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Determination of Aroma Characteristics of Commercial Garlic Powders Distributed in Korea via Instrumental and Descriptive Sensory Analyses

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ABSTRACT: Garlic (*Allium sativum*), a species in the onion Genus, plays an important role in Korean cuisine. However, because garlic is perishable, garlic powder is often used instead. Garlic powder is prepared by freeze-drying, spray-freeze drying, and/or microwave-vacuum drying. The aim of this study was to determine the aroma and sensory characteristics of commercial garlic powders using instrumental and descriptive analyses. A sensory lexicon describing 13 aroma characteristics of dried garlic powder was developed, and 35 volatile compounds were identified. This study confirmed several key compounds related to fresh-garlic aroma, including dimethyl disulfide and allyl methyl trisulfide, and identified allyl dimethyl trisulfide as a compound related to roasted-garlic aroma, with non-sulfur-containing compounds involved. The findings of this study can improve the understanding of organosulfur compounds that develop in dried garlic products during processing.

Keywords: aroma, garlic, gas chromatography-mass spectrometry, sulfides

INTRODUCTION

Garlic (*Allium sativum*) is a species in the onion Genus that shares aroma characteristics with onions, shallots, leeks, and chives. These plants all have a distinctive, pungent odor (Yang et al., 2019). Garlic plays an important role in Korean cuisine by adding versatile flavors to various recipes (Jin et al., 2001). The functional properties of garlic have previously been reported, including therapeutic effects, such as immunomodulatory, anti-cancer, hypolipidemic, antihypertensive, hepatoprotective, and antioxidant properties (Banerjee et al., 2003; Rahman, 2007; Jang et al., 2008; Kim et al., 2011; Kim et al., 2012).

Although raw garlic has many beneficial effects, it has a short shelf-life; therefore, dried garlic powder, prepared by various drying technologies, is distributed in the market instead. Hot-air drying is a typical, traditional drying method for various foods due to its convenience and low cost; however, thermal treatment confers the possibility of losing bioactive properties, including antioxidative ability (Chang and Kim, 2011). To overcome this problem, indirect or non-thermal drying methods have been developed, including freeze-drying, spray-drying, spray-freeze drying, and/or microwave-vacuum drying technologies. These processing methods differ in the presence of heat during treatment and in granulation methods (Chung and Choi, 1990; Li et al., 2007; Poojitha et al., 2020). Poojitha et al. (2020) studied spray-dried and freeze-dried garlic powder with ultrasound-assisted extraction and reported that spray-drying with maltodextrin as a carrier material could result in spherical-shaped powder formation with increased shelf-life, including physicochemical properties. Chung and Choi (1990) compared the flavor differences between hot-air-dried and freeze-dried garlic powder by comparing volatile compounds. Their study revealed that diallyl disulfide was a key compound of garlic, and that garlic powder freeze-dried at −30°C had the highest content of this volatile aromatic compound. However, all the dried garlic powder samples in this study had lower diallyl disulfide contents compared to raw garlic.

Research into flavor has been studied in various forms of garlic, including fresh garlic, black garlic, garlic extract, and garlic powder. In a study investigating the volatile compounds of fresh garlic and black garlic, fresh garlic had more diverse sulfur-containing compounds, including

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allyl methyl trisulfide, and black garlic had more non-sulfur-containing volatile compounds (Molina-Calle et al., 2017). Brodnitz et al. (1971) studied the volatile compounds in garlic extract and revealed that six sulfur-containing compounds (namely, allyl alcohol, methyl allyl disulfide, diallyl disulfide, dimethyl trisulfide, methyl allyl trisulfide, and diallyl trisulfide) were present in the garlic extracts. Li et al. (2021) reported that 49 volatile compounds (mainly heterocyclic, sulfur-containing compounds) were identified in five different garlic seasoning powders. The same study applied quantitative descriptive analysis and identified toasted, pungent, cooked garlic, burnt, and herbal attributes as the main sensory attributes of garlic seasoning powders (Li et al., 2021). Although the sensory analysis results of aged garlic extract revealed higher intensities of pungent and seasoning characteristics (Abe et al., 2020), most garlic seasonings are distributed in powder form due to its convenience of storage compared to the extract form.

The manufacturing process for garlic powder differs from product to product. However, due to the high contents of nitrogen-free solids in garlic, a slicing procedure is generally used for the removal of water from garlic in normal methods of drying (Chang and Kim, 2011). Although heat treatment during the food manufacturing process can change the aroma characteristics of garlic, research into the aroma characteristics of commonly consumed dried garlic powder products in Korea remains limited.

In this study, eight dried garlic powders that are distributed in the Korean market were analyzed using gas chromatography-mass spectrometry (GC-MS) as well as descriptive sensory analysis using a highly trained descriptive sensory panel to characterize the flavor characteristics.

