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Major Sensory Attributes and Volatile Compounds of Korean Rice Liquor (*yakju*) Affecting Overall Acceptance by Young Consumers

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Abstract: The sensory characteristics and volatile compounds that affect consumers' acceptance of rice liquors were investigated. A total of 80 consumers evaluated 12 yakju samples and descriptive analysis by 11 trained panelists was conducted. Solvent-assisted flavor evaporation-gas chromatography-mass spectrometry analysis also was conducted revealing 120 volatile compounds in the *yakju* samples. Sensory attributes (n = 31) except appearance attributes were used for principal component analysis (PCA). As results, fruit odor (apple, hawthorn, omija, and pineapple odor) and flower odor (chrysanthemum, pine, and peppermint odor) were placed on the positive side of PC1 whereas persimmon vinegar odor, bitter taste, alcohol flavor, stinging and coating mouthfeel were located on the negative side of PC1. The *yakju* samples were mainly characterized by their alcohol content and supplementary ingredients. Sensory descriptors (n = 31; except appearance attributes and p > 0.05) and volatile compounds (n = 30; p > 0.5 correlation coefficient with overall acceptance) were chosen for multiple factor analysis (MFA). The MFA correlation map showed that ethyl propanoate, ethyl-2-hydroxy-2-methylbutanoate, methyl 2-furoate, γ -butyrolactone, 4-ethoxycarbonyl-γ-butyrolactone, hawthorn odor, apple flavor, grape flavor, and sweet taste were positively correlated with young consumers' overall acceptance. Additionally, negative correlation with overall acceptance was found in 1,3-butanediol, 2,3-butanediol, and 1,1-diethoxy-3-methylbutane.

Keywords: descriptive analysis; Korean rice liquor; SAFE-GC/MS; yakju

1. Introduction

Yakju is a representative traditional Korean rice wine along with *makgeolli*. *Yakju* is made with rice as a starch source, water, and *nuruk*. *Nuruk* is a starter made from grains and it plays an important role in the flavor of *yakju* during fermentation because it contains various microorganisms, including fungi and wild types of yeast [1,2]. Thus, during fermentation, sugars, various organic acids, and numerous volatile compounds are produced, affecting the flavors of the *yakju* [3]. The sensory properties of *yakju* are affected by various factors, and they are influenced not only by the yeast strain [2] but also by the fermentation process and the starch ingredients [4], and by the degree of milling of the rice used for the *nuruk* [5]. In addition, supplementary ingredients, such as mulberry [6], *Codonopsis*



lanceolate [7], *Ganoderma lucidum* [8], and buckwheat sprouts [9] are used to improve the flavor and biological activities of *yakju*.

Regarding the studies on *yakju*, Lee et al. [10] characterized five commercial rice wines containing supplementary ingredients through descriptive and physicochemical analyses. Additionally, Lee [11] reported the extrinsic factors, such as brand and familiarity, that affect the acceptance of *yakju*. However, limited information is available on the specific volatile compounds and sensory attributes associated with consumer acceptance of *yakju*.

Studies focusing on the characterization of liquors using instrumental analysis were recently conducted. For example, Kang et al. [12] discriminated traditional Korean liquor (*makgeolli* and *yakju*) and Japanese (sake samples) using solid-phase microextraction gas chromatography-mass spectrometry (SPME-GC/MS). Kang et al. [13] also discriminated Korean rice wine (*makgeolli*) samples using electronic tongues (e-tongues) and LC-MS/MS. However, the bitterness of the *makgeolli* samples caused by amino acids was not well predicted by the e-tongue in the study of Kang et al. [13]. Xiao et al. [14] reported on the aroma profiles of three types of liquor (strong/light/sauce odor type) using GC-MS. They characterized the samples based on sensory attributes and volatile compounds through partial least squares (PLS) regression. Xiao et al. [15] also conducted a sensory evaluation as well as an instrumental analysis of liquors, and they investigated the correlation between sensory attributes and volatile compounds in cherry wines. They reported that volatile compounds such as 1-propanol, 2-ethyl-1-hexanol, geraniol, ethyl hexanoate, ethyl octanoate, and butanoic acid were positively correlated with sweet aroma (>0.9) in cherry wine samples.

A limited number of studies have focused on volatile compounds, sensory attributes, and consumers' acceptance of *yakju* products containing various supplementary ingredients, which might produce complex and diverse flavors. Furthermore, Kim et al. [16] reported that young consumers in Korea tended to prefer soju (diluted liquor) and beer over the traditional liquor because of price, flavor, and hangover. To overcome the low acceptance of traditional liquor among young consumers, the key sensory attributes and volatile compounds that affect the acceptance of *yakju* containing supplementary ingredients must be identified. Therefore, this study was conducted to identify the key sensory attributes and volatile compounds that affect the acceptance of *yakju* containing supplementary ingredients by investigating the relationship of sensory attributes and volatile compounds with young consumers' acceptance of *yakju*.

2. Materials and Methods

2.1. Yakju Samples

The 12 *yakju* samples used in this study were selected based on their availability in online stores and based on the specialty of the liquor-producing regions in Korea. Information on the ingredients of the products obtained from the label of each product, is shown in Table 1. The samples were refrigerated at 4 °C until they were used.

| Sample | Ingredients | Place of Manufacture | Ethanol Content (%) |
|--------|---|-------------------------|------------------------|
| Y1 | Water, rice, starch, high-fructose corn syrup, ginseng, <i>Schizandra</i> <i>chinensis</i> fruit (omija), <i>Poria</i> cocos, <i>Lycium chinense</i> fruit, <i>Cornus</i> <i>officinalis</i> , <i>Dioscoreae Rhizoma</i> , <i>Crataegi fructus</i> , <i>Hydrangea macrophylla</i> , ginger, licorice, <i>Astragalus propinquus</i> , <i>Acanthopanax sessiliflorus</i> , yeast, wheat <i>koji</i> , citric acid | Gangwon-do, Korea | 13.0 |
| Y2 | Corn starch, rice, high-fructose corn syrup, sugar, <i>Crataegi fructus,</i> lactic acid, <i>koji, Cornus officinalis,</i> yeast, water | Gyeonggi-do, Korea | 13.0 |
| Y3 | Water, corn starch, rice, high-fructose corn syrup, sugar, <i>koji</i> , lactic acid, orange peel, yeast, dandelion | Gyeonggi-do, Korea | 13.0 |

Table 1. Information for the *yakju* samples ¹.

| Sample | Ingredients | Place of Manufacture | Ethanol Content (%) |
|--------|--|-------------------------|------------------------|
| Y4 | Water, rice, sweet pumpkin, nuruk | Gangwon-do, Korea | 17.0 |
| Y5 | Water, starch, glutinous rice, high-fructose corn syrup, wild chrysanthemum, acacia honey, <i>nuruk</i> , yeast, purified-yeast, citric acid | Gyeonggi-do, Korea | 12.5 |
| Y6 | Water, popped rice, rice, glutinous rice, glucose, isomaltooligosaccharide, <i>nuruk</i> , balloon flower (root) concentrate, stevioside | Gyeongsang-do, Korea | 13.0 |
| ¥7 | Water, rice, glutinous rice, popped rice, glucose, isomaltooligosaccharide, <i>nuruk</i> , balloon flower (root) concentrate, stevioside | Gyeongsang-do, Korea | 16.0 |
| ¥8 | Glutinous rice, rice, <i>nuruk</i> , water, wild chrysanthemum, soybean, ginger, red pepper | Chuncheong-do, Korea | 18.0 |
| Y9 | Water, glutinous/nonglutinous rice, <i>nuruk</i> , yeast, (<i>Dendropanax morbifera</i> Lev./licorice/ <i>Prunus mume</i>), <i>Lentinus edodes</i> Mycelia, mold starter, refined liquor, aspartame | Jeolla-do, Korea | 13.0 |
| Y10 | Water, rice, <i>Schizandra chinensis</i> fruit (omija), <i>Cornus officinalis, Lycium chinense</i> fruit, <i>nuruk</i> , crude amylolytic enzyme, fructose, sugar, steviol glycoside, glucose, citric acid, malic acid | Jeolla-do, Korea | 12.0 |
| Y11 | Water, rice, <i>ipguk (koji)</i> , <i>Setaria italica</i> Beauv., <i>nuruk</i> , yeast, refined yeast, licorice, <i>Sasa borealis</i> , <i>Artemisia apiacea</i> Hance, high-fructose corn syrup | Jeju, Korea | 15.0 |
| Y12 | Rice, <i>nuruk</i> , dried orange peel, yeast, crude amylolytic enzyme, purified yeast, high-fructose corn syrup, citric acid, steviol glycoside | Jeju, Korea | 11.0 |

Table 1. Cont.

¹ Information on ingredients of products was obtained from the label of each product.

2.2. Descriptive Analysis

Descriptive analysis and consumer test were approved by the institutional review board of Dankook University (DKU 2018-10-002). Eleven panelists (females, aged 37–49 years) were selected from 30 preliminary panelists based on their ability to discriminate and describe tastes and flavors in a screening test. These panelists participated in 15 training sessions (2 h per session, twice a week). During the panel training, 48 sensory descriptors (appearance = 3, odor = 17, taste/flavor = 13, mouthfeel = 6, and aftertaste/after-mouthfeel = 9), definitions, reference materials, and intensity of reference materials for the 12 yakju products were developed (Table 2). The samples were evaluated in an individual sensory booth equipped with a computerized data collection system (Korea Food Research Institute, Wanju-gun, Korea) using a 15 cm line scale (0: none to 15: very strong). The panelists evaluated six samples for each session, and they were given 15 min to test one sample. After testing three samples, they were required to rest for 15 min to prevent sensory fatigue. The yakju samples (40 g) were monadically presented in a glass cup (55 mL, diameter of top of cup = 5 cm) coded with a three-digit randomized number and were presented in a randomized order to prevent bias. Each sample was covered with a watch glass (7 cm in diameter) to minimize changes in odor during the evaluation. Filtered water and crackers were provided as palate cleansers. A spit cup was also provided for panelists to use when they did not want to swallow the samples. Evaluation sessions were conducted in three replications.

| Attributes | Definition | Reference Materials | Reference Intensity |
|---|---|--|------------------------------|
| Appearance | | | |
| Clearness | Degree of turbidity | Water | 15.0 |
| Redness | Degree of redness | Pantone color book (Pantone, NJ, USA) | 487 C = 7.5; 485 C = 15.0 |
| Yellowness | Degree of yellowness | Pantone color book (Pantone, NJ, USA) | 129 C = 7.5; 131 C = 15.0 |
| Odor | | | |
| Alcohol | Odor associated with alcohol | Soju (Chamisul Fresh, Hitejinro Co., Ltd., Seoul, Korea) | 8.44 |
| Chrysanthemum | Aroma associated with chrysanthemum | Chrysanthemum teabag (PurunSan Agricultural Co., Seoul, Korea) | 14.5 |
| Ginseng | Aroma associated with ginseng | Ginseng powder (PurunSan Agricultural Co., Seoul, Korea) | 14.5 |
| Grape | Aroma associated with grape | Grape | 14.5 |
| Hawthorn (Crataegi fructus) | Aroma associated with hawthorn fruit (Crataegi fructus) | Dried hawthorn fruit (PurunSan Agricultural Co., Seoul, Korea) | 14.5 |
| Maesil (Prunus mume fruit) | Maesil aroma | Maesil | 14.5 |
| Makgeolli | Sour and sweet aroma associated with <i>Nuruk, ipguk</i> (<i>koji</i>), and fermentation. | Rice <i>Makgeolli</i> (Kooksoondang Brewery Co., Ltd., Gangwon-do, Korea) | 10.9 |
| Omija (<i>Schizandra chinensis</i> fruit) | Aroma associated with omija | omija | 14.5 |
| Peppermint | Aroma associated with peppermint | Peppermint (Lipton peppermint herb tea, Unilever, London, England) | 14.5 |
| Persimmon vinegar | Sweet and acidic aroma associated with persimmon vinegar | Persimmon vinegar (Chungjungone Co., Seoul, Korea) | 13.3 |
| Pine | Aroma associated with pine | Pine Bud Drink (Lotte Co., Ltd., Seoul, Korea) | 13.6 |
| Roasted grain | Savory aroma associated with roasted grain | Cornsilk Tea Drink (Kwangdong Pharmaceutical Co., Ltd., Seoul, Korea | 11.7 |

Table 2. Descriptors, definitions, and reference materials of 12 *yakju* samples.

| Attributes | Definition | Reference Materials | Reference Intensity |
|----------------------------|--|---|---|
| Sour | Sour aroma associated with vinegar, fruit | Vinegar solution (Brewed vinegar, Ottogi, Gyeonggi-do, Korea) | Vinegar vs. water (1:1) = 7.60; (2:1) = 11.4 |
| Sweet | Sweet aroma associated with honey, syrup | Rice syrup (Chungjungone Co., Seoul, Korea) | 10.0 |
| Tangerine peel | Aroma associated with tangerine peel | Tangerine peel powder (PurunSan Agricultural Co., Seoul, Korea) | 14.5 |
| Yeast | Salty and moldy aroma associated with <i>meju</i> , soy sauce, and soybean paste | Yeast (Jeonwon Foods Co., Gyeonggi-do, Korea) | 12.2 |
| Taste/Flavor | | | |
| Bitterness | Fundamental taste of bitterness | 1.0% (<i>w/w</i>) guarana solution (Guarana extract powder, Cremar, Seoul, Korea) | 5.3 |
| Saltiness | Fundamental taste of saltiness | 0.1% (w/w) NaCl solution (Morton iodized salt, Morton Salt, Inc., Chicago, IL, USA) | 2.5 |
| Sourness | Fundamental taste of sourness | 0.1% (<i>w/w</i>) citric acid solution (EdentownF&B, Incheon, Korea) | 5.5 |
| Sweetness | Fundamental taste of sweetness | 1.0% (<i>w</i> / <i>w</i>) sucrose solution (CJ Cheiljedang Co., Seoul, Korea) | 2.40 |
| Umami | Fundamental taste of umami | 0.5% MSG solution (Miwon, Daesang Co., Seoul, Korea) | 13.0 |
| Alcohol | Flavor associated with alcohol | Soju (Chamisul Fresh, Hitejinro Co., Ltd., Seoul, Korea) | 7.50 |
| Apple | Flavor associated with apple | Apple | 14.5 |
| Balloon flower root | Flavor associated with balloon flower root | Balloon flower root | 14.5 |
| Grape | Flavor associated with grape | Grape | 14.5 |
| Maesil (Prunus mume fruit) | Flavor associated with maesil | Maesil | 14.5 |
| Roasted grain | Savory flavor associated with roasted grain | Cornsilk Tea Drink (Kwangdong Pharmaceutical Co., Ltd., Seoul, Korea) | 13.1 |
| Tangerine peel | Flavor associated with tangerine peel | Tangerine peel powder (PurunSan Agricultural Co., Seoul, Korea) | 14.5 |

