.062 ± 0.010a	MS/RI
32	871	2,4,6-Trimethyl-heptane	2613-61-8	NDb	0.014 ± 0.000a	NDb	NDb	MS/RI
33	899	Nonane	111-84-2	0.018 ± 0.001b	0.022 ± 0.002a	NDc	NDc	MS/RI
34	954	4-Ethyl-octane	15869-86-	0.012 ± 0.001b	0.014 ± 0.001a	NDc	NDc	MS/RI

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35	961	4-Methyl-nonane	17301-94-9	0.086 ± 0.008a	0.102 ± 0.006a	NDb	0.099 ± 0.016a	MS/RI
36	970	3-Methyl-nonane	5911-04-6	0.038 ± 0.006b	0.045 ± 0.005a	NDc	NDc	MS/RI
37	987	2,2,4,6,6-Pentamethyl-heptane	13475-82-6	0.097 ± 0.014a	0.114 ± 0.020a	0.114 ± 0.021a	NDb	MS/RI
38	999	Decane	124-18-5	2.109 ± 0.331b	2.547 ± 0.183ab	2.563 ± 0.161ab	2.839 ± 0.543a	MS/RI
39	1012	2,6-Dimethyl-nonane	17302-28-2	0.649 ± 0.084a	0.698 ± 0.050a	0.772 ± 0.059a	0.772 ± 0.098a	MS/RI
40	1023	2,2,7,7-Tetramethyl-octane	1071-31-4	0.116 ± 0.013a	0.127 ± 0.014a	NDb	NDb	MS/RI
41	1029	5-Ethyl-2,2,3-trimethyl-heptane	62199-06-8	0.147 ± 0.010a	0.070 ± 0.005b	NDc	NDc	MS
42	1168	2,4-dimethyl-dodecane	6117-99-3	NDb	0.051 ± 0.001a	NDb	NDb	MS/RI
43	1197	Dodecane	112-40-3	2.083 ± 0.333b	2.678 ± 0.152a	1.951 ± 0.032b	1.790 ± 0.112b	MS/RI
44	1212	4,8-Dimethyl-undecane	17301-33-6	0.079 ± 0.015b	0.097 ± 0.004a	NDc	NDc	MS/RI
45	1279	2,6,11-Trimethyl-dodecane	31295-56-4	NDb	NDb	NDb	0.257 ± 0.040a	MS/RI
46	1398	Tetradecane	629-59-4	0.697 ± 0.030a	0.879 ± 0.175a	0.377 ± 0.066b	0.306 ± 0.058b	MS/RI
		Sum of hydrocarbons		7.517 (50.11 %)	9.022 (51.34 %)	7.764 (45.83 %)	8.068 (53.20 %)	

[1] Retention indices were determined using *n*-paraffins C_7-C_{22} as external references

[2] Average of each peak area compared to that of the internal standard (n=3) ± standard deviation

[3] Not detected

[4] There are significant differences ($p<0.05$) among samples according to heating methods by using Duncan's multiple comparison test between the samples having different letter in low

[5] Sum of each peak area compared to that of the internal standard (n=3)

[6] Sum and their relative percentages of each peak areas according to chemical groups