MATERIALS AND METHODS

Samples included in this study

The samples included in this study were selected based on market share and the availability of garlic powder samples (GPs) in the market. All samples were purchased through the online market, except for GP 1, which was purchased through direct contact with the supplier. The physiochemical characteristics, including pH, moisture content, and color, were analyzed using the standard methods of analysis described in the Korean Food Standard Codex (Ministry of Food and Drug Safety, 2012). In brief, pH was analyzed using 10% (w/v) garlic powder diluted with distilled water using a pH meter (Lab 850, SCHOTT Instruments, Deutschland, Germany). Moisture content was measured using a moisture analyzer (WBA-110M, Daihan Scientific Co., Wonju, Korea), and color was measured using a color analyzer (CR-10 Plus, Konica Minolta, Tokyo, Japan). Values for lightness (L^*), redness (a^*), and yellowness (b^*) were recorded.

Descriptive analysis

Descriptive sensory analysis was conducted for the garlic powder samples using a highly trained sensory panel that consisted of six panelists, aged 22 to 39 years. Each panelist had more than 400 h of prior experience in the evaluation of various food products using the Spectrum™ method (Murray et al., 2001). Each sample was diluted to 10% (w/v) with deodorized water, and 20 mL of each sample was served in 56 mL plastic cups with plastic lids (Yeongdong Packaging, Seoul, Korea) that were labeled with random three-digit numbers. All samples were prepared 1 h before evaluation and maintained at room temperature.

Before evaluation, a 40-h calibration and training session was conducted to calibrate the use of the 15-point universal scale in the Spectrum™ method using a basic taste solution, as described by Kim et al. (2016). Only orthonasal sensory attributes were evaluated. Samples were served in a randomized order (Latin square design), and panelists rested for at least 5 min between samples to obviate fatigue and minimize carry-over effects during evaluation. Sensory lexicons were developed during the training session, and sensory references were selected for each sensory attribute. The 15-point universal scale was used to measure the aroma intensity of the lexicons. Paper ballots were used for data collection, and all intensity evaluations were conducted in triplicate.

Ethics statement

This study protocol was reviewed and approved by the Institutional Review Board of Jeonbuk National University (IRB no. JBNU-2022-03-019-001).

Instrumental aroma analysis

Volatile aroma analysis of garlic powder samples was conducted using an automated purge and trap (P&T) sampler (JTD-505III, Japan Analytical Industry, Tokyo, Japan), followed by GC-MS (QP2010 Plus, Shimadzu, Kyoto, Japan). Two grams of garlic powder was prepared without dilution, and 500 mg/kg of benzyl alcohol was used as an internal standard. The AQ-200 liquid sampler (Japan Analytical Industry) was applied before the P&T sampling to capture the volatile aromatic compounds. The garlic powder samples underwent a bubbling process in the liquid sampler at a rate of 50 mL/min for 30 min at a temperature of 60°C. The volatilized compounds were absorbed in a Tenax GR system (Japan Analytical Industry), and the Tenax GR was transferred to the P&T sampler. The desorption temperature in the P&T sampler was set at 280°C for 30 min at a rate of 50 mL/min;

then the cold-trap process was conducted at a temperature of −40°C. The cold-trap process was followed by pyrolysis at 280°C. The temperatures of the transfer line and needle heater were also set at 280°C. The pressure of the head press for the P&T sampler was set at 86 MPa, and the column flow rate was 1.0 mL/min with a 1/100 split ratio.

The QP2010 Plus system was used for GC-MS quantification and qualification of volatile flavor analysis, and a DB-624 column (30 m×0.251 mm×1.40 mm; Agilent Technologies, Wilmington, DE, USA) was used. The temperature in the GC-MS oven was programmed at 40°C for a 3 min hold, and was then increased at a rate of 10°C/min up to 260°C, and was finally held at 260°C for 5 min. The mass spectrometer was operated in positive electron impact ionization mode with 70 eV of electron energy and in scan mode with the scan range set between 45 and 500 m/z for the full scan setting. The volatile compounds were identified by their mass spectra in the Wiley mass spectral databases (Wiley and NIST 08). Volatile aroma analysis was conducted at the Center for University-Wide Research Facilities at Jeonbuk National University. Quantification of the volatile compounds was conducted using the calculation of peak area ratio of volatile compound to internal standard, as shown in the following equation:

$$\text{Concentration } (\mu g/kg) = \frac{\text{GC peak area } (\%) \times \text{Concentration of internal standard}}{\text{GC peak area } (\%) \text{ of internal standard}}$$

Statistical analysis

One-way analysis of variance was conducted with Fisher's least significant difference test to determine the significance of sample differences at a level of α=0.05. Principal component analysis (PCA) was used to identify the correlation between sensory and odor-active compounds in the instrumental flavor analysis results from eight different garlic powder samples. XLSTAT (Addinsoft, Paris, France) was used for all data analyses in this study.

RESULTS AND DISCUSSION

Physiochemical quality characteristics of garlic powders

Table 1 lists the information for eight commercially available garlic powder samples, including ingredients from the package nutrition panels. All samples, except GP 6 and GP 7, were made with 100% garlic, harvested in Korea. GP 6 contained 70% garlic from China and 30% corn flour from South America, and GP 7 had only 33.4% garlic as well as ingredients such as bread powder and corn flour. Significant differences were observed in the pH values of the garlic powder samples (P<0.05). The pH of the garlic powder products ranged from 5.86 to 6.85. GP 1 had the highest pH value of all samples, and GP 5 had the lowest pH value (P<0.05). The addition of other ingredients in the garlic powder samples did not seem to influence the pH value. The moisture content ranged from 10.33% to 13.69%, but was not significantly different between samples (P>0.05). The color measurement results revealed significant differences between samples (P<0.05): GP 6 and GP 7, which had other ingredients besides garlic, had lower a^* values, indicating less intense red colors compared to the other samples (P<0.05). GP 7 had the lowest b^* value of all samples, indicating that it had a less intense yellow color than the other samples (P<0.05). The presence of starchy materials, such as corn flour and bread powder, in GP 6 and GP 7 may have influenced their color differences. The drying methods of the garlic powder samples included in this study were unknown.