Table 2. Cont.

| Attributes | Definition | Reference Materials | Reference Intensity |
|----------------------|---|--|---------------------|
| Mouthfeel | | | |
| Astringent | Dry and a feeling of shrank skin in the mouth | 0.3% (<i>w/w</i>) alum solution (Alum, McCormick & Co., Inc., Baltimore, MD, USA) | 2.6 |
| Body | Mouthfeel associated richness and heaviness | Soju (Chamisul Fresh, Hitejinro Co., Ltd., Seoul, Korea) | 4.5 |
| Burning | Mouthfeel associated with mouth-burning feeling caused by alcohol | Soju (Chamisul Fresh, Hitejinro Co., Ltd., Seoul, Korea) | 8.7 |
| Coating | Mouthfeel associated with wrapping the mouth with a soft and slippery feeling | Soju (Chamisul Fresh, Hitejinro Co., Ltd., Seoul, Korea) | 4.6 |
| Pungent | Mouthfeel associated with stimulation of the nasal cavity and mouth | Persimmon vinegar (Chungjungone Co., Seoul, Korea) | 14.5 |
| Stinging | Mouthfeel associated with stinging, tingling sensation | Radish sprouts | 13.5 |
| Aftertaste/mouthfeel | | | |
| Bitterness | Taste of bitterness after swallowing | 1.0% (<i>w/w</i>) Guarana solution (Guarana extract powder, Cremar, Seoul, Korea) | 5.26 |
| Sourness | Taste of sourness after swallowing | 0.1% (<i>w/w</i>) citric acid solution (EdentownF&B, Incheon, Korea) | 5.47 |
| Sweetness | Taste of sweetness after swallowing | 1.0% (<i>w/w</i>) sucrose solution (CJ Cheiljedang Co., Seoul, Korea) | 2.40 |
| Umami | Taste of umami after swallowing | 0.5% MSG solution (Miwon, Daesang Co., Seoul, Korea) | 13.0 |
| Astringent | Mouthfeel of drying, shrinking after swallowing | 0.3% (<i>w/w</i>) alum solution (Alum, McCormick & Co., Inc., Baltimore, MD, USA) | 2.6 |
| Burning | Mouthfeel of alcohol after swallowing | Soju (Chamisul Fresh, Hitejinro Co., Ltd., Seoul, Korea) | 8.7 |
| Coating | Mouthfeel of wrapping the mouth with a soft and slippery feeling after swallowing | Soju (Chamisul Fresh, Hitejinro Co., Ltd., Seoul, Korea) | 4.6 |
| Residue | Mouthfeel of residue after swallowing | Milk (SeoulMilk, Seoul, Korea) | 9.60 |
| Stinging | Mouthfeel of stinging, tingling sensation after swallowing | Soju (Chamisul Fresh, Hitejinro Co., Ltd., Seoul, Korea) | 13.5 |

2.3. Consumer Test

All of the consumers who participated in this study were users of *yakju*, and they were recruited based on them having no allergies to alcohol or *yakju* and based on their willingness to participate in the study. A total of 80 consumers (in their 20 s; male = 38, female = 42) participated in the evaluations. The samples were served following the Williams Latin Square design. The participants evaluated the overall acceptance using a 9-point hedonic scale. Six samples were provided for one evaluation session. Each sample (30 mL) was served in a paper cup (70 mL), and filtered water was provided as a palate cleanser. The consumers were asked not to consume any food for at least 1 h before the evaluation.

2.4. Identification of Volatile Compounds by GC-MS

Prior to the GC-MS analysis, the volatile compounds of the samples were extracted using the liquid-liquid continuous extraction (LLCE)/solvent-assisted flavor evaporation (SAFE) method. Each sample (1150 mL) and 3-heptanol (328 μ g; internal standard) were placed in LLCE apparatus and extracted for 8 h at room temperature using 300 mL of redistilled dichloromethane as an extraction solvent. The LLCE extracts were frozen for 12 h at -20 °C to remove water and then concentrated to 120 mL using a gentle N₂ gas stream. SAFE was applied to remove non-volatile compounds and impurities, such as pigments. The extract (120 mL) was distilled for 30 min at 40 °C under 8.0 × 10⁻³ Pa, and the SAFE extract was frozen for 12 h at -20 °C and then dried over anhydrous sodium sulfate (3 g). This extract was concentrated to 500 µL using a gentle N₂ gas stream and then analyzed by GC-MS.

Analyses were performed using an Agilent 7890B GC/Agilent 5977A mass selective detector (Agilent, Santa Clara, CA, USA) with a DB-wax column (60 m length \times 0.25 mm i.d. \times 0.25 µm film thickness; J&W Scientific, Folsom, CA, USA). Helium was used as a carrier gas with a flow rate of 1.0 mL/min. The oven temperature was initially set at 40 °C for 5 min and then increased at a rate of 5 °C/min to 200 °C, which was held for 20 min. The samples (1 µL) were injected into GC-MS apparatus at split mode (50:1). The injector and detector temperatures were 200 °C and 250 °C, respectively. The ionization voltage was 70 eV, and the mass range was 33–350 *m/z*. Analyses were conducted in triplicate.

Volatile compounds were identified based on their retention indices (RI), Wiley Registry of Mass Spectral Data (9th ed.) and by using a NIST08 database (Agilent). The concentration of the volatile compounds was semi-quantified using Formula (1), wherein the correlation coefficient of the peak area ratio and the amount ratio was assumed to be 1.

$$Concentration (ppb) = \frac{Peak area ratio \times \mu g \text{ of } 3\text{-heptanol}}{L \text{ of samples}}$$
(1)

2.5. Statistical Analysis

The descriptive analysis results and the consumers' acceptance data were analyzed by analysis of variance (ANOVA) to determine the differences among the samples. Student–Newman–Keuls (SNK) multiple comparison was used when a significant difference was found among the samples (p < 0.05). Principal component analysis (PCA) was conducted to summarize the results of the sensory characteristics of the *yakju* samples. Pearson's correlation analysis was performed to investigate the relationship between consumers' acceptance and volatile compounds. Moreover, multiple factor analysis (MFA) was performed to investigate the relationship among sensory attributes, volatile compounds, and consumers' overall acceptance of the 12 *yakju* samples. All statistical analyses were conducted using XLSTAT (Ver. 2017.1, Addinsoft, Paris, France).

3. Results and Discussion

3.1. Descriptive Analysis of Yakju Samples

The results of the ANOVA showed significant differences in 33 attributes of the 48 attributes (Table 3). Among the samples, Y10 and Y8 showed the highest scores for redness and yellowness,

respectively. The redness of Y10 might be due to the red color of the raw materials, such as the *Schizandra chinensis* fruit (omija), *Cornus officinalis*, and the *Lycium chinense* fruit. Similarly, some of the samples were characterized by attributes induced by their raw materials. For example, Y5, which contained chrysanthemum, had a strong chrysanthemum, peppermint, and pine odor. Y1 was highest in ginseng and hawthorn odor and Y7 had the highest score for bitterness and bitter aftertaste. This result might be due to saponin, which has a bitter taste [17]. Other ingredients such as fructose, also seemed to affect sweet taste in the Y2 and Y10 samples. In terms of alcohol flavor, samples with high alcohol contents such as Y7 (16.0%) and Y8 (18.0%) tended to be the highest in alcohol flavor, whereas Y10 (12.0%) and Y12 (11.0%), which contained relatively low alcohol content, were the lowest in alcohol flavor. Especially, Y8, which had the highest alcohol content, was the highest in body, coating and residue mouthfeel.

Y4 showed the highest scores for persimmon vinegar odor, sourness, stinging mouthfeel, and stinging aftertaste. This result is possibly due to the formation of acetic acid during fermentation, considering that acetic acid is associated with vinegar scent and a pungent odor [18].

The PCA result for the 31 sensory descriptive attributes of the 12 *yakju* samples is shown in Figure 1. Appearance-related descriptors (degree of clearness/redness/yellowness) were excluded to focus on the odor and flavor of the *yakju* samples, and attributes that had significant difference among the samples (n = 31) were used for the PCA. A total of 67.6% of variance could be explained by PC1 (40.4%) and PC2 (27.3%). Characteristics related to sweetness, flower, and fruit ingredients were located on the positive side of PC1, whereas bitter taste, burning mouthfeel, and alcohol odor and flavor were located on the negative side of PC1. Specifically, samples such as Y1, Y2, Y3, Y5, Y10, and Y12, which contained fruit or floral ingredients, were located on the positive side of PC1; these samples had *hawthorn* odor, sweet taste, sour taste, apple flavor, grape flavor, or tangerine peel odor/flavor.



Biplot (axes F1 and F2: 67.61 %)

Figure 1. PCA plot of the 31 sensory descriptors of the 12 *yakju* samples. O, T, F, M, AT, and AM stand for odor, taste, flavor, mouthfeel, aftertaste, and after-mouthfeel, respectively.