Table 1. Garlic powder samples included in this study

Sample	Ingredient listed	pH	Moisture content	Color		
				L^*	a^*	b^*
GP 1	Garlic 100% (Korea)	6.85±0.01^a	10.33±0.45^{ns}	64.37±1.68^{de}	7.13±0.74^d	24.53±1.31^a
GP 2	Garlic 100% (Korea)	5.84±0.00^d	13.07±0.16	67.17±1.65^{cd}	7.60±0.29^{cd}	21.57±0.90^b
GP 3	Garlic 100% (Korea)	5.82±0.01^c	11.80±0.35	68.8±1.28^{bc}	9.23±0.48^a	24.80±1.69^a
GP 4	Garlic 100% (Korea)	5.78±0.00^e	12.73±0.62	71.53±0.48^b	8.20±0.08^{bc}	21.4±0.14^b
GP 5	Garlic 100% (Korea)	5.86±0.00^e	12.00±0.13	61.8±1.04^e	7.53±0.12^{cd}	18.83±0.29^c
GP 6	Garlic 70% (China), corn flour 30% (South America)	6.03±0.00^c	10.80±0.05	76.13±0.46^a	4.57±0.05^e	16.93±0.17^{cd}
GP 7	Garlic 33.4% (China), bread powder 33.3% (wheat flour, yeast, glucose, vegetable oil, refined salt), corn flour 33.3% (imported)	6.31±0.01^b	13.69±2.24	67.60±0.82^c	4.10±0.08^e	12.40±0.22^e
GP 8	Garlic 100% (Korea)	6.30±0.02^b	12.42±0.49	51.53±2.70^f	9.00±0.70^{ab}	15.77±2.26^d

Values are presented as mean±SD.
Numbers in a column that do not share the same letter (a-f) represent significant differences at α=0.05.
GP, garlic powder sample; ns, not significant.

Descriptive sensory analysis

The descriptive sensory analysis results of eight commercially available garlic powder samples, including the sensory lexicon, with definitions and references for each term that describes the aroma characteristics, are listed in Table 2. The lexicon identified a total of 13 descriptors for the sensory characteristics of dried garlic powder: fresh garlic, roasted garlic, steamed garlic, menthol, sweet aromatics, green onion, lemon peel, brothy, soy sauce, pasta water, oxidized oil, beef extract powder, and plastic aromatic attributes. Several previous studies, identified from current literature reviews, have conducted aroma profile evaluations. One previous study compared the aroma-active compounds in fresh and black garlic and reported that fresh garlic had intense sulfur-like and gasoline-like aromatics, whereas black garlic was characterized as having a sulfur-like odor and soy-sauce-like, sour, roasted, and sweet aromatics (Yang et al., 2019). The previous study reported that black garlic had a milder flavor than fresh garlic because black garlic had more diverse aromatics, which reduced the pungent characteristics. Abe et al. (2020) reported the sensory differences between aged garlic extract and fresh garlic with eight sensory attributes: acid, fatty, metallic, caramel, seasoning, pungent, cooked potato, and cabbage. Fresh garlic had a distinctively high intensity of pungent attributes, while aged garlic extract had a relatively high intensity of seasoning attributes, with low pungent odor. The limitation of this previous study was the use of semi-trained panelists for the evaluation of the aroma characteristics, because the focus of the work was defining the key aroma-active compounds in black garlic and fresh garlic using instrumental flavor analysis (GC-olfactometry-MS and GC-time-of-flight MS).

Li et al. (2021) studied the differences in the sensory characteristics between garlic powders with different drying times. Five sensory lexicons were developed in this study using quantitative descriptive analysis: toasted, pungent, cooked garlic, burnt, and herbal. Similar to the current study, roasted, cooked garlic, burnt, and pungent attributes (which can be provided by fresh garlic) were used for the garlic powders. The burnt attribute was found to be significantly increased as drying time increased, while the toasted and cooked garlic attributes were high-