| - | | | | | | | | | | | | |
|---|-------------------------------|-------------------------------|-------------------------------|-------------------------------|-----------------------------|-------------------------------|------------------------------|------------------------------|---------------------------------|-------------------------------|-------------------------------|------------------------------|
| Samples | Y1 | Y2 | Y3 | Y4 | Y5 | Y6 | Y7 | Y8 | Y9 | Y10 | Y11 | Y12 |
| Appearance | - | | | | | | | | | | | |
| Clearness | 14.4 ± 0.95 | 14.4 ± 0.94 | 14.6 ± 0.57 | 14.2 ± 1.39 | 14.3 ± 2.59 | 14.2 ± 1.29 | 14.2 ± 1.37 | 13.8 ± 1.81 | 14.3 ± 2.60 | 14.0 ± 1.87 | 14.6 ± 0.64 | 14.4 ± 0.92 |
| Redness *** | 1.25 ^c ± 1.39 | $2.07^{b} \pm 2.25$ | $0.00^{\rm d} \pm 0.02$ | $0.03 \ ^{\rm d} \pm 0.08$ | $0.43^{\rm d} \pm 2.45$ | $0.04 \ ^{\rm d} \pm 0.11$ | $0.03^{\rm d} \pm 0.09$ | $0.08^{\rm d} \pm 0.22$ | $0.02^{\rm d} \pm 0.09$ | 11.5 ^a ± 2.72 | $0.01 \ ^{\rm d} \pm 0.03$ | 0.00 ^d ± 0.01 |
| Yellowness *** | $4.20^{\ c} \pm 3.50$ | $2.98 ^{\text{d}} \pm 3.41$ | $2.02 \ ^{\rm d} \pm 1.67$ | $6.95^{b} \pm 2.90$ | $1.40^{\rm ~d} \pm 0.71$ | $7.58 ^{\mathrm{b}} \pm 2.46$ | 9.18 ^a ± 2.30 | 10.1 ^a ± 1.99 | $1.37^{\rm ~d} \pm 0.88$ | $1.71 \ ^{\rm d} \pm 4.00$ | $1.98 ^{\text{d}} \pm 1.41$ | 4.67 ^c ± 2.06 |
| Odor | | | | | | | | | | | | |
| Apple ** | $0.21^{ab} \pm 0.56$ | $0.43~^{ab}\pm1.06$ | $0.46~^{\rm ab}\pm0.74$ | $0.22~^{ab}\pm0.45$ | $0.12^{\text{ b}} \pm 0.34$ | $0.12^{\text{ b}} \pm 0.38$ | $0.03 b \pm 0.09$ | $0.19^{ab} \pm 0.65$ | $0.29^{ab} \pm 0.53$ | $0.32^{ab} \pm 0.91$ | $0.71 \ ^{a} \pm 1.24$ | 0.19 ± 0.51 |
| Alcohol | 3.65 ± 2.03 | 3.53 ± 1.83 | 3.75 ± 2.06 | 3.71 ± 1.87 | 3.50 ± 1.95 | 3.83 ± 1.91 | 3.81 ± 1.67 | 3.56 ± 2.14 | 3.78 ± 1.99 | 3.48 ± 1.94 | 3.56 ± 2.19 | 3.20 ± 1.84 |
| Chrysanthemum *** | $0.09^{b} \pm 0.41$ | $0.03^{b} \pm 0.11$ | $0.02^{\text{ b}} \pm 0.09$ | $0.05^{b} \pm 0.18$ | 2.09 ^a ± 2.98 | $0.19^{b} \pm 0.46$ | $0.15^{b} \pm 0.34$ | $0.16^{b} \pm 0.51$ | $0.02^{\text{ b}} \pm 0.12$ | $0.60^{b} \pm 2.66$ | $0.26^{b} \pm 1.19$ | $0.64^{b} \pm 1.45$ |
| Ginseng *** | $1.97 \ ^{a} \pm 2.41$ | $0.06^{b} \pm 0.22$ | $0.46^{b} \pm 1.78$ | $0.21 \text{ b} \pm 0.83$ | $0.09^{b} \pm 0.37$ | $0.06^{b} \pm 0.18$ | $0.39^{\text{ b}} \pm 1.47$ | $0.53^{b} \pm 1.67$ | $0.40^{\text{ b}} \pm 1.12$ | $0.11 \text{ b} \pm 0.57$ | $0.34^{\text{ b}} \pm 1.22$ | 0.03 ^b ± 0.08 |
| Hawthorn *** | 1.38 ^a ± 2.11 | $0.80^{\text{ abc}} \pm 1.57$ | $0.29 \text{ bc} \pm 1.21$ | $0.24^{bc} \pm 0.56$ | $0.48^{bc} \pm 1.15$ | $0.22 {}^{bc} \pm 0.45$ | $0.19^{bc} \pm 0.36$ | $0.10^{\text{ bc}} \pm 0.22$ | $0.01 \ ^{\rm c} \pm 0.03$ | $0.86^{ab} \pm 0.87$ | $0.02 \ ^{\rm c} \pm 0.06$ | $0.67^{bc} \pm 1.32$ |
| Maesil (<i>Prunus mume</i> fruit) | 0.74 ± 1.27 | 1.22 ± 2.12 | 0.63 ± 0.81 | 0.82 ± 1.14 | 0.31 ± 0.55 | 0.77 ± 0.85 | 0.66 ± 1.06 | 0.88 ± 1.21 | 0.51 ± 0.88 | 0.75 ± 1.39 | 0.64 ± 0.80 | 0.59 ± 0.95 |
| Makgeolli | 2.22 ± 1.81 | 2.21 ± 1.73 | 2.70 ± 1.96 | 2.26 ± 1.41 | 1.89 ± 2.02 | 2.57 ± 1.63 | 2.45 ± 2.17 | 1.96 ± 1.33 | 2.49 ± 1.75 | 1.32 ± 1.43 | 2.69 ± 1.97 | 1.63 ± 1.35 |
| Omija (Schizandra chinensis fruit) *** | 0.98 ^b ± 1.49 | $0.50^{\text{ b}} \pm 0.87$ | $0.01 ^{b} \pm 0.02$ | $0.27 ^{\mathrm{b}} \pm 0.94$ | 0.17 ^b ± 0.38 | $0.17^{\text{ b}} \pm 0.50$ | 0.13 ^b ± 0.32 | 0.16 ^b ± 0.48 | $0.00^{b} \pm 0.02$ | 5.75 ^a ± 3.63 | $0.01 ^{\mathrm{b}} \pm 0.02$ | 0.28 ^b ± 0.64 |
| Peppermint *** | $0.13^{b} \pm 0.50$ | $0.07^{b} \pm 0.41$ | $0.05^{\rm b} \pm 0.17$ | $0.26^{b} \pm 1.04$ | $1.11 \ ^{a} \pm 1.86$ | $0.07 {}^{b} \pm 0.38$ | $0.00^{b} \pm 0.02$ | $0.07^{b} \pm 0.41$ | 0.00 ^b \pm 0.01 | $0.17 {}^{b} \pm 0.64$ | $0.00^{b} \pm 0.02$ | $0.47^{\text{ b}} \pm 1.02$ |
| Persimmon Vinegar *** | $0.34 \text{ bcd} \pm 0.81$ | $0.36 \text{ bcd} \pm 0.64$ | 0.11 ^d \pm 0.24 | $1.09^{a} \pm 1.65$ | $0.06^{\rm d} \pm 0.17$ | $0.98 \ ^{ab} \pm 1.25$ | $0.74 \ ^{abcd} \pm 1.11$ | $0.86 \ ^{abc} \pm 1.33$ | $0.24 \ ^{\rm cd} \pm 0.44$ | $0.64 \ ^{abcd} \pm 1.34$ | $0.31^{bcd} \pm 0.64$ | $0.21 \ ^{\rm cd} \pm 0.37$ |
| Pine *** | $0.10^{b} \pm 0.28$ | $0.01 \ ^{\rm b} \pm 0.02$ | $0.06^{b} \pm 0.23$ | $0.14^{\rm b} \pm 0.81$ | $2.05^{a} \pm 2.49$ | $0.00^{\text{ b}} \pm 0.01$ | 0.01 $^{\rm b} \pm 0.02$ | $0.05^{b} \pm 0.27$ | $0.00^{\text{ b}} \pm 0.02$ | $0.10^{\text{ b}} \pm 0.47$ | $0.01 \text{ b} \pm 0.02$ | $0.71^{\text{ b}} \pm 1.51$ |
| Pineapple ** | $0.02^{b} \pm 0.06$ | $0.14^{\text{ b}} \pm 0.35$ | $0.30^{b} \pm 1.31$ | $0.20^{b} \pm 0.89$ | $0.15^{b} \pm 0.50$ | $0.27 {}^{b} \pm 0.81$ | 0.01 ^b \pm 0.03 | $0.02^{b} \pm 0.09$ | $0.11 \ ^{\mathrm{b}} \pm 0.22$ | $0.14^{\text{ b}} \pm 0.58$ | $1.02 \ ^{a} \pm 2.63$ | $0.07^{b} \pm 0.34$ |
| Roasted grain *** | $0.72^{ab} \pm 1.37$ | $0.49^{b} \pm 0.82$ | $0.93^{ab} \pm 1.21$ | $1.32^{ab} \pm 2.14$ | $0.30^{b} \pm 0.59$ | 1.53 ^a ± 2.20 | $1.15^{ab} \pm 1.60$ | 1.60 ^a ± 2.01 | $0.43^{b} \pm 0.61$ | $0.78^{ab} \pm 1.15$ | $0.41^{\text{ b}} \pm 0.55$ | 0.43 ^b ± 0.58 |
| Sour | 2.94 ± 2.87 | 3.11 ± 2.76 | 2.70 ± 2.41 | 3.67 ± 3.00 | 2.69 ± 2.59 | 3.00 ± 2.70 | 2.64 ± 2.57 | 3.60 ± 3.35 | 2.36 ± 2.53 | 4.15 ± 3.09 | 3.04 ± 2.70 | 3.01 ± 3.02 |
| Sweet | 2.93 ± 1.84 | 2.65 ± 1.88 | 2.42 ± 1.47 | 2.99 ± 1.58 | 2.40 ± 1.98 | 3.05 ± 1.83 | 2.58 ± 1.68 | 3.28 ± 1.54 | 2.58 ± 1.51 | 3.68 ± 2.30 | 3.06 ± 2.03 | 2.94 ± 1.52 |
| Tangerine peel *** | $0.25^{b} \pm 0.67$ | $0.08^{b} \pm 0.25$ | $0.22^{b} \pm 0.96$ | $0.37^{b} \pm 0.82$ | $0.19^{b} \pm 0.38$ | $0.11 ^{\mathrm{b}} \pm 0.22$ | $0.40^{b} \pm 0.86$ | $0.20^{b} \pm 0.43$ | $0.07 {}^{\rm b} \pm 0.23$ | $0.33 b \pm 0.94$ | $0.09^{b} \pm 0.36$ | 1.13 ^a ± 1.32 |
| Yeast *** | $0.89^{b} \pm 1.38$ | $1.86^{b} \pm 2.10$ | $0.91 {}^{\rm b} \pm 1.00$ | 3.26 ^a ± 2.05 | $0.65^{b} \pm 0.88$ | $3.60^{a} \pm 2.70$ | 3.73 ^a ± 2.55 | 3.46 ^a ± 2.27 | $1.00^{b} \pm 1.22$ | $0.86^{b} \pm 1.25$ | $1.68^{b} \pm 1.98$ | $1.25^{\text{ b}} \pm 1.18$ |
| Taste/Flavor | | | | | | | | | | | | |
| Bitterness ** | $2.59^{abc} \pm 1.96$ | $1.77 \ ^{\rm c} \pm 1.54$ | $2.19^{\text{ abc}} \pm 1.57$ | $2.96 \ ^{abc} \pm 2.18$ | $2.65 \ ^{abc} \pm 1.67$ | $2.78 \ ^{abc} \pm 1.48$ | $3.55^{a} \pm 1.96$ | $3.24^{ab} \pm 2.10$ | $3.05 \ ^{abc} \pm 1.81$ | $2.40~^{\rm abc}\pm1.48$ | $2.60^{\text{ abc}} \pm 1.87$ | $2.12^{\text{ bc}} \pm 1.45$ |
| Saltiness *** | $0.85 \text{ bc} \pm 0.90$ | $0.85 \text{ bc} \pm 0.93$ | $0.85 \text{ bc} \pm 1.00$ | $1.52^{ab} \pm 1.19$ | $0.53 \ ^{\rm c} \pm 0.72$ | $1.35^{ab} \pm 1.17$ | $1.37 \ ^{ab} \pm 1.19$ | 1.89 ^a ± 1.42 | $0.48 \ ^{\rm c} \pm 0.62$ | $1.23^{\text{ abc}} \pm 1.07$ | $0.90 \text{ bc} \pm 0.93$ | $1.04 \text{ bc} \pm 0.93$ |
| Sourness *** | $3.26^{bcd} \pm 1.93$ | $3.81^{abc} \pm 2.25$ | $2.86^{bcd} \pm 1.87$ | 4.74 ^a ± 2.07 | $2.39^{\text{ d}} \pm 1.66$ | $2.97 ^{bcd} \pm 1.67$ | $2.97^{bcd} \pm 1.84$ | $2.62 ^{\text{cd}} \pm 2.23$ | $2.33^{\rm ~d} \pm 2.08$ | $4.23^{ab} \pm 1.60$ | $4.07^{ab} \pm 1.80$ | $2.88 \text{ bcd} \pm 1.46$ |
| Sweetness *** | $2.80^{\text{ abc}} \pm 1.57$ | 3.22 ^a ± 1.75 | $3.03^{ab} \pm 1.56$ | $2.67 \ ^{abc} \pm 1.94$ | $2.60^{abc} \pm 1.47$ | $1.85 \text{ cd} \pm 1.16$ | $1.93 \text{ bcd} \pm 1.38$ | $3.06^{ab} \pm 1.58$ | $1.36^{\rm d} \pm 0.89$ | 3.37 ^a ± 1.73 | $2.59^{\text{ abc}} \pm 1.86$ | $2.83^{abc} \pm 1.40$ |
| Umami *** | $0.83^{b} \pm 1.08$ | $0.77^{\text{ b}} \pm 1.04$ | $0.81 \ ^{\rm b} \pm 0.94$ | $1.27 \ ^{ab} \pm 1.71$ | $0.71 \text{ b} \pm 1.19$ | $1.21 \ ^{ab} \pm 1.94$ | $1.03 {}^{b} \pm 1.69$ | $2.14^{a} \pm 2.20$ | $0.50^{\text{ b}} \pm 1.10^{}$ | $1.04 {}^{\rm b} \pm 1.43$ | $0.70^{\rm b} \pm 0.93$ | $1.48^{ab} \pm 1.58$ |
| Alcohol *** | $3.78^{ab} \pm 1.82$ | $3.56^{ab} \pm 1.76$ | $3.55~^{ab}\pm1.77$ | $4.49^{ab} \pm 2.21$ | $3.89~^{ab}\pm1.78$ | $4.56\ ^{ab}\pm1.55$ | 4.86 ^a ± 2.01 | $4.79^{a} \pm 1.77$ | $4.76 \ ^{a} \pm 1.84$ | $3.25^{b} \pm 1.40$ | $4.13^{ab} \pm 1.90$ | 3.27 ^b ± 1.69 |
| Apple ** | 0.64 ^{ab} ± 1.42 | 0.89 ^a ± 1.29 | $0.81^{ab} \pm 1.50$ | 0.38 ^{ab} ± 1.06 | $0.40^{ab} \pm 0.88$ | $0.13^{ab} \pm 0.28$ | 0.03 ^b ± 0.09 | 0.03 ^b ± 0.07 | 0.13 ^{ab} ± 0.37 | 0.59 ^{ab} ± 1.21 | 0.66 ^{ab} ± 1.33 | 0.54 ^{ab} ± 1.24 |

Table 3. Mean intensity scores of the sensory attributes for 12 yakju samples ^{1–3}.

Table 3. Cont.

| Samples | Y1 | Y2 | Y3 | ¥4 | ¥5 | Y6 | ¥7 | Y8 | Y9 | Y10 | Y11 | Y12 |
|-------------------------------|-------------------------------|-------------------------------|-------------------------------|-----------------------------|-------------------------------|-------------------------------|-------------------------------|---------------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|
| Balloon flower root | 1.10 ± 1.67 | 0.23 ± 0.54 | 0.58 ± 1.94 | 0.60 ± 1.56 | 0.84 ± 1.38 | 0.70 ± 1.19 | 0.69 ± 1.21 | 0.74 ± 1.69 | 1.12 ± 2.13 | 0.33 ± 0.64 | 0.61 ± 1.48 | 0.33 ± 0.60 |
| Grape *** | $0.73^{ab} \pm 1.40$ | $0.87 \ ^{a} \pm 1.44$ | $0.61^{ab} \pm 1.50$ | $0.33^{ab} \pm 0.95$ | $0.31^{ab} \pm 0.88$ | $0.09^{b} \pm 0.31$ | $0.01^{b} \pm 0.03$ | $0.02^{b} \pm 0.09$ | $0.05^{b} \pm 0.14$ | 0.89 ^a ± 1.39 | $0.79^{ab} \pm 1.53$ | $0.26^{ab} \pm 0.80$ |
| Kudzu * | $0.23^{ab} \pm 0.44$ | $0.03^{\text{ b}} \pm 0.07$ | $0.37~^{ab}\pm1.40$ | $0.51 \ ^{ab} \pm 1.17$ | $0.40~^{ab}\pm0.81$ | $0.54^{ab} \pm 1.27$ | 0.88 ^a ± 1.92 | $0.68~^{ab}\pm1.50$ | $0.30^{\text{ ab}} \pm 0.67$ | $0.08 \ ^{ab} \pm 0.26$ | $0.15 \ ^{ab} \pm 0.63$ | $0.19^{ab} \pm 0.39$ |
| Maesil (Prunus mume fruit) | 0.99 ± 2.18 | 1.05 ± 1.72 | 0.78 ± 1.54 | 0.91 ± 1.57 | 0.60 ± 1.07 | 0.29 ± 0.55 | 0.17 ± 0.36 | 0.45 ± 0.62 | 0.23 ± 0.47 | 1.11 ± 1.63 | 0.64 ± 1.10 | 0.78 ± 1.45 |
| Roasted grain | 1.23 ± 1.77 | 0.83 ± 1.35 | 1.36 ± 1.65 | 1.57 ± 1.97 | 0.67 ± 0.83 | 1.84 ± 2.51 | 0.97 ± 1.20 | 1.97 ± 2.26 | 0.79 ± 1.31 | 1.26 ± 1.54 | 0.86 ± 1.04 | 1.09 ± 1.17 |
| Tangerine peel ** | $0.40^{\rm b} \pm 0.88$ | $0.18^{\rm b} \pm 0.42$ | $0.27 {}^{\rm b} \pm 0.90$ | $0.55^{\rm b} \pm 0.88$ | $0.51^{\rm b} \pm 0.73$ | $0.42^{\text{ b}} \pm 1.00$ | $0.53^{\rm b} \pm 1.28$ | $0.20^{b} \pm 0.39$ | $0.33^{b} \pm 0.96$ | $0.61^{\rm b} \pm 1.03$ | $0.47 {}^{b} \pm 1.20$ | $1.22 \ ^{a} \pm 1.44$ |
| Mouthfeel | | | | | | | | | | | | |
| Astringent | 1.01 ± 0.82 | 1.16 ± 0.80 | 1.12 ± 0.89 | 1.26 ± 1.17 | 0.82 ± 0.74 | 1.05 ± 0.91 | 1.13 ± 0.81 | 0.93 ± 0.83 | 0.99 ± 1.09 | 1.24 ± 0.67 | 1.15 ± 0.85 | 0.83 ± 0.63 |
| Body *** | $3.04 \ ^{\rm abc} \pm 2.12$ | $2.82 ^{bc} \pm 2.21$ | $2.14 \ ^{c} \pm 1.69$ | $3.82^{ab} \pm 2.36$ | $2.23 \ ^{\rm c} \pm 1.79$ | $3.08 \ ^{\rm abc} \pm 1.95$ | $3.15^{\rm \ abc} \pm 1.92$ | $4.43 \ ^{a} \pm 2.48$ | $1.98 \ ^{\rm c} \pm 1.43$ | $3.32^{\text{ abc}} \pm 1.97$ | $2.56 {}^{bc} \pm 2.10$ | $3.11 \ ^{\rm abc} \pm 1.91$ |
| Burning | 3.56 ± 2.65 | 3.25 ± 2.74 | 3.62 ± 2.74 | 4.67 ± 3.01 | 3.54 ± 2.58 | 4.00 ± 2.53 | 4.80 ± 2.80 | 4.84 ± 2.95 | 4.77 ± 2.60 | 2.91 ± 2.30 | 4.21 ± 2.76 | 3.14 ± 2.47 |
| Coating ** | $1.84^{b} \pm 1.42$ | $1.72^{\rm \ b} \pm 1.48$ | $1.60^{b} \pm 1.33$ | $2.64^{ab} \pm 1.71$ | $1.66^{b} \pm 1.14$ | $2.00^{ab} \pm 1.31$ | $2.14^{ab} \pm 1.46$ | $2.92 \ ^{a} \pm 1.94$ | $2.02^{ab} \pm 1.35$ | $1.67^{\rm b} \pm 1.24$ | $1.83^{b} \pm 1.33$ | $1.77 {}^{b} \pm 1.44$ |
| Pungent | 0.87 ± 1.07 | 0.75 ± 1.16 | 0.65 ± 0.98 | 1.67 ± 2.16 | 0.70 ± 0.82 | 1.08 ± 1.51 | 1.36 ± 2.34 | 1.15 ± 1.45 | 1.11 ± 1.44 | 1.21 ± 1.85 | 1.02 ± 1.34 | 0.84 ± 1.19 |
| Stinging *** | $1.54 {\ b} \pm 1.48$ | $1.26^{b} \pm 1.22$ | $1.48 {}^{b} \pm 1.48$ | $3.03^{a} \pm 2.69$ | $1.77~^{\rm ab}\pm1.69$ | $2.31~^{ab}\pm2.01$ | $2.62 \ ^{ab} \pm 2.00$ | $2.70^{ab} \pm 2.53$ | $2.42 \ ^{ab} \pm 2.19$ | $1.43^{b} \pm 1.42$ | $1.87~^{\rm ab}\pm1.64$ | $1.49^{b} \pm 1.40$ |
| Aftertaste/mouthfeel | | | | | | | | | | | | |
| Bitterness ** | $1.75~^{\rm ab}\pm1.70$ | $1.25 ^{\rm b} \pm 1.37$ | $1.65~^{ab}\pm1.47$ | $2.21~^{ab}\pm2.06$ | $1.86~^{ab}\pm1.32$ | $2.11 ^{\text{ab}} \pm 1.19$ | $2.72^{a} \pm 1.80$ | $2.29~^{ab}\pm1.80$ | $2.18~^{ab}\pm1.76$ | $1.53~^{ab}\pm1.03$ | $2.00 \ ^{ab} \pm 1.61$ | $1.43^{b} \pm 1.23$ |
| Sourness *** | $2.16 \text{ bc} \pm 1.62$ | $2.38 \text{ bc} \pm 1.72$ | $1.77 \text{ bc} \pm 1.23$ | $3.44^{a} \pm 1.78$ | $1.41 \ ^{\rm c} \pm 1.00$ | $2.06 \text{ bc} \pm 1.46$ | $2.19 \text{ bc} \pm 1.55$ | $1.78\ ^{\mathrm{bc}} \pm 1.65$ | $1.38 \ ^{\rm c} \pm 1.45$ | $2.72 \ ^{ab} \pm 1.43$ | $2.68~^{ab}\pm1.54$ | $1.91 \ ^{\rm bc} \pm 1.08$ |
| Sweetness *** | $1.78~^{\rm abc}\pm0.65$ | $2.04~^{ab}\pm0.84$ | $1.93~^{ab}\pm0.70$ | $1.63 \text{ bcd} \pm 0.78$ | $1.40 \ ^{\rm cd} \pm 0.56$ | $1.23 \ ^{de} \pm 0.63$ | $1.18 \ ^{\rm de} \pm 0.67$ | $2.16^{a} \pm 0.80$ | $0.84^{e} \pm 0.60$ | $1.94~^{ab}\pm0.95$ | $1.41 \ ^{\rm cd} \pm 0.67$ | $1.84~^{\rm abc}\pm0.75$ |
| Umami ** | $0.81^{\text{ abc}} \pm 0.99$ | $0.59^{\text{ abc}} \pm 0.50$ | $0.79^{\text{ abc}} \pm 1.11$ | $1.05^{\rm ~abc} \pm 1.45$ | $0.46\ ^{\mathrm{bc}}\pm0.81$ | $0.83^{\text{ abc}} \pm 1.36$ | $0.75^{\rm \ abc} \pm 1.15$ | $1.36^{a} \pm 0.91$ | $0.23 \ ^{\rm c} \pm 0.31$ | $0.97^{\text{ abc}} \pm 1.35$ | $0.52 \ ^{\rm bc} \pm 0.87$ | $1.09^{ab} \pm 1.15$ |
| Astringent | 0.79 ± 0.88 | 0.88 ± 0.73 | 0.81 ± 0.77 | 0.95 ± 0.95 | 0.84 ± 0.74 | 0.87 ± 0.69 | 1.07 ± 1.12 | 0.83 ± 0.76 | 0.76 ± 0.98 | 1.05 ± 0.70 | 1.01 ± 0.89 | 0.68 ± 0.50 |
| Burning | 2.74 ± 2.35 | 2.18 ± 2.08 | 2.67 ± 2.05 | 3.39 ± 2.24 | 2.40 ± 2.17 | 3.15 ± 2.28 | 3.34 ± 2.03 | 3.67 ± 2.56 | 3.60 ± 2.42 | 1.91 ± 1.46 | 3.10 ± 2.17 | 2.31 ± 2.04 |
| Coating | 1.22 ± 0.91 | 1.15 ± 1.01 | 1.26 ± 1.03 | 1.70 ± 1.27 | 1.29 ± 1.04 | 1.39 ± 0.90 | 1.52 ± 1.06 | 1.86 ± 1.23 | 1.55 ± 0.95 | 1.08 ± 0.89 | 1.35 ± 1.07 | 1.33 ± 1.02 |
| Residue ** | $0.92 \ ^{ab} \pm 0.92$ | $0.92 \ ^{ab} \pm 0.84$ | $0.81 \ ^{ab} \pm 0.74$ | $1.47~^{ab}\pm1.26$ | $0.83^{ab} \pm 0.78$ | $1.33^{ab} \pm 1.55$ | $1.09~^{\rm ab}\pm1.00$ | $1.58\ ^{a}\pm 1.09$ | $0.72^{b} \pm 0.67$ | $1.21 \ ^{ab} \pm 1.21$ | $0.80~^{ab}\pm0.70$ | $1.04~^{ab}\pm0.96$ |
| Stinging *** | $1.30^{\text{ abc}} \pm 1.49$ | $0.86 \ ^{\rm c} \pm 0.79$ | $1.06^{bc} \pm 1.00$ | 2.32 ^a ± 2.44 | $1.32^{\text{ abc}} \pm 1.50$ | $1.74^{\text{ abc}} \pm 1.88$ | $2.07^{\text{ abc}} \pm 1.58$ | $2.16^{ab} \pm 1.96$ | $1.97^{\text{ abc}} \pm 1.76$ | $0.88 \ ^{\rm c} \pm 0.90$ | $1.33^{\text{ abc}} \pm 1.54$ | $1.19^{\text{ abc}} \pm 1.13$ |

¹ Mean values with different alphabet mean significantly different. ² *, **, *** means significantly different at p < 0.05, p < 0.01, and p < 0.001 respectively. ³ 15 cm line scale was used; 0 cm = none, 15 cm = very stron.

On the contrary, samples containing Supplementary Materials other than fruit or floral ingredients, such as sweet pumpkin (Y4), balloon flower root (Y6 and Y7), or soy bean (Y8), were located in quadrant 2. These samples were mainly characterized by yeast odor, roasted grain odor, alcohol flavor, and body and stinging attributes. Characteristics related to mouthfeel and taste were located on the positive side of PC2, whereas most of the attributes related to odor were located on the negative side of PC2. Particularly, salty taste, sour taste/aftertaste, and body mouthfeel were located on the positive side of PC2, whereas pine odor, peppermint odor, and chrysanthemum odor were located on the negative side of PC2. Samples with high alcohol levels were located on the positive side of PC2, whereas those with relatively low alcohol content, such as Y5 and Y12, were located on the negative side of PC2. Among the samples, Y9 was the only sample located in quadrant 3, and it had a low score for sour, sweet, umami, and fruit-related notes compared with other samples.

Overall, the present results indicated that the yakju samples were characterized mainly by their supplementary raw materials and alcohol content, which affect the overall odor and flavor of the samples.

3.2. Consumers' Acceptance

The mean overall acceptance scores by 80 young consumers are shown in Table 4. Significant differences among the 12 samples were found in the overall acceptance scores. Overall acceptance was highest for Y3 (6.71), followed by Y10 (6.41), and Y2 (6.34) and was lowest for Y8 (3.25). Generally, overall acceptance was higher for samples with fruit-related supplementary ingredients (Y2, Y3, and Y10) than that for samples with root-related bitter ingredients (Y7 and Y8). Apparently, consumers preferred those samples with fruit-related characteristics over the bitter and yeasty samples. Similarly, Lee and Lee [19] studied the sensory attributes and acceptance of 10 *yakju* samples with supplementary ingredients, and they reported that the acceptance of various clusters of consumers were positively associated with fruit flavor, sweet aroma, and medicinal herb aroma but astringent mouthfeel, bitter taste, and yeast flavor were negatively associated with consumers' acceptance. Moreover, Kwak et al. [20] reported that the key liking factors of rice wine by American panelists were sweet, sour, and apricot flavors, whereas the key disliking factors were yeasty and nutty characteristics. They reported positive correlations between overall acceptance and fruit-related characteristics (apple, peach, and pear), confirming the key liking factors for the *yakju* samples [20].

| Samples | Overall Acceptance Scores |
|---------|----------------------------------|
| Y1 | $5.12^{\text{d}} \pm 2.10$ |
| Y2 | $6.34^{ab} \pm 1.82$ |
| Y3 | $6.71^{a} \pm 1.34$ |
| Y4 | $4.01 e \pm 2.13$ |
| Y5 | $5.59 \text{ cd} \pm 1.80$ |
| Y6 | $4.02 e \pm 1.71$ |
| Y7 | $3.43^{\text{ f}} \pm 1.57$ |
| Y8 | $3.25^{\text{ f}} \pm 2.25$ |
| Y9 | $4.06 e \pm 1.72$ |
| Y10 | $6.41^{ab} \pm 1.76$ |
| Y11 | $4.30^{e} \pm 1.66$ |
| Y12 | $5.82^{bc} \pm 1.63$ |

Table 4. Mean scores for the consumers' acceptance of the *yakju* samples 1,2 .

¹ Average scores of 80 consumers; 1 = dislike extremely, 9 = like extremely. ² Mean values with different alphabet mean significantly different at p < 0.05.

3.3. Volatile Compounds Identified Using GC/MS

A total of 120 volatile compounds (acids = 14, alcohols = 26, aldehydes = 2, esters = 32, furans = 2, ketones = 11, lactones = 7, phenols = 8, terpenoids = 6, and miscellaneous = 12) were identified (Table 5). Thirty-five volatile compounds were found in all of the samples.