Table 2. Descriptive sensory analysis results of garlic powder samples

No	Attribute	Definition and references	GP 1	GP 2	GP 3	GP 4	GP 5	GP 6	GP 7	GP 8
1	Fresh garlic	Aromatics associated with freshly minced garlic (Ref: freshly minced garlic; intensity: 6.0)	1.67^{a}	0.00^{c}	0.30^{bc}	0.68^{b}	0.60^{b}	0.13^{c}	0.00^{c}	1.80^{a}
2	Roasted garlic	Aromatics associated with roasted garlic (Ref: roasting minced garlic in 30 min at 200°C using convection oven; intensity: 2.0)	0.00^{c}	2.20^{a}	1.77^{b}	1.73^{b}	1.60^{b}	0.00^{c}	0.00^{c}	0.00^{c}
3	Steamed garlic	Aromatics associated with steamed garlic (Ref: steaming whole garlic using steamer for 20 min; intensity: 1.5)	0.13^{cd}	0.40^{bc}	0.27^{bcd}	0.03^{d}	0.10^{cd}	0.80^{a}	0.48^{ab}	0.00^{d}
4	Menthol	Aromatics associated with menthol (Ref: peppermint candy; intensity: 4.0)	0.77^{a}	0.00^{b}	0.00^{b}	0.23^{b}	0.13^{b}	0.00^{b}	0.00^{b}	0.80^{a}
5	Sweet aromatics	Aromatics associated with corn syrup (Ref: oligosaccharides (CJ Cheil-jedang); intensity: 3.0)	0.40^{c}	1.57^{a}	1.35^{a}	0.90^{b}	1.27^{ab}	0.10^{c}	0.00^{c}	0.00^{c}
6	Green onion	Aromatics associated with green onion (Ref: chopped green onion root (white portion); intensity: 6.0)	0.40^{ab}	0.00^{c}	0.00^{c}	0.20^{bc}	0.23^{bc}	0.00^{c}	0.00^{c}	0.53^{a}
7	Lemon peel	Aromatics associated with fresh lemon peel (Ref: freshly peeled lemon peel; intensity: 4.0)	0.37^{bc}	0.00^{d}	0.00^{cd}	0.37^{bc}	0.43^{b}	0.00^{d}	0.00^{d}	0.93^{a}
8	Brothy	Aromatics associated with broth (Ref: buckwheat broth (Myunsarang); intensity: 3.0)	0.00^{c}	1.07^{a}	1.13^{a}	0.57^{b}	0.63^{b}	0.00^{c}	0.00^{c}	0.00^{c}
9	Soy sauce	Aromatics associated with Japanese soy sauce (Ref: Japanese soy sauce (MizKan); intensity: 4.0)	0.00^{b}	0.67^{a}	0.60^{a}	0.87^{a}	0.63^{a}	0.00^{b}	0.00^{b}	0.00^{b}
10	Pasta water	Aromatics associated with pasta water (Ref: drained water after boiling spaghetti noodles; intensity: 2.0)	0.10^{c}	0.00^{c}	0.00^{c}	0.00^{c}	0.00^{c}	1.53^{b}	1.97^{a}	0.20^{c}
11	Oxidized oil	Aromatics associated with oxidized fats and oil (Ref: oil put out in the air for 48 h in room temperature; intensity: 3.0)	0.00^{c}	0.00^{c}	0.00^{c}	0.00^{c}	0.00^{c}	1.17^{b}	2.17^{a}	0.00^{c}
12	Beef extract powder	Aromatics associated with beef-flavored flavor enhancer [Ref: Dasida (CJ Cheiljedang); intensity: 2.5]	1.08^{ab}	0.37^{cd}	0.20^{d}	0.00^{d}	0.30^{d}	1.47^{a}	0.77^{bc}	0.20^{d}
13	Plastic	Aromatics associated with plastic packaging materials (Ref: Zip-Loc sandwich bag; intensity: 2.0)	0.23^{b}	0.00^{b}	0.00^{b}	0.00^{b}	0.00^{b}	0.00^{b}	0.00^{b}	1.43^{a}

Numbers represents mean values of triplicate analyses of each sensory attribute by six highly trained panelists using the 15-pt universal scale in the Spectrum™ method.
Numbers in a row that do not share the same alphabetical letter represent significant differences at α=0.05.
All P-values <0.001.
GP, garlic powder sample.

est in the middle of the drying time, and decreased thereafter.

In the current study, the intensity of each aroma attribute was relatively low due to the decrease in aroma intensity caused by sample dilution (10% w/v with deodorized water), which was performed to realize the aroma characteristics of the samples in water-soluble form. Since the 15-point universal scale used in the Spectrum method does not increase in an exactly proportional manner (Dus et al., 2018), the intensities of the samples were diluted. In a previous study, some aroma characteristics of orange juice also showed low intensities (Kim et al., 2016). Differences in the intensities of all aroma attributes were observed among the samples ($P<0.05$). The fresh garlic attribute was present in GP 1 and GP 8, and these samples were characterized as having fresh garlic, green onion, and lemon peel aromatics. More specifically, GP 1 and GP 8 had scores of 1.67 and 1.80 for fresh-garlic aromatics, and scores of 0.40 and 0.53 for green onion aromatics; these results were significantly higher than those for the other samples ($P<0.05$). In contrast, the roasted garlic attribute was significantly higher in GPs 2～5 compared to the other samples, with intensity scores of 2.20, 1.77, 1.73, and 1.60, respectively ($P<0.05$). Similarly, GPs 2～5 had higher aroma intensities for the sweet, brothy, and soy sauce aromatics compared with other samples ($P<0.05$). Steamed garlic aromatics were present in GP 6 (score: 0.80) and GP 7 (score: 0.48), while steamed garlic aromatics were absent in the other samples. GP 6 and GP 7 also shared similar sensory attributes, including pasta water, oxidized oil, and beef extract powder, and these were present at higher intensities compared with other samples ($P<0.05$). Notably, GP 6 and GP 7 were the garlic powder samples that included corn flour and bread flour, and the presence of flour as part of the garlic powder may have influenced the aromatics of pasta water and oxidized oil in these samples. Pasta water aromatics were previously reported as aromatics associated with a lipid oxidation flavor in whey protein concentrate and whey protein isolate flavors, and other aromatics noted were cardboard, wet paper, cucumber, and doughy/fatty aromatics (Carunchia Whetstine et al., 2005).

Instrumental flavor analysis results

The instrumental flavor analysis results for the eight dried garlic powder samples are presented in Table 3 (Ruth, 1986; Cai et al., 2006; Chung et al., 2007; Niu et al., 2011; Dong et al., 2013; Moreira et al., 2013; Rong et al., 2018; Abe et al., 2020; Jing, 2020). Overall, 35 volatile compounds were identified in eight garlic powder samples, comprising five alcohols, two aldehydes, one ester, one ether, five hydrocarbons, one ketone, and 20 sulfur-containing compounds, including mono-, di-, and polysulfide compounds. Nine compounds (carbon oxide sulfide, methanethiol, ethanol, 2-propenal, carbon disulfide, thiophene, diallyl sulfide, 2-vinylthiophene, and diallyl disulfide) were detected in all samples, and were mostly sulfur-containing compounds. These compounds are found at high levels in heat-treated garlic compared to raw garlic (Tamaki et al., 2008). In addition, 2-propen-1-ol, allyl methyl disulfide, phenol, acetophenone, and 3-vinyl-1,2-dithiacyclohex-4-ene were detected in most (all except one or two) samples.

GP 4 showed the most diverse flavor profile on instrumental flavor analysis, and was shown to contain 31 compounds. Most of the compounds were identified at significantly high concentrations ($P<0.05$). The next most diverse flavor profile was that of GP 5, with 27 compounds, all of which showed relatively high concentrations. As described above, diallyl sulfide and diallyl disulfide were identified in every sample, and the concentrations of these compounds were significantly high in GP 4 and GP 5 ($P<0.05$). Methanethiol was also present at a significantly high concentration in GP 4 and GP 5 ($P<0.05$). Allyl methyl trisulfide was detected in only GPs 1, 4, 5, and 8. These sulfur-containing compounds are derived from allicin, which is present in high concentrations in allium species, and is known as the primary thiosulfinate compound that drives the olfactory sensation of fresh garlic (Cavallito and Bailey, 1944). Allicin is produced by alliin, an odorless compound derived from L-cysteine, by an interaction with allinase when fresh garlic cloves are crushed (Lanzotti, 2006). Thiosulfinates are highly unstable; consequently, they act as precursors of several sulfur-containing compounds formed by nonenzymatic degradation (Wang et al., 2008), including dimethyl disulfide, diallyl disulfide, diallyl sulfide, and allyl methyl trisulfide (Ariga and Seki, 2006; Lanzotti, 2006; Martins et al., 2016). These sulfides can also be precursors of other compounds; for example, diallyl disulfide can become allyl mercaptan by reduction (Trio et al., 2014), and the presence of methanethiol occurs via the demethiolation of methionine by methionine γ-lyase (Landaud et al., 2008).

Because raw garlic is perishable, garlic is often consumed in a processed form. Most drying methods involve heat treatment, which can easily induce many chemical interactions, including the Maillard reaction. Thiophene is produced from L-cysteine and sugar via reaction with mercaptoaldehyde (Yang et al., 2012), and thiazole is produced by the reaction between glucose and alliin (Wang et al., 2008). Fig. 1 displays the possible mechanisms of organosulfur compound formation. In addition to dried garlic powder, steamed garlic, aged garlic extract, and black garlic are other widely consumed forms of garlic. The key compounds in these products have been examined to determine their common volatile compounds. Block (1985) used the extraction method to identify dif-