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1376

3-hexen-1-ol

 8.91 ± 2.54

 9.43 ± 1.44

ND

ND

 9.57 ± 0.95

ND

ND

ND

ND

ND

ND

ND

| DI ² | Compound | | | | | | Concentra | tion (µg/L) | | | | | |
|------------------------|-----------------------------|-------------------|-------------------|-------------------|---------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|---------------------|-------------------|
| KI | compound | Y1 | Y2 | ¥3 | ¥4 | ¥5 | ¥6 | ¥7 | Y8 | ¥9 | Y10 | Y11 | Y12 |
| | Acids | | | | | | | | | | | | |
| 1434 | acetic acid | 1314 ± 319 | 837 ± 176 | 414 ± 60.8 | $14,700 \pm 1524$ | 894 ± 117 | 1230 ± 330 | 1281 ± 447 | 5038 ± 301 | 1558 ± 371 | 1436 ± 185 | 2266 ± 407 | 624 ± 69.8 |
| 1492 | formic acid | ND | ND | ND | ND | ND | ND | 11.3 ± 6.7 | ND | ND | ND | ND | ND |
| 1524 | propanoic acid | 43.0 ± 7.06 | 41.7 ± 10.0 | 32.6 ± 3.78 | 751 ± 115 | 843 ± 95.1 | 72.6 ± 7.00 | 90.7 ± 17.0 | 65.3 ± 4.60 | 200 ± 38.4 | 76.9 ± 7.36 | 113 ± 12.6 | 78.5 ± 5.50 |
| 1554 | 2-methyl propanoic acid | 479 ± 85.3 | 494 ± 80.5 | 453 ± 50.0 | 187 ± 59.4 | 662 ± 82.6 | 110 ± 21.0 | 138 ± 24.9 | 309 ± 20.6 | 105 ± 13.6 | 465 ± 48.4 | 451 ± 70.3 | 313 ± 18.8 |
| 1613 | butanoic acid | 148 ± 25.3 | 172 ± 27.8 | 137 ± 16.4 | 84.4 ± 27.9 | 140 ± 19.9 | 104 ± 23.3 | 114 ± 18.9 | 207 ± 14.3 | 20.1 ± 2.82 | 146 ± 16.0 | 178 ± 27.0 | 162 ± 11.9 |
| 1656 | 3-methyl butanoic acid | 422 ± 61.5 | 385 ± 73.7 | 303 ± 27.1 | 254 ± 67.2 | 540 ± 59.8 | 143 ± 52.2 | 145 ± 16.9 | 330 ± 28.2 | 104 ± 8.31 | 324 ± 49.7 | 399 ± 21.2 | 226 ± 45.2 |
| 1724 | pentanoic acid | ND | 16.2 ± 0.69 | ND | 22.3 ± 9.64 | ND | 43.2 ± 6.05 | 56.6 ± 1.40 | 44.2 ± 8.33 | ND | 73.1 ± 5.70 | ND | 29.1 ± 6.62 |
| 1759 | 2-butenoic acid | ND | ND | ND | ND | ND | ND | 12.4 ± 1.79 | ND | ND | ND | ND | ND |
| 1771 | 2-methyl-2-butenoic acid | 33.2 ± 7.04 | 26.1 ± 8.91 | 33.2 ± 14.3 | ND | 35.2 ± 10.3 | 25.9 ± 6.13 | 39.2 ± 11.4 | ND | 10.9 ± 0.86 | 76.3 ± 32.1 | 51.0 ± 32.2 | 17.7 ± 5.12 |
| 1832 | hexanoic acid | 189 ± 28.7 | 232 ± 35.3 | 182 ± 18.5 | 301 ± 151 | 308 ± 37.4 | 168 ± 7.83 | 206 ± 21.6 | 282 ± 22.2 | 56.7 ± 6.48 | 194 ± 19.7 | 232 ± 48.7 | 304 ± 34.5 |
| 2034 | octanoic acid | 88.1 ± 9.82 | 144 ± 24.4 | 122 ± 4.65 | 113 ± 78.7 | 89.6 ± 16.8 | 142 ± 2.54 | 155 ± 31.0 | 60.3 ± 21.3 | 112 ± 17.9 | 122 ± 15.4 | 171 ± 27.8 | 293 ± 16.7 |
| 2170 | lactic acid | 350 ± 131 | 1777 ± 421 | 1018 ± 149 | 2277 ± 1032 | 211 ± 135 | 215 ± 62.7 | 81.8 ± 48.2 | 73.3 ± 91.7 | ND | 80.3 ± 16.6 | ND | ND |
| 2256 | decanoic acid | 19.4 ± 9.54 | 19.9 ± 4.46 | 15.8 ± 2.74 | 33.5 ± 16.5 | 14.7 ± 1.26 | ND | ND | ND | ND | ND | ND | 30.6 ± 6.40 |
| 2422 | benzoic acid | 36.6 ± 5.43 | 24.2 ± 7.04 | 26.2 ± 5.82 | 46.7 ± 7.00 | 38.3 ± 8.53 | 16.2 ± 4.79 | 39.5 ± 12.1 | 19.6 ± 3.92 | 27.6 ± 8.33 | 31.0 ± 5.63 | 17.8 ± 1.53 | 57.0 ± 9.00 |
| | Subtotal | 3122 ± 362 | 4170 ± 473 | 2737 ± 174 | $18,\!770 \pm 1855$ | 3776 ± 232 | 2269 ± 342 | 2372 ± 453 | 6429 ± 319 | 2194 ± 374 | 3024 ± 204 | 3879 ± 420 | 2135 ± 95.4 |
| | Alcohols | | | | | | | | | | | | |
| 1025 | 1-propanol | 777 ± 142 | 727 ± 125 | 735 ± 68.1 | 510 ± 106 | 663 ± 37.7 | 814 ± 161 | 985 ± 101 | 500 ± 42.7 | 487 ± 86.9 | 963 ± 110 | 1969 ± 155 | 954 ± 95.0 |
| 1085 | 2-methyl-1-propanol | $10,\!085\pm1526$ | $12,313 \pm 1612$ | 9109 ± 517 | 4251 ± 1018 | $10,221 \pm 919$ | 6989 ± 1035 | 7703 ± 631 | 9469 ± 706 | 4303 ± 558 | 7171 ± 815 | 7366 ± 754 | 8548 ± 886 |
| 1134 | 1-butanol | 247 ± 28.8 | 300 ± 52.4 | 735 ± 43.4 | 44.8 ± 9.4 | 132 ± 11.4 | 456 ± 75.5 | 460 ± 48.8 | 119 ± 6.45 | 288 ± 41.2 | 1160 ± 107 | 289 ± 36.0 | 511 ± 77.8 |
| 1210 | isoamyl alcohol | $41,034 \pm 5751$ | $44,\!956\pm5725$ | $39,\!224\pm1480$ | $15{,}567\pm8214$ | $45,\!159\pm4540$ | $31,676 \pm 5269$ | $36,373 \pm 2475$ | $34,\!114\pm1974$ | $18,\!342\pm2092$ | $32,\!551\pm2998$ | $35{,}548 \pm 4850$ | $30,988 \pm 1902$ |
| 1241 | 3-methyl-3-buten-1-ol | 43.3 ± 4.29 | 30.0 ± 6.04 | 28.9 ± 2.88 | 43.7 ± 20.1 | 75.3 ± 3.78 | 49.6 ± 3.62 | 67.2 ± 15.8 | 70.9 ± 2.74 | 14.8 ± 2.45 | 48.8 ± 2.22 | 53.9 ± 10.58 | 25.5 ± 2.20 |
| 1248 | 1-pentanol | ND | ND | ND | ND | ND | ND | ND | 16.7 ± 2.49 | ND | 17.5 ± 6.76 | ND | ND |
| 1311 | 2-methyl-2-buten-1-ol | 17.0 ± 2.28 | ND | ND | 50.7 ± 15.1 | ND | ND | 14.2 ± 3.21 | 10.6 ± 0.61 | ND | 10.5 ± 0.69 | 24.5 ± 5.31 | 25.6 ± 1.86 |
| 1322 | 3-methyl-1-pentanol | 12.0 ± 4.04 | 18.2 ± 2.01 | 16.6 ± 1.19 | ND | 16.5 ± 1.57 | 8.91 ± 0.21 | 12.4 ± 0.78 | 8.01 ± 1.09 | ND | 14.2 ± 1.71 | 12.6 ± 8.13 | 8.59 ± 0.41 |
| 1342 | 1-hexanol | 71.8 ± 11.1 | ND | ND | ND | 171 ± 16.3 | 111 ± 11.6 | 169 ± 16.1 | 164 ± 7.98 | 17.2 ± 1.52 | 34.0 ± 7.23 | 36.6 ± 6.48 | 20.6 ± 1.37 |
| 1370 | 3-ethoxy-1-propanol | 1684. ± 296 | 507 ± 78.2 | 868 ± 86.6 | 248 ± 89.7 | 294 ± 36.3 | 619 ± 123 | 822 ± 149 | 571 ± 48.7 | 200 ± 38.0 | 1677 ± 75.5 | 3874 ± 678 | 1254 ± 83.8 |

Table 5. Volatile compounds of the 12 *yakju* samples 1 .

Table 5. Cont.

| DI ² | Compound | Concentration (µg/L) | | | | | | | | | | | |
|------------------------|-------------------------------------|----------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|---------------------|---------------------|-------------------|-------------------|-------------------|
| KI | compound | Y1 | Y2 | Y3 | Y4 | ¥5 | ¥6 | Y7 | Y8 | Y9 | Y10 | Y11 | Y12 |
| 1444 | 1-heptanol | 17.4 ± 2.44 | 11.9 ± 1.33 | 10.0 ± 0.73 | ND | 37.1 ± 4.39 | 17.9 ± 1.68 | 24.3 ± 6.99 | 26.5 ± 0.80 | 15.7 ± 6.10 | 16.4 ± 5.28 | 22.8 ± 2.80 | 16.2 ± 0.66 |
| 1477 | 2-ethyl-1-hexanol | ND | ND | ND | ND | 70.5 ± 2.89 | 10.4 ± 0.82 | 15.4 ± 4.60 | 41.8 ± 1.34 | 15.3 ± 2.75 | 18.9 ± 3.70 | ND | ND |
| 1532 | 1,3-butanediol | 2895 ± 664 | 1260 ± 257 | 1124 ± 181 | 3457 ± 3398 | 1382 ± 247 | 2155 ± 550 | 2586 ± 766 | 3387 ± 792 | 803 ± 354 | 1653 ± 138 | 3193 ± 1286 | 1483 ± 103 |
| 1535 | 2,6-dimethyl-4-heptanol | ND | 104 ± 11.4 | 141 ± 1.3 | ND | 60.7 ± 25.8 | ND | ND | ND | ND | ND | ND | ND |
| 1546 | 1-octanol | ND | ND | ND | 30.4 ± 9.35 | 12.5 ± 1.71 | 11.6 ± 1.27 | 17.6 ± 1.61 | 16.4 ± 1.55 | ND | 19.7 ± 8.90 | 26.3 ± 5.46 | 13.9 ± 0.83 |
| 1565 | 2,3-butanediol | 684 ± 169 | 199 ± 37.0 | 205 ± 35.8 | 1070 ± 561 | 256 ± 46.0 | 425 ± 121 | 506 ± 176 | 989 ± 302 | 144 ± 66.6 | 319 ± 36.1 | 685 ± 355 | 251 ± 17.4 |
| 1581 | propylene glycol | 138 ± 31.1 | 143 ± 29.5 | 120 ± 19.7 | 180 ± 109 | 117 ± 21.5 | 154 ± 40.2 | 168 ± 59.2 | 315 ± 109 | 58.8 ± 23.0 | 115 ± 15.4 | 177 ± 97.4 | 72.9 ± 4.45 |
| 1604 | 4-methyl-3-hexanol | 193 ± 41.8 | 48.0 ± 8.95 | 22.4 ± 3.04 | 27.6 ± 8.63 | 79.7 ± 37.0 | 121 ± 47.0 | 125 ± 47.6 | 47.0 ± 3.92 | 76.5 ± 7.81 | 79.6 ± 5.82 | 87.8 ± 23.1 | 65.4 ± 4.64 |
| 1649 | 2-furanmethanol | 81.8 ± 34.4 | 38.9 ± 8.42 | 22.5 ± 7.85 | 231 ± 132 | 199 ± 34.0 | 152 ± 36.1 | 324 ± 197 | 347 ± 30.2 | 23.7 ± 2.42 | 1048 ± 307 | 248 ± 157 | 144 ± 25.0 |
| 1687 | 2,3-hexanediol | ND | ND | ND | ND | 10.3 ± 1.67 | 7.12 ± 3.19 | 11.1 ± 4.59 | 4.89 ± 0.95 | ND | ND | ND | ND |
| 1711 | methionol | 2450 ± 395 | 1894 ± 191 | 1930 ± 141 | 651 ± 196 | 511 ± 65.0 | 431 ± 59.0 | 603 ± 60.6 | 1263 ± 73.7 | 631 ± 49.8 | 1441 ± 218 | 1818 ± 223 | 1915 ± 251 |
| 1872 | phenylmethanol | 54.6 ± 16.3 | 53.4 ± 19.6 | 14.7 ± 2.00 | 42.1 ± 22.7 | 48.5 ± 9.78 | 56.1 ± 6.11 | 99.9 ± 7.43 | 25.2 ± 5.37 | 70.8 ± 6.65 | 26.9 ± 2.80 | 28.3 ± 16.9 | 38.7 ± 8.93 |
| 1919 | 2-phenylethanol | $28,\!572\pm4339$ | $24,\!887\pm3540$ | $20,\!061\pm414$ | $20,658 \pm 2895$ | $16{,}549\pm1734$ | $20{,}417\pm410$ | $22,932 \pm 2428$ | $17{,}507 \pm 1758$ | $16,\!920\pm1319$ | $20,\!396\pm2146$ | $23,\!663\pm1692$ | $24,511 \pm 973$ |
| 2310 | glycerol | ND | ND | ND | ND | 25.7 ± 7.64 | 75.9 ± 16.1 | 41.7 ± 17.9 | 41.1 ± 29.4 | ND | ND | ND | ND |
| 2325 | 2-(4-methoxy phenyl)ethanol | ND | ND | ND | ND | 29.1 ± 1.17 | 11.8 ± 0.96 | ND | ND | ND | ND | ND | ND |
| | Subtotal | $89,064\pm7414$ | $87,499 \pm 6931$ | $74,\!368\pm1642$ | $47,062 \pm 9426$ | $76,131 \pm 4953$ | $64,\!769\pm5420$ | $74,058 \pm 3622$ | $69,\!054\pm2869$ | $42,\!411 \pm 2563$ | $68,\!782\pm3801$ | $79,\!122\pm5413$ | $70,\!847\pm2334$ |
| | Aldehydes | | | | | | | | | | | | |
| 1456 | furfural | 107 ± 3.15 | 88.0 ± 9.41 | 69.4 ± 3.36 | 69.1 ± 4.76 | 84.9 ± 8.55 | 318 ± 20.2 | 468 ± 104 | 145 ± 46.8 | 16.7 ± 0.90 | 447 ± 31.4 | 84.8 ± 32.5 | 189 ± 22.8 |
| 2491 | 5-(hydroxymethyl)- 2-furaldehyde | ND | 28.0 ± 7.32 | 39.2 ± 1.78 | ND | 127 ± 18.4 | 39.0 ± 7.85 | ND | ND | ND | 84.2 ± 16.8 | ND | ND |
| | Subtotal | 107 ± 3.15 | 116 ± 11.9 | 109 ± 3.80 | 69.1 ± 4.76 | 212 ± 20.3 | 357 ± 21.7 | 468 ± 104 | 145 ± 46.8 | 16.7 ± 0.90 | 531 ± 35.6 | 84.8 ± 32.5 | 189 ± 22.8 |
| | Esters | | | | | | | | | | | | |
| 946 | ethyl propanoate | 33.6 ± 3.12 | 38.7 ± 6.17 | 17.7 ± 1.22 | ND | 28.1 ± 10.4 | 17.6 ± 10.5 | ND | ND | ND | 32.2 ± 6.42 | ND | 43.3 ± 1.78 |
| 953 | ethyl 2-methyl propanoate | ND | 40.2 ± 4.19 | 28.3 ± 2.25 | ND | 40.2 ± 9.84 | 4.6 ± 0.85 | ND | ND | ND | 23.4 ± 8.53 | 5.32 ± 1.71 | 18.1 ± 0.90 |
| 970 | propyl acetate | ND | 20.0 ± 2.31 | ND | ND | 13.4 ± 3.49 | 26.1 ± 2.95 | ND | ND | 13.8 ± 8.65 | 38.4 ± 12.2 | ND | 31.7 ± 1.98 |
| 998 | 2-methylpropyl acetate | 95.7 ± 17.3 | 59.4 ± 5.64 | 28.6 ± 1.90 | ND | 60.7 ± 12.9 | 55.0 ± 2.19 | 55.1 ± 4.80 | ND | 15.6 ± 8.30 | 110 ± 58.7 | 71.2 ± 33.0 | 71.9 ± 5.95 |
| 1022 | ethyl butanoate | 53.6 ± 9.59 | 57.2 ± 5.79 | 37.9 ± 2.78 | ND | 40.6 ± 5.98 | 38.9 ± 2.13 | 32.2 ± 4.70 | 16.2 ± 4.23 | 6.98 ± 1.31 | 36.8 ± 8.62 | 36.9 ± 3.75 | 61.6 ± 5.02 |
| 1039 | ethyl 2-methyl butanoate | 19.7 ± 3.92 | ND | ND | ND | ND | ND |
| 1112 | isoamyl acetate | 540 ± 115 | 359 ± 36.1 | 298 ± 6.71 | 373 ± 415 | 365 ± 49.8 | 486 ± 21.3 | 467 ± 134 | 30.4 ± 10.5 | 100 ± 35.4 | 807 ± 335 | 625 ± 142 | 562 ± 45.3 |

Table 5. Cont.