Table 3. Volatile flavor analysis results of garlic powder samples

Compound name	RT (min)	Odor description	Ref[1)]	Concentration (mg/kg)							
				GP 1	GP 2	GP 3	GP 4	GP 5	GP 6	GP 7	GP 8
Alcohol											
1-Butanol	2.099	Sweet	4	16.7^{a}	22.7^{a}	23.7^{a}	10.2^{ab}	16.3^{a}		11.2^{ab}	
1,3-Difluoro-2-propanol	2.373	–			6.7^{a}	7.6^{a}			9.3^{a}	8.3^{a}	
Ethanol	3.200	Sweet, alcohol	1	30.2^{de}	52.4^{cde}	87.9^{bcd}	112.1^{bc}	17.2^{e}	136.5^{b}	6.8^{e}	253.7^{a}
2-Propen-1-ol	5.210	Pungent, mustard	4	54.7^{de}	187.8^{bcd}		487.2^{a}	280.5^{b}	503.2^{a}	132.4^{cde}	220.4^{bc}
Phenol	16.212	Medicinal, sweet	4	11.0^{a}	11.1^{a}	10.3^{a}	9.9^{a}			10.3^{a}	9.8^{a}
Aldehyde											
2-Propenal (acrolein)	3.529	Burnt, sweet	4	116.9^{cd}	202.9^{abc}	222.5^{ab}	261.7^{a}	287.4^{a}	253.9^{a}	65.1^{d}	146.4^{bcd}
Propanal	3.615	Oxidized apple, green, fruity	3		11.1^{b}	12.5^{b}	23.0^{a}	14.7^{b}			
Ester											
Ethyl 3-hydroxybutyrate	10.757	Fresh, fruity	7				32.2^{a}			9.3^{b}	
Hydrocarbon											
Chloromethane	1.999	–						16.0^{a}			
1,3-Cyclohexadiene	6.553	–					11.3^{a}				
2-Ethyl-1-hexene	9.827	–									11.1^{a}
3,3,5-Trimethylcyclohexene	11.088	–					17.8^{a}	12.8^{ab}		8.5^{b}	17.7^{a}
Butylated hydroxytoluene	23.618	–		14.8^{a}	12.5^{a}	13.0^{a}	14.2^{a}		7.9^{a}	10.0^{a}	7.9^{a}
Ketone											
Acetophenone	17.067	Sweet, almond	4	19.2^{abc}	15.4^{bcd}	8.4^{cd}	34.0^{ab}	25.6^{abc}		8.0^{cd}	35.7^{a}
Ether											
Dioctyl ether	25.060	–					192.5^{a}				
Thiol											
Methanethiol	2.422	Decay vegetable	2	133.7^{ab}	17.6^{d}	11.5^{d}	156.7^{a}	105.1^{bc}	28.9^{d}	10.9^{d}	74.5^{c}
2-Propen-1-thiol (allyl mercaptan)	5.383	Garlic-like, sulfury, burnt	4, 8				163.1^{a}	46.1^{b}			30.5^{c}
Monosulfide											
Carbon oxide sulfide (COS)	1.774	–		87.1^{ab}	38.5^{c}	19.3^{c}	55.3^{bc}	96.6^{a}	34.7^{c}	21.2^{c}	38.4^{c}
(E)-1-methylthio-1-propene	7.984	–		51.2^{ab}			39.6^{b}	65.0^{a}	9.9^{c}	8.7^{c}	18.1^{c}
Diallyl sulfide	12.015	Pungent, fresh garlic	8	77.2^{bc}	51.9^{cd}	33.4^{d}	161.1^{a}	98.1^{b}	44.0^{cd}	31.9^{d}	57.6^{cd}
Disulfide											
Sulfur dioxide	1.934	–					149.5^{a}	53.0^{b}			
Carbon disulfide	3.916	Disagreeable, sweet	4	38.8^{b}	23.8^{bcd}	11.8^{d}	82.9^{a}	91.1^{a}	20.7^{bcd}	13.1^{cd}	32.9^{bc}
Dimethyl disulfide	9.414	Fried garlic, towngas, sulfury	2, 6	45.3^{a}			9.9^{bc}	17.4^{b}			
Allyl methyl disulfide	13.512	Fresh garlic	8	76.6^{b}	18.3^{c}	10.7^{c}	86.1^{b}	137.7^{a}			29.1^{c}
Diallyl disulfide	16.869	Pungent, fresh garlic	8	133.3^{bc}	101.6^{cd}	55.8^{cd}	404.7^{a}	239.4^{b}	46.5^{cd}	11.7^{d}	227.3^{b}
(E)-1-Propenyl 2-propenyl disulfide	17.181	–					38.4^{a}	14.2^{c}			20.1^{b}
Polysulfide											
Dimethyl trisulfide	14.781	Fried garlic/onion	2	57.8^{a}			32.3^{b}	11.7^{bc}			10.0^{bc}
Allyl methyl trisulfide	18.104	Sulfury, eggy	8	36.4^{a}			39.8^{a}	10.8^{b}			18.1^{ab}

Table 3. Continued

Compound name	RT (min)	Odor description	Ref[1)]	Concentration (mg/kg)							
				GP 1	GP 2	GP 3	GP 4	GP 5	GP 6	GP 7	GP 8
Cyclic sulfide											
Thiophene	7.377	Aromatic	4	16.5^{cd}	18.7^{cd}	11.1^{d}	48.8^{a}	32.3^{b}	21.2^{c}	17.2^{cd}	19.1^{cd}
Thiazole	9.239	Foul smell	5				10.3^{a}			9.2^{a}	9.1^{a}
3-Methyl thiophene	10.184	Sulfury, skunky	6					5.1^{a}			
2-Vinylthiophene	12.905	–		41.1^{b}	28.0^{b}	13.2^{b}	123.4^{a}	107.5^{a}	31.1^{b}	37.5^{b}	23.2^{b}
Cyclobutanone ethylene dithioketal	18.683	–					13.9^{a}	8.4^{b}			
3-Vinyl-1,2-dithiacyclohex-4-ene	19.269	Spicy	9	30.0^{c}			318.5^{a}	197.9^{b}	38.6^{c}	25.7^{c}	19.8^{c}
Thieno[3,4-b]thiophene	19.597	–		38.5^{bc}	18.9^{d}		63.0^{a}	44.4^{ab}	20.0^{cd}	22.7^{cd}	16.8^{de}

1)Reference information for odor description: (1) Dong et al., 2013; (2) Chung et al., 2007; (3) Moreira et al., 2013; (4) Ruth, 1986; (5) Rong et al., 2018; (6) Cai et al., 2006; (7) Niu et al., 2011; (8) Abe et al., 2020; (9) Jing, 2020.
Numbers in a row that do not share the same letter (a-e) represent significant differences at α=0.05.
RT, retention time; GP, garlic powder sample.

ferent sulfur compounds in fresh garlic and found that the formation of diallyl disulfide occurred with steam treatment at 100°C, whereas allicin developed at 25°C with ethyl alcohol and water. Allyl methyl disulfide, diallyl disulfide, diallyl sulfide, dimethyl disulfide, allyl mercaptan, and allyl methyl trisulfide have been identified as the key aroma-active compounds of aged garlic extract (Abe et al., 2020). In a study of black garlic, allyl methyl trisulfide, diallyl sulfide, and diallyl disulfide were identified as the key aroma-active compounds (Yang et al., 2019). These three compounds are commonly present in several garlic products and could be important contributors to the garlic-specific aroma. In line with these findings, our results showed that GP 4 and GP 5 showed the highest concentrations of diallyl sulfide and diallyl disulfide, as well as the most intense garlic-specific aromas.

In contrast, GPs 2, 3, 6, and 7 had the lowest number of sulfur-containing compounds (six compounds in the volatile compounds profile). These samples had low garlic-related sensory descriptive analysis results, consistent with the absence of allyl mercaptan, thiazole, dimethyl disulfide, 3-methyl thiophene, dimethyl trisulfide, and allyl methyl trisulfide, compounds considered to be key to aroma. GP 6 and GP 7 contained other starchy ingredients, such as corn flour and/or bread powder, and these two samples had low or absent concentrations of sulfur-containing compounds, such as methanethiol and allyl methyl disulfide, compared with other samples. Reflecting the descriptive sensory analysis results, which identified stronger garlic-related attributes in GP 6 than in GP 7, the instrumental flavor analysis showed that GP 6 had higher concentrations of most sulfur-containing compounds (e.g., diallyl sulfide) compared to GP 7. Both samples, but especially GP 7, had low concentrations of odor-active compounds. GP 2 and GP 3 both contained six odor-active sulfur-containing compounds, but these samples had the roasted-garlic attribute (Table 2). These results suggest that allyl methyl disulfide could be a significant contributor to the roasted-garlic aroma.

Correlations between the instrumental flavor analysis and descriptive sensory analysis results

A PCA biplot of the instrumental flavor analysis results and the descriptive analysis results of the eight garlic powders is presented in Fig. 2. Only odor-active compounds that had odor descriptions in previous studies were included as part of the PCA biplot for a comparison of the sensory characteristics. This PCA biplot explained 58.52% of the total variability of the dataset; F1, representing the primary axis, explained 35.72%, and F2, representing the secondary axis, explained 22.80% of the total variability.

The positions of the garlic-related sensory attributes were separated: roasted garlic and steamed garlic were positioned in the positive F2 axis, while fresh garlic was

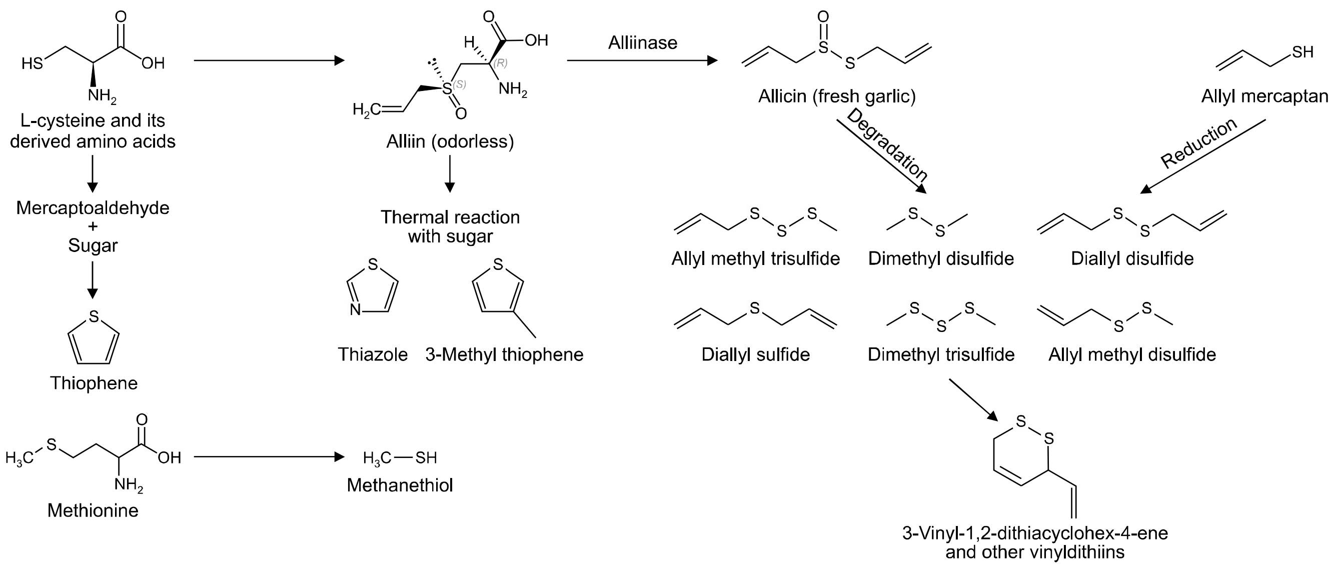

Fig. 1. Possible mechanisms of organosulfur compound formation in eight commercial dried garlic powders distributed in Korea.