| PI ² | Compound | Concentration (µg/L) | | | | | | | | | | | |
|------------------------|---|----------------------|-------------------|------------------|-------------------------|------------------|------------------|-------------------|------------------|-----------------|-----------------|------------------|-----------------|
| KI | | Y1 | Y2 | Y3 | ¥4 | Y5 | Y6 | ¥7 | Y8 | Y9 | Y10 | Y11 | Y12 |
| 1125 | ethyl pentanoate | ND | ND | ND | ND | ND | 5.51 ± 0.19 | 6.95 ± 0.60 | ND | ND | ND | ND | ND |
| 1227 | ethyl hexanoate | 80.1 ± 10.4 | 57.0 ± 5.74 | 50.8 ± 3.94 | 72.3 ± 62.3 | 87.0 ± 10.7 | 51.2 ± 0.96 | 57.1 ± 18.7 | 91.3 ± 7.62 | 11.5 ± 2.17 | 72.2 ± 21.8 | 68.0 ± 13.9 | 82.8 ± 5.19 |
| 1258 | ethyl pyruvate | 49.8 ± 6.56 | 38.4 ± 3.34 | 31.1 ± 3.29 | ND | 19.8 ± 2.20 | 21.3 ± 2.25 | 37.7 ± 6.26 | 12.0 ± 2.08 | 82.6 ± 6.55 | 161 ± 21.0 | 43.8 ± 3.52 | 78.2 ± 7.34 |
| 1309 | methyl lactate | ND | 132 ± 33.9 | 46.5 ± 6.77 | ND | ND | ND | ND | 12.6 ± 0.93 | ND | ND | ND | ND |
| 1338 | ethyl lactate | 5843 ± 864 | $16,778 \pm 1949$ | $11,329 \pm 631$ | $45,\!468 \pm 11,\!102$ | 3536 ± 394 | 2311 ± 220 | 2563 ± 182 | 3585 ± 170 | 747 ± 62.0 | 2330 ± 78.8 | 936 ± 151 | 1337 ± 76.3 |
| 1404 | ethyl 2-hydroxy butanoate | 9.52 ± 1.78 | 10.2 ± 0.91 | 6.77 ± 0.59 | 11.9 ± 1.60 | 6.95 ± 1.34 | 8.49 ± 1.15 | 10.8 ± 2.01 | ND | ND | ND | 14.5 ± 2.13 | 6.98 ± 0.34 |
| 1419 | ethyl-2-hydroxy- 2-methylbutanoate | 30.6 ± 5.88 | 28.7 ± 2.46 | 27.8 ± 7.79 | ND | 31.6 ± 4.20 | 16.2 ± 1.11 | 19.8 ± 4.20 | ND | 7.15 ± 0.96 | 45.5 ± 13.4 | ND | 19.2 ± 0.72 |
| 1424 | ethyl octanoate | 26.3 ± 3.95 | 19.3 ± 1.86 | 18.1 ± 1.69 | ND | 151 ± 17.2 | 12.2 ± 0.67 | 23.5 ± 3.10 | ND | 34.0 ± 5.66 | 28.7 ± 9.52 | ND | 58.2 ± 2.09 |
| 1429 | ethyl 2-(1-ethoxyethoxy) propanoate | ND | 16.0 ± 6.40 | 5.49 ± 1.71 | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 1451 | isobutyl lactate | 91.7 ± 10.8 | 165 ± 5.73 | 71.2 ± 7.76 | 128 ± 48.1 | ND | ND | ND | 146 ± 80.0 | 13.3 ± 6.67 | 99.3 ± 5.41 | 28.8 ± 9.60 | ND |
| 1511 | ethyl 3-hydroxybutanoate | 178 ± 38.6 | 229 ± 29.4 | 80.8 ± 6.74 | 159 ± 46.6 | 180 ± 20.7 | 257 ± 32.8 | 289 ± 54.3 | 1035 ± 21.5 | 18.5 ± 2.62 | 196 ± 23.8 | 205 ± 31.3 | 243 ± 12.2 |
| 1562 | isoamyl lactate | 138. ± 21.7 | 261. ± 25.7 | 144. $\pm~20.9$ | 446. ± 203 | 55.6 ± 9.06 | 30.6 ± 2.61 | 37.4 ± 10.46 | 68.8 ± 5.53 | 11.6 ± 2.60 | 120 ± 94.1 | 49.0 ± 18.10 | 31.6 ± 0.57 |
| 1626 | ethyl methyl succinate | 35.7 ± 5.31 | ND | ND | 21.8 ± 9.27 | ND | 9.14 ± 2.86 | 14.7 ± 1.98 | ND | ND | ND | 47.8 ± 8.72 | 38.8 ± 3.19 |
| 1665 | diethyl succinate | 5462 ± 1207 | 456 ± 50.1 | 491 ± 42.5 | 8015 ± 1590 | 344 ± 53.8 | 255 ± 10.1 | 371 ± 22.4 | 1183 ± 85.2 | 199 ± 42.2 | 411 ± 127 | 317 ± 5.98 | 483 ± 23.9 |
| 1669 | ethyl 3-hydroxyhexanoate | ND | 109 ± 16.8 | 49.2 ± 9.78 | ND | 73.5 ± 11.9 | 26.3 ± 11.0 | 46.1 ± 18.6 | 20.7 ± 7.02 | 24.3 ± 3.62 | 53.5 ± 3.56 | 31.3 ± 7.69 | 23.7 ± 6.00 |
| 1782 | ethyl phenylacetate | 28.0 ± 14.0 | 59.6 ± 36.9 | 39.4 ± 25.3 | 61.2 ± 16.3 | 27.8 ± 18.3 | 17.2 ± 5.70 | 13.7 ± 7.86 | ND | ND | ND | ND | ND |
| 1799 | ethyl 4-hydroxybutanoate | 4225 ± 635 | 3229 ± 860 | 2905 ± 119 | 1454 ± 249 | 3713 ± 426 | 4079 ± 655 | 4698 ± 522 | 3958 ± 376 | 2091 ± 814 | 3455 ± 307 | 1916 ± 214 | 3002 ± 463 |
| 1815 | 2-phenylethyl acetate | 378 ± 67.6 | ND | 180 ± 35.7 | 125 ± 71.1 | 118 ± 36.5 | 600 ± 25.9 | 759 ± 41.7 | 26.9 ± 6.11 | 188 ± 67.5 | 538 ± 139.3 | 557 ± 109 | 257 ± 29.7 |
| 1899 | ethyl 3-methylbutyl succinate | 47.2 ± 12.3 | 1287 ± 189 | 1171 ± 53.1 | 101 ± 47.9 | 128 ± 14.7 | 10.9 ± 2.66 | 19.8 ± 3.16 | 13.7 ± 3.04 | 26.3 ± 3.24 | 39.6 ± 38.3 | 47.2 ± 12.3 | 1287 ± 189 |
| 1997 | methyl 2-furoate | 64.3 ± 13.9 | 51.8 ± 6.98 | 41.9 ± 2.67 | ND | 70.7 ± 10.7 | 13.6 ± 1.05 | 16.6 ± 2.29 | 19.6 ± 9.42 | ND | 186 ± 32.0 | 64.3 ± 13.9 | 51.8 ± 6.98 |
| 2031 | diethyl malate | 120 ± 33.3 | 133 ± 41.2 | 161 ± 12.2 | ND | ND | ND | ND | ND | 100 ± 15.4 | 36.8 ± 7.37 | 120 ± 33.3 | 133 ± 41.2 |
| 2098 | diethyl 2-hydroxypentanedioate | 43.3 ± 12.0 | 117 ± 17.0 | 85.5 ± 12.9 | ND | 43.5 ± 6.64 | 22.6 ± 1.90 | 22.7 ± 6.90 | ND | 11.8 ± 2.49 | 32.0 ± 19.7 | 43.3 ± 12.0 | 117 ± 17.0 |
| 2278 | ethyl 2-hydroxy-3-phenyl propanoate | 313 ± 21.6 | 112 ± 13.8 | 157 ± 4.37 | 416 ± 58.1 | 119 ± 10.4 | 144 ± 12.3 | 152 ± 54.1 | 43.1 ± 16.3 | 298 ± 71.6 | 139 ± 28.0 | 82.3 ± 20.2 | 273 ± 43.4 |
| 2367 | ethyl hydrogen succinate | 5490 ± 772 | 2504 ± 483 | 3442 ± 245 | 6056 ± 622 | 4328 ± 284 | 2009 ± 171 | 1956 ± 826 | 1268 ± 544 | 712 ± 260 | 720 ± 115 | 250 ± 64.2 | 887 ± 147 |
| 2454 | ethyl citrate | ND | ND | ND | ND | ND | ND | ND | ND | ND | 34.1 ± 10.0 | ND | ND |
| | Subtotal | 23,396 ± 1796 | $26,367 \pm 2195$ | $20,944 \pm 693$ | $62,908 \pm 11,246$ | $13,\!580\pm652$ | $10{,}527\pm714$ | $11,669 \pm 1007$ | $11,530 \pm 694$ | 4713 ± 865 | 9744 ± 528 | 5333 ± 329 | 7707 ± 498 |

Table 5. Cont.

| PI ² | Compound | Compound Concentration (µg/L) | | | | | | | | | | | |
|-----------------|---|-------------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| NI | r | Y1 | Y2 | ¥3 | ¥4 | ¥5 | Y6 | Y7 | Y8 | ¥9 | Y10 | Y11 | Y12 |
| | Furans | | | | | | | | | | | | |
| 1453 | 2-(diethoxymethyl)furan | 73.4 ± 22.7 | ND ³ | 41.3 ± 7.99 | ND | 135 ± 21.3 | 235 ± 107 | 130 ± 77.7 | ND | ND | ND | 203 ± 136 | 370 ± 13.9 |
| 1498 | 2-acetylfuran | 38.1 ± 9.28 | 14.0 ± 0.69 | 12.5 ± 1.05 | ND | 16.0 ± 2.10 | 7.47 ± 0.97 | 10.0 ± 2.11 | ND | 30.0 ± 8.24 | 35.5 ± 17.8 | 36.1 ± 37.0 | 17.7 ± 1.01 |
| | Subtotal | 112 ± 24.6 | 14.0 ± 0.69 | 53.8 ± 8.06 | ND | 151 ± 21.4 | 242 ± 107 | 140 ± 77.7 | ND | 30.0 ± 8.24 | 35.5 ± 17.8 | 239 ± 141 | 388 ± 14.0 |
| | Ketones | | | | | | | | | | | | |
| 971 | 2,3-butanedione | 70.8 ± 11.8 | ND | 14.2 ± 3.68 | 27.3 ± 19.5 | ND | 26.7 ± 3.69 | ND | 33.6 ± 21.0 | 41.3 ± 25.3 | 24.7 ± 12.6 | 27.8 ± 10.8 | 27.6 ± 3.39 |
| 1116 | 3-penten-2-one | 19.4 ± 2.77 | 36.6 ± 6.23 | 29.6 ± 0.84 | ND | 8.30 ± 2.38 | 29.7 ± 5.01 | 16.8 ± 3.28 | ND | ND | 16.8 ± 1.16 | 9.82 ± 0.70 | 21.2 ± 3.56 |
| 1280 | 3-hydroxy-2-butanone (acetoin) | 868 ± 103 | 233 ± 24.2 | 58.4 ± 6.73 | 135 ± 75.3 | 114 ± 13.4 | 33.2 ± 5.00 | 76.7 ± 13.3 | 153 ± 12.8 | 728 ± 101 | 129 ± 8.78 | 246 ± 38.9 | 99.2 ± 13.1 |
| 1294 | 1-hydroxy-2-propanone (acetol) | 93.2 ± 14.8 | 48.1 ± 18.9 | 35.0 ± 13.9 | ND | 64.9 ± 8.79 | 9.13 ± 3.73 | 8.36 ± 1.69 | 18.8 ± 7.88 | 190 ± 29.6 | 66.4 ± 33.0 | 70.3 ± 23.8 | 18.4 ± 9.63 |
| 1343 | 3,3,6-trimethyl-1,5- heptadien-4-one | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | 141 ± 31.9 | ND |
| 1721 | 3-methyl-2(5 H)-furanone | ND | ND | ND | ND | 56.7 ± 7.34 | ND | ND | ND | 9.57 ± 0.57 | ND | 7.82 ± 0.67 | 9.61 ± 1.38 |
| 1736 | piperitone | ND | ND | ND | ND | 25.3 ± 7.28 | ND |
| 1825 | tetrahydro-4-methyl- 2H-pyran-2-one | ND | ND | ND | 16.4 ± 1.45 | ND | 27.7 ± 2.11 | ND | 41.9 ± 7.64 | ND | 24.5 ± 6.50 | 57.5 ± 2.59 | 62.0 ± 7.78 |
| 1969 | maltol | ND | ND | ND | 41.3 ± 24.0 | ND |
| 2042 | 5-acetyldihydro- 2(3H)-furanone | ND | ND | ND | ND | 16.0 ± 2.28 | ND |
| 2473 | 5-hydroxymethyl dihydrofuran-2-one | 41.0 ± 13.0 | 19.8 ± 8.30 | 45.2 ± 9.52 | ND | ND | ND | 29.6 ± 6.46 | ND | ND | ND | ND | ND |
| | Subtotal | 1093 ± 105 | 338 ± 32.4 | 182 ± 18.6 | 220 ± 81.4 | 285 ± 19.4 | 126 ± 9.07 | 131 ± 15.2 | 247 ± 26.9 | 969 ± 109 | 262 ± 37.0 | 560 ± 56.7 | 238 ± 18.7 |
| | Lactones | | | | | | | | | | | | |
| 1637 | γ -butyrolactone | 2386 ± 530 | 2782 ± 232 | 2813 ± 208 | 544 ± 156 | 2874 ± 415 | ND | 2161 ± 121 | 596 ± 58.7 | 580 ± 200 | 3130 ± 270 | 300 ± 34.3 | 993 ± 21.4 |
| 1707 | γ -hexalactone | ND | ND | ND | 12.4 ± 3.39 | 11.5 ± 1.31 | 13.5 ± 1.22 | 12.4 ± 0.99 | 13.0 ± 1.08 | ND | ND | 17.5 ± 1.99 | 14.4 ± 1.95 |
| 1725 | γ -ethoxybutyrolactone | 22.3 ± 6.66 | ND | 24.0 ± 2.98 | 11.0 ± 1.45 | 76.4 ± 9.84 | ND | ND | 21.7 ± 4.22 | 10.5 ± 3.74 | ND | 36.7 ± 6.16 | ND |
| 2026 | pantolactone | 55.3 ± 9.02 | 74.1 ± 12.3 | ND | 51.5 ± 7.13 | 248 ± 37.5 | 113 ± 10.8 | 193 ± 71.6 | 57.3 ± 18.1 | ND | ND | 85.0 ± 34.1 | 203 ± 15.0 |
| 2032 | γ -nonalactone | ND | ND | ND | 83.9 ± 17.0 | 112 ± 15.3 | 37.1 ± 0.99 | 52.6 ± 14.5 | 50.4 ± 16.3 | ND | 5151 ± 1094 | 14.7 ± 3.71 | 18.5 ± 1.56 |
| 2230 | 4-ethoxycarbonyl- γ-butyrolactone | 128 ± 35.3 | 90.0 ± 8.35 | 101 ± 7.77 | 34.6 ± 3.65 | 112 ± 11.1 | 60.6 ± 3.21 | 66.8 ± 20.4 | 22.4 ± 8.26 | 26.2 ± 7.40 | 57.7 ± 11.5 | ND | 43.4 ± 6.49 |
| 2377 | 4-(1-hydroxyethyl)- γ-butyrolactone | 65.9 ± 14.9 | 33.8 ± 12.9 | 39.1 ± 4.03 | 159 ± 23.2 | 127 ± 16.6 | 67.7 ± 15.3 | 69.7 ± 31.7 | 65.9 ± 28.1 | 19.6 ± 8.22 | ND | ND | 21.0 ± 3.99 |
| | Subtotal | 2657 ± 532 | 2980 ± 233 | 2977 ± 209 | 896 ± 159 | 3562 ± 417 | 291 ± 19.1 | 2555 ± 146 | 827 ± 70.2 | 637 ± 201 | 8339 ± 1127 | 454 ± 48.9 | 1294 ± 27.3 |