positioned in the negative F2 axis. The roasted garlic attribute showed higher correlation with non-sulfur-containing compounds, such as propanal, 1-butanol, phenol, and 2-propenal, rather than with sulfur-containing compounds, and correlated with sweet aromatics, soy sauce, and brothy attributes. These results could be due to the direct heat treatment during the drying procedure; Molina-Calle et al. (2017) identified that garlic treated with a longer heating time had more roasted and sweet aromatics, with non-sulfur-containing odor-active volatile compounds. Roasted-like appearance can also be correlated with high a^* and b^* values. In the case of the steamed garlic attribute, it rarely correlated with odor-active compounds, except phenol, even when it also had heat-treated characteristics. A high correlation with moisture content could therefore affect the aroma attributes. The fresh garlic attribute was positioned in the F2 negative axis, together with most of the odor-active sulfur-containing compounds. Most of the fresh-related and strong aroma characteristics were also closely correlated with the fresh garlic attributes, such as green onion, menthol, plastic, and lemon peel. These results indicate that differences in the drying procedure, especially the presence of thermal treatment for GPs 1, 4, 5, and 8, could have triggered the

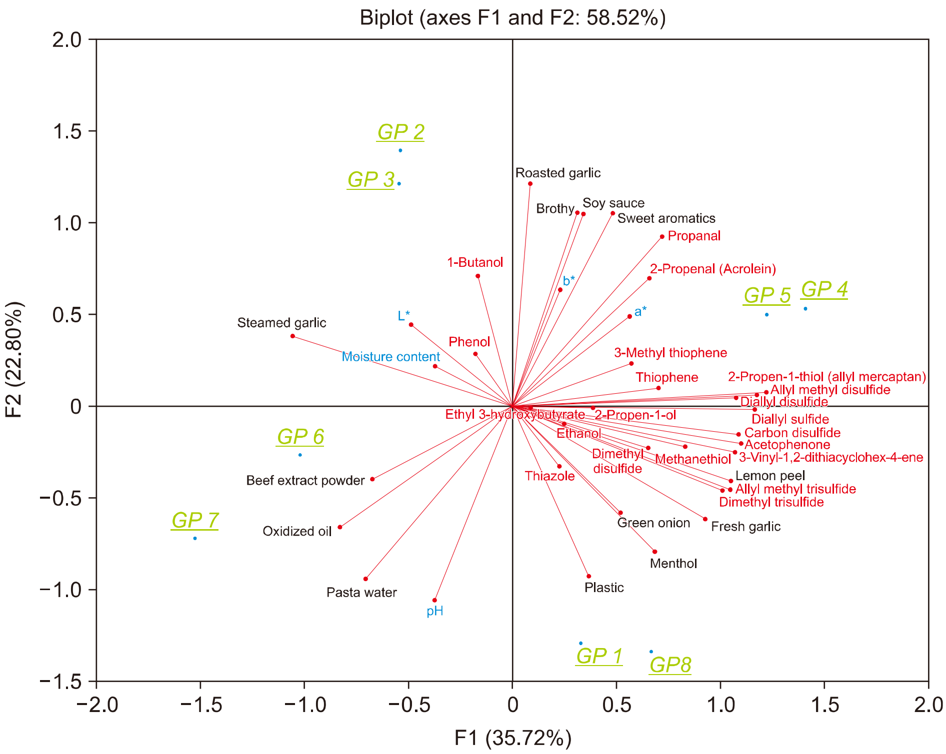

Fig. 2. Principal component analysis biplot of sensory and odor-active instrumental volatile flavor compounds analysis on eight commercial dried garlic powder products. GP, garlic powder sample.

differences between the two groups, even though these samples were all composed of 100% garlic.

In this study, instrumental flavor analysis using GC-MS and descriptive sensory analysis was conducted to identify the aroma characteristics of eight commercially available dried garlic powders. Six organosulfur compounds from dried garlic powder (allyl mercaptan, thiazole, dimethyl disulfide, 3-methyl thiophene, dimethyl trisulfide, and allyl methyl trisulfide) were considered notable contributors to the fresh-garlic aroma, while allyl methyl disulfide and non-sulfur-containing compounds, including propanal and 2-propenal, were identified as key contributors to the roasted-garlic aroma. Samples containing starchy materials showed oxidized, lipid-related aromas due to lower quantities of sulfur-containing compounds. These findings can provide a better understanding of the sensory characteristics of garlic powder by defining the formation of organosulfur compounds in dried garlic products during processing to produce consumer-attractive products.

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AUTHOR DISCLOSURE STATEMENT

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

Concept and design: MKK. Analysis and interpretation: ISH. Data collection: ISH. Writing the article: ISH. Critical revision of the article: MKK. Final approval of the article: all authors. Statistical analysis: ISH. Obtained funding: MKK. Overall responsibility: all authors.

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