Table 5. Cont.

| RI ² | Compound | Concentration (µg/L) | | | | | | | | | | | | |
|-----------------|---------------------------------------|----------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|--|
| | | Y1 | Y2 | ¥3 | Y4 | ¥5 | ¥6 | ¥7 | Y8 | Y9 | Y10 | Y11 | Y12 | |
| | Phenols | | | | | | | | | | | | | |
| 1857 | guaiacol | ND | 14.6 ± 3.81 | ND | ND | ND | ND | ND | 11.9 ± 6.64 | 23.5 ± 13.4 | 17.2 ± 6.01 | ND | ND | |
| 1983 | phenol | ND | ND | 11.2 ± 1.44 | 58.9 ± 8.54 | 21.6 ± 7.85 | 10.5 ± 0.11 | 13.3 ± 0.61 | 15.4 ± 2.79 | 8.51 ± 0.95 | 11.2 ± 2.90 | 17.0 ± 3.06 | 27.4 ± 4.41 | |
| 2016 | 4-ethylguaiacol | ND | ND | ND | 559 ± 69.4 | ND | |
| 2063 | 4-methylphenol | ND | ND | ND | 38.6 ± 7.06 | ND | ND | ND | ND | ND | 12.9 ± 5.0 | ND | ND | |
| 2165 | 4-ethylphenol | ND | ND | ND | 88.6 ± 9.39 | ND | |
| 2192 | 2-methoxy-4-vinylphenol | 120 ± 18.8 | 326 ± 94.8 | 58.1 ± 16.9 | 50.8 ± 19.5 | 21.5 ± 7.57 | 29.4 ± 4.37 | 58.2 ± 19.4 | 369 ± 82.7 | ND | 68.6 ± 18.4 | 52.7 ± 23.1 | 202 ± 18.4 | |
| 2203 | 2-methyl-5- (1-methylethyl) phenol | ND | ND | ND | ND | ND | 11.7 ± 1.70 | ND | 21.9 ± 9.40 | ND | 28.1 ± 11.5 | ND | 35.1 ± 6.53 | |
| 2382 | 4-vinylphenol | 111 ± 42.0 | 73.1 ± 26.6 | 85.4 ± 8.02 | ND | 36.8 ± 2.12 | |
| | Subtotal | 231 ± 46.0 | 414 ± 98.5 | 155 ± 18.8 | 796 ± 73.5 | 43.1 ± 10.9 | 51.6 ± 4.69 | 71.5 ± 19.4 | 418 ± 83.5 | 32.0 ± 13.5 | 138 ± 23.3 | 69.7 ± 23.3 | 301 ± 20.2 | |
| | Terpenoids | | | | | | | | | | | | | |
| 1223 | eucalyptol | 26.1 ± 6.87 | ND | ND | ND | 120 ± 5.21 | ND | ND | 25.2 ± 7.50 | ND | 24.6 ± 11.7 | 38.6 ± 2.63 | ND | |
| 1600 | 4-terpineol | ND | ND | 7.46 ± 0.80 | ND | 52.5 ± 11.0 | 15.5 ± 8.13 | 17.3 ± 9.28 | ND | ND | ND | 24.2 ± 10.7 | 53.6 ± 10.1 | |
| 1692 | α-terpineol | 9.08 ± 1.10 | ND | 8.86 ± 0.82 | 19.2 ± 4.04 | 12.9 ± 1.25 | ND | ND | ND | ND | 52.7 ± 10.3 | ND | 41.9 ± 5.41 | |
| 1701 | borneol | 15.9 ± 2.01 | ND | ND | ND | 133 ± 15.1 | ND | ND | ND | ND | 25.1 ± 4.34 | ND | ND | |
| 1842 | p-cymen-8-ol | ND | ND | ND | ND | 14.6 ± 1.93 | ND | 9.38 ± 2.64 | ND | ND | ND | 18.7 ± 3.41 | 27.6 ± 0.72 | |
| 2073 | p-cymen-7-ol | ND | ND | ND | ND | 12.7 ± 2.0 | ND | |
| | Subtotal | 51.1 ± 7.24 | ND | 16.3 ± 1.15 | 19.2 ± 4.04 | 346 ± 19.6 | 15.5 ± 8.13 | 26.7 ± 9.65 | 25.2 ± 7.50 | ND | 102 ± 16.2 | 81.4 ± 11.5 | 123 ± 11.5 | |
| | Miscellaneous | | | | | | | | | | | | | |
| 969 | 1-(1-ethoxyethoxy) propane | 18.8 ± 10.7 | 47.5 ± 18.6 | 18.3 ± 6.18 | ND | 24.7 ± 8.26 | 11.9 ± 1.93 | 15.0 ± 0.90 | ND | 8.12 ± 1.20 | 34.2 ± 19.2 | 56.4 ± 13.5 | 25.6 ± 4.82 | |
| 974 | 2,4,5-trimethyl- 1,3-dioxolane | 10.7 ± 1.90 | 10.9 ± 2.55 | ND | ND | 5.62 ± 1.22 | ND | 9.66 ± 4.54 | ND | 14.1 ± 5.28 | 12.5 ± 8.12 | 26.3 ± 19.4 | ND | |
| 987 | 1-(1-ethoxyethoxy) butane | 111 ± 66.2 | 387 ± 165 | 117 ± 42.4 | ND | 199 ± 66.3 | 49.9 ± 10.7 | 44.3 ± 14.7 | 15.2 ± 9.02 | 192 ± 118 | 109 ± 26.0 | 80.0 ± 24.1 | 106 ± 21.5 | |
| 1053 | 2-methyl-1,3-dioxane | 15.7 ± 2.83 | 25.7 ± 3.20 | 15.7 ± 5.18 | ND | ND | ND | ND | ND | 9.46 ± 4.96 | 16.5 ± 4.62 | ND | 7.91 ± 2.47 | |
| 1068 | 1,1-diethoxy- 2-methylbutane | ND | 37.3 ± 22.6 | 12.9 ± 3.26 | 20.8 ± 17.8 | 17.0 ± 8.31 | 83.1 ± 6.08 | 21.2 ± 6.64 | 21.8 ± 12.4 | 13.1 ± 6.88 | ND | 12.8 ± 7.53 | 17.7 ± 1.91 | |
| 1069 | 1,1-diethoxy- 3-methylbutane | 21.5 ± 7.31 | 39.8 ± 10.3 | 19.6 ± 3.18 | 49.4 ± 28.4 | 28.7 ± 4.86 | 20.2 ± 2.28 | 51.4 ± 38.1 | 142 ± 88.1 | 53.6 ± 20.5 | 21.8 ± 14.0 | 36.9 ± 16.6 | 54.0 ± 7.11 | |
| 1098 | 1-(1-ethoxyethoxy) pentane | 262 ± 171 | 817 ± 366 | 338 ± 130 | ND | 540 ± 183 | 164 ± 43.7 | 169 ± 91.2 | 41.8 ± 25.1 | 598 ± 367 | 279 ± 64.7 | 248 ± 103 | 268 ± 51.6 | |

| RI ² | Compound | Concentration (µg/L) | | | | | | | | | | | |
|-----------------|------------------------------------|----------------------|-------------------|-----------------|---------------------|-----------------|-----------------|-----------------|-----------------|---------------|-----------------|---------------|-----------------|
| | | ¥1 | Y2 | ¥3 | Y4 | ¥5 | ¥6 | ¥7 | Y8 | Y9 | Y10 | Y11 | Y12 |
| 1225 | 1,1-diethoxyhexane | 18.9 ± 3.33 | 12.1 ± 2.15 | 13.7 ± 1.83 | ND | 31.6 ± 3.99 | ND | 50.5 ± 23.6 | ND | ND | ND | ND | 10.4 ± 0.39 |
| 1494 | 5-hydroxy- 2-methyl-1,3-dioxane | 66.3 ± 14.1 | 195 ± 31.1 | 115 ± 16.4 | ND | 126 ± 18.5 | 27.9 ± 6.90 | 48.3 ± 17.9 | 8.43 ± 0.10 | 21.9 ± 2.76 | 86.8 ± 6.32 | 24.3 ± 4.32 | 25.1 ± 2.78 |
| 1629 | ethyl acetamide | 10.7 ± 1.91 | 11.5 ± 1.70 | 11.7 ± 6.86 | ND | 33.1 ± 13.7 | 7.30 ± 0.53 | 9.27 ± 0.39 | 10.9 ± 3.59 | ND | 21.6 ± 11.2 | ND | ND |
| 1862 | N-(3-methylbutyl) acetamide | 22.9 ± 13.5 | 24.1 ± 9.49 | ND | ND | 16.3 ± 8.18 | 15.3 ± 5.51 | 47.0 ± 42.2 | 38.9 ± 5.69 | 12.8 ± 1.57 | ND | 54.4 ± 30.7 | 57.8 ± 8.15 |
| 1970 | 2-acetylpyrrole | ND | ND | ND | ND | ND | ND | 10.0 ± 2.17 | 11.4 ± 2.00 | ND | 11.3 ± 2.51 | ND | ND |
| | Subtotal | 558 ± 184 | 1607 ± 404 | 661 ± 138 | 70.2 ± 33.5 | 1022 ± 197 | 380 ± 46.3 | 475 ± 113 | 290 ± 93.2 | 923 ± 386 | 593 ± 75.4 | 540 ± 114 | 573 ± 57.3 |
| | Total | 120,390 ± 7658 | 123,505 ± 7301 | 102,204 ± 1808 | 130,811 ± 14,792 | 99,108 ± 5023 | 79,030 ± 5479 | 91,968 ± 3794 | 88,966 ± 2972 | 51,925 ± 2767 | 91,550 ± 4006 | 90,364 ± 5443 | 83,795 ± 2390 |

Table 5. Cont.

¹ Mean value of 3 replications ± SD. ² RI (Retention indices) were determined on DB-wax using C6-C26 as external reference. ND stands for not detected.

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Generally, acetic acid is responsible for the pungent odor in vinegar [18]. Huh et al. [21] reported that high acetic acid content could decrease the taste of a liquor, although the threshold for acetic acids is approximately 280,000 μ g/L in a 10% ethanol system [22]. Among the 12 samples, Y4 had the highest acetic acid content (14,700 μ g/L) followed by Y8 (5038 μ g/L), while Y12 had the lowest acetic acid content (624 μ g/L). Y7 was the only sample contained formic acid (11.3 μ g/L) and 2-butenoic acid (12.4 μ g/L). Fan and Qian [23] suggested that free fatty acids such as hexanoic acid and octanoic acid were produced by bacteria during the fermentation process. Y9 had the lowest content of 2-methyl propanoic acid (105 μ g/L), butanoic acid (20.1 μ g/L), 3-methyl butanoic acid (104 μ g/L), and hexanoic acid (56.7 μ g/L), which seemed to be associated with the degree of fermentation.

Alcohols, the largest group of volatile compounds in alcoholic beverages such as red wine [24,25], are produced during yeast metabolism, and alcohol content varies depending on the yeast starters [26]. Fusel alcohols, such as *n*-propyl alcohol, *iso*-butyl alcohol, and *iso*-amyl alcohol, are produced during the fermentation of amino acid in yeast [12,27]. Among them, isoamyl alcohol has the highest amount of the volatile compounds as reported by Kim et al. [28]. Isoamyl alcohol is known to boost aroma and flavor when it exists in low amounts [27]. Additionally, 2-methyl-1-propanol, which is one of the aliphatic alcohols related to alcohol odor [1], was high in Y1 (10,085 μ g/L), Y2 (12,313 μ g/L), and Y5 (10,222 μ g/L).

Esters are produced during alcoholic fermentation by yeast; they have a fruit-like aroma and thus they positively influence the aroma quality of liquors [29–31]. The large number of volatile ester compounds (n = 32) could be explained by the various supplementary ingredients used to produce the samples. Isoamyl acetate is related to sweet and fruity aromas, and Mamede et al. [29] reported that low concentration of isoamyl acetate results in low consumers' acceptance of sparkling wine samples. Isoamyl acetate content was highest in Y10 and lowest in Y8. Considering that overall acceptance of Y10 (6.41) was much higher than that of Y4 (3.25) as in Table 4, the results of this study confirmed that isoamyl acetate might be one of the key compounds affecting consumer acceptance. Among the samples, Y4 contained high amounts of ethyl lactate (45,468 µg/L), isoamyl lactate (446 µg/L), diethyl succinate (8015 μ g/L), and ethyl hydrogen succinate (6056 μ g/L). Apostolopoulou et al. [32] showed that the ethyl lactate content of samples of bottled Greek distillates (tsipouro) differs from that of homemade samples, suggesting that production methods might influence the amount of ethyl lactate. Argyri et al. [33] also analyzed the volatile compounds of meat samples under different temperatures. As the temperature increased (from 0 °C to 15 °C), the amount of ethyl lactate also increased. These results suggested that the ethyl lactate content of Y4 was affected by the manufacturing environment, such as production methods and temperature.

Ethyl octanoate contents in all of the samples was less than the threshold, which was reported to be 170 μ g/L [34]. Y7 had the highest level of 2-phenylethyl acetate (759 μ g/L), followed by Y6 (600 μ g/L). This might be due to the specific yeast used in Y6 and Y7, considering that this compound is known to be formed during fermentation by yeast [34]. The intensity of 2-phenylethyl acetate in Y7 and Y6 was much higher than threshold (180 μ g/L), which was reported in [34].

A high amount of terpenes is associated with a flower-like odor [35]. Given that eucalyptol, α -terpineol, and 4-terpineol are found in Chrysanthemum morifolium R. [36], the five terpenes (eucalyptol, 4-terpineol, α -terpineol, borneol, and p-cymen-8-ol) found in Y5 might have originated from Chrysanthemum morifolium R., which was that sample's major supplementary ingredient.

3.4. Relationship among Sensory Attributes, Volatile Compounds and Consumers' Acceptance of Yakju Samples by MFA

For the MFA, 30 volatile compounds (acids = 3; alcohol = 8; aldehyde = 1; ester = 8; ketone = 2; lactone = 3; phenol = 1; miscellaneous = 4) were selected from the 120 volatile compounds based on a correlation coefficient of >0.5 with consumers' overall acceptance. A correlation map of descriptive attributes, volatile compounds, and consumer acceptance is in Figure 2a and loading of 12 *yakju* samples in the first two dimensions by MFA is shown in Figure 2b. A total of 59.3% of variance was explained by F1 (47.7%) and F2 (11.7%). Similar to the results in Figure 1, fruit-related sensory

attributes were placed on the negative side of F1, while bitterness, mouthfeel, and alcohol flavor were placed on the positive side of F1 (Figure 2a).

Of the volatile compounds, all esters (n = 8) were in quadrant 2 and 3, along with fruit-related sensory attributes such as omija odor, hawthorn odor, tangerine peel odor/flavor, apple flavor and grape flavor. These sensory attributes and volatile ester compounds were closely related with consumers' overall acceptance. Not only two esters (ethyl propanoate, r = 0.80; ethyl-2-hydroxy-2-methylbutanoate, r = 0.92), but also two lactones (γ -butyrolactone, r = 0.85; 4-ethoxycarbonyl- γ -butyrolactone r = 0.78), two miscellaneous volatile compounds (2-methyl-1,3-dioxane, r = 0.72; 5-hydroxy-2-methyl-1,3-dioxane, r = 0.72), and one phenols (4-vinylphenol, r = 0.64) were highly correlated with consumers' overall acceptance (Figure 2a). The volatile esters such as ethyl propanoate and ethyl 2-methyl propanoate in Y1, Y2, Y3, Y5, Y10, and Y12 are known to be found in strawberry juice [37] and durian [38], and methyl 2-furoate is known to be found in dried omija fruit samples [39]. The results of this study implied that the odor characteristics of *yakju* samples might be affected by the volatiles from the fruit or medicinal herbs used in the *yakju* samples. In addition to these volatile esters, ethyl butanoate, known to have a fruity aroma, with an odor threshold of 20 µg/L [40], was positively associated with the overall acceptance scores of most samples (Y1, Y2, Y3, Y5, Y6, Y7, Y,10, Y11, and Y12). However, those of Y4, Y8, and Y9, which were negatively associated with ethyl butanoate, contained under the threshold of ethyl butanoate (Y4 = not detected; Y8 = $16.2 \mu g/L$; Y9 = $7.0 \mu g/L$).

One of the abundant and important volatile lactones found in wine is γ -butyrolactone [25], which has a fruity aroma [31]. Not only γ -butyrolactone but also 4-ethoxycarbonyl- γ -butyrolactone was positively related with consumers' acceptance. Considering that the content of volatile lactones varies depending on aging time and type of yeast strain in sherry wine [41], the type of yeast strain in the *nuruk* used for the *yakju* samples might have influenced the production of those volatile compounds. In addition, Lee et al. [40] reported that *nuruk* generally contains various kinds of microorganisms compared with to *ipguk* (koji), which only contains *Aspergilus oryzae*, leading to the production of more volatile compounds in *nuruk* than in *ipguk*. Lee et al. [42] reported an absence of 2,3-butanediol in *ipguk* samples, and a relatively low amount of 2,3-butanediol in Y2 (198.9 µg/L) and Y3 (205.5 µg/L) in this study implies that Y2 and Y3 samples might be made of *ipguk*.

Volatile alcohol compounds have a pungent mouthfeel and a pungent and "herbaceous" odor [43]. Described as having a grassy, medicinal, fusel, and spirituous odor, 1-butanol has an odor threshold of 150,000 μ g/L [22,43,44]. Although 1-butanol correlated with consumers' acceptance, the odor of 1-butanol may be imperceptible considering that the 1-butanol contents in the samples ranged from 44.8 μ g/L to 1,159.9 μ g/L, which was considerably lower than the threshold value.

Volatile phenolic compounds are also formed primarily through alcoholic fermentation [45] and are known to be important in the overall aroma of wine [46] and flavors of dark beer [47]. Generally, volatile phenolic compounds such as 4-vinylphenol have a "nutty" odor similar to "almond shell" [45], spicy, and medicinal-like aromas [48]. Butkhup et al. [46] also reported that these compounds had "heavy pharmaceutical" odor. The existence of 4-vinylphenol was only found in Y1 (111 μ g/L), Y2 (73.1 μ g/L), Y3 (85.4 μ g/L), and Y12 (36.8 μ g/L). Although this compound had a positive relationship with young consumers' acceptance (*r* = 0.64), the effect of 4-vinylphenol on young consumers' overall acceptance for *yakju* might be negligible considering its threshold (610 μ g/L) [46].

Some volatile alcohols (3-butanediol, 2,3-butanediol, 1-octanol, and propylene glycol), acetic acid, γ -hexalactone, and 1,1-diethoxy-3-methylbutane were associated with persimmon vinegar odor, roasted grain odor, yeast odor, kudzu flavor, and coating mouthfeel. Y4 and Y8, which had lower consumer acceptance than other samples, were associated with volatile alcohols such as 1,3-butanediol, 2,3-butanediol, and propylene glycol. This result implied that lower consumer acceptance of these samples might be caused by those volatile alcohol compounds and bitter taste, bitter aftertaste, alcohol flavor, and kudzu flavor. Butanediols were produced from carbohydrates during the alcoholic fermentation primarily by *S. cerevisiae*, a major yeast in *yakju* [49–51]. In particular, 2,3-butanediol is the dominant volatile compound in wine, and it has a bitter taste [50]. The highest contents of

these compounds in Y4 and Y8 might be due to yeast, such as *S. cerevisiae*, which is involved in the fermentation of rice or supplementary starch ingredients, such as sweet pumpkin and soybean. This result suggested pungent and sour odors like persimmon vinegar odor, related to volatile acetic acid, and some volatile alcohols such as 1,3-butanediol, 2,3-butanediol, and propylene glycol, might negatively affect young consumers' acceptance.



Variables (axes F1 and F2: 59.33 %)

Figure 2. Correlation map of (**a**) descriptive attributes (magenta: O, odor; T, taste; F, flavor; M, mouthfeel; AT, aftertaste; AM, after-mouthfeel), volatile compounds (green) and consumers' acceptance (red) and (**b**) *yakju* samples in the first two dimension of MFA.

Furthermore, one of the volatile acetals, 1,1-diethoxy-3-methylbutane, was also associated with Y4 (49.4 µg/L) and Y8 (142 µg/L), which had lower consumers' acceptance than other samples. This compound was found in liquor samples such as Chinese liquor [23] and Italian grape marc spirit [52]. Volatile acetals are known to be produced by aldehydes in the presence of excessive content of ethanol [23,53]. This suggested that 1,1-diethoxy-3-methylbutane might be produced by relatively high contents of ethanol or aldehyde-related compounds, as in Y4 and Y8. Significant differences in fruit-related attributes were found among the samples, even though the intensities of fruit-related attributes and volatile compounds were closely related with consumer acceptance. The fruit-related sensory attributes and volatile esters were placed in quadrant 2 and 3, while roasted grain odor, yeast odor, root-related flavor (kudzu flavor), and all mouthfeel attributes were placed in quadrant 1 and 4. The result of this study confirmed the results by Jung et al. [1], who reported that rice wine samples were distinguished by their volatile alcohol and volatile ester. Furthermore, descriptors such as yeast odor and cereal flavor were located opposite to the fruit and sweet aroma in the PCA map.

In addition to volatile compounds that might originate from supplementary ingredients, major ingredients such as different sources of starch might affect the flavors and therefore consumers' acceptance. While most of the samples were produced mainly from rice, Y1, Y2, Y3, and Y5 used corn starch in addition to rice. Kim et al. [4] conducted a sensory evaluation of traditional liquor samples made from various starch sources. They showed that the acceptance of liquors made with corn starch and brown rice was higher than that of liquors made with glutinous rice or non-glutinous rice or potato starch. Liquors made with corn starch or brown rice tended to contain a relatively lower amount of acetic acid and a higher amount of fructose than the liquors made with non-glutinous rice, suggesting that the type of starch could affect the sensory and chemical properties of liquors. Apart from starch source, rice protein in *yakju* samples increases the pH of *yakju* samples, causing the formation of off-flavor [54]. Moreover, León-Rodríguez et al. [55] suggested that the presence of minor compounds (e.g., some volatile ethyl esters, terpenes, acids, and furans) at low concentrations could cause a synergic effect with other volatile compounds, leading to the production different odor characteristics. Therefore, further investigation on interactions among volatile compounds is needed to understand the odor characteristics and volatile compounds that affect consumers' overall acceptance.

4. Conclusions

Twelve *yakju* samples were characterized, based on sensory descriptors by PCA. The result of the PCA showed that *yakju* samples were characterized mainly by their supplementary raw materials, which affect their overall odor and flavor. As shown by the result of the MFA correlation map, the *yakju* samples tended to be classified by sensory attributes and volatile compounds. The results of this study showed that acceptance of *yakju* samples was largely influenced not only by their alcohol content but also by their supplementary ingredients, especially those related to fruit-related aroma. On the contrary, volatile acetic acid, and some volatile alcohols (1,3-butanediol, 2,3-butanediol, and propylene glycol), and 1,1-diethoxy-3-methylbutane were related with persimmon vinegar odor, roasted grain odor, and yeast odor, and negatively correlated with young consumers' acceptance of *yakju*. This is a first report on how the major sensory attributes and volatile compounds of Korean rice liquor (*yakju*) affect overall acceptance by young consumers, even though the number of consumers who participated in this study (n = 80) was not sufficient. Overall, the results of this study suggested that acceptance of yakju products could be improved by controlling some volatile esters that resulted from supplementary ingredients, or specific volatile alcohols and acids produced during fermentation. Considering that the aroma and flavor of *yakju* could vary depending on the starch source, supplementary ingredients, yeast strains, and fermentation process, further investigation is needed on the specific yeast strains and fermentation conditions that affect the formation of volatile compounds and consumers' acceptance of *yakju*. Along with this, further research on interactions among volatile compounds is also needed to understand the odor characteristics that affect consumers' overall acceptance.

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References

- Jung, H.Y.; Lee, S.J.; Lim, J.H.; Kim, B.K.; Park, K.J. Chemical and sensory profiles of *makgeolli*, Korean commercial rice wine, from descriptive, chemical, and volatile compound analyses. *Food Chem.* 2014, 152, 624–632. [CrossRef]
- 2. Kim, H.R.; Kim, J.H.; Bae, D.H.; Ahn, B.H. Characterization of *yakju* brewed from glutinous rice and wild-type yeast strains isolated from *nuruks*. *J. Microbiol. Biotechnol.* **2010**, *20*, 1702–1710. [PubMed]
- 3. Yang, S.; Lee, J.; Kwak, J.; Kim, K.; Seo, M.; Lee, Y. Fungi associated with the traditional starter cultures used for rice wine in Korea. *J. Korean Soc. Appl. Biol. Chem.* **2011**, *54*, 933–943. [CrossRef]
- 4. Kim, H.R.; Jo, S.J.; Lee, S.J.; Ahn, B.H. Physicochemical and sensory characterization of a Korean traditional rice wine prepared from different ingredients. *Korean J. Food Sci. Technol.* **2008**, *40*, 551–557.
- 5. Choi, J.S.; Yeo, S.H.; Choi, H.S.; Jeong, S.T. The effect of rice *nuruk* prepared from rice with different degrees of milling on quality changes in *yakju*. *Korean J. Food Sci. Technol.* **2017**, *49*, 265–273.
- 6. Kwak, E.J.; Lee, J.Y.; Choi, I.S. Physicochemical properties and antioxidant activities of Korean traditional alcoholic beverage, *yakju*, enriched with mulberry. *J. Food Sci.* **2012**, *77*, 752–758. [CrossRef] [PubMed]
- Jin, T.Y.; Lee, W.G.; Lee, I.S.; Wang, M.H. Changes of physicochemical, sensory and antioxidant activity characteristics in rice wine, *yakju* added with different ratios of *Codonopsis anceolate*. *Korean J. Food Sci. Technol.* 2008, 40, 201–206.
- 8. Kim, J.H.; Lee, D.H.; Lee, S.H.; Choi, S.Y.; Lee, J.S. Effect of *Ganoderma lucidum* on the quality and functionality of Korean traditional rice wine, *yakju. J. Biosci. Bioeng.* **2004**, *97*, 24–28. [CrossRef]
- 9. Lee, J.O.; Kim, C.J. The influence of adding buckwheat sprouts on the fermentation characteristics of *yakju*. *Korean J. Food Cult*. **2011**, *26*, 72–79.
- 10. Lee, S.J.; Kwon, Y.H.; Kim, H.R.; Ahn, B.H. Chemical and sensory characterization of Korean commercial rice wines (*yakju*). *Food Sci. Biotechnol.* **2007**, *16*, 374–380.
- 11. Lee, S.J. Effect of brand recognition and familiarity on consumer preferences for commercial rice wines (*yakju*). *Korean J. Food Sci. Technol.* **2011**, *43*, 23–29. [CrossRef]
- Kang, H.R.; Hwang, H.J.; Lee, J.E.; Kim, H.R. Quantitative analysis of volatile flavor components in Korean alcoholic beverage and Japanese sake using SPME-GC/MS. *Food Sci. Biotechnol.* 2016, 25, 979–985. [CrossRef] [PubMed]
- Kang, B.S.; Lee, J.E.; Park, H.J. Electronic tongue-based discrimination of Korean rice wines (*makgeolli*) including prediction of sensory evaluation and instrumental measurements. *Food Chem.* 2014, 151, 317–323. [CrossRef] [PubMed]
- Xiao, Z.; Yu, D.; Niu, Y.; Ma, N.; Zhu, J. Characterization of different aroma-types of Chinese liquors based on their aroma profile by gas chromatography-mass spectrometry and sensory evaluation. *Flavour Fragr. J.* 2016, *31*, 217–227. [CrossRef]
- Xiao, Z.; Liu, S.; Gu, Y.; Xu, N.; Shang, Y.; Zhu, J. Discrimination of cheery wines based on their sensory properties and aromatic fingerprinting using HS-SPME-GC-MS and multivariate analysis. *J. Food Sci.* 2014, 79, 284–294. [CrossRef] [PubMed]
- 16. Kim, E.H.; Ahn, B.H.; Lee, M.A. Analysis of consumer consumption status and demand of rice-wine. *J. Korean Soc. Food Sci. Nutr.* **2013**, *42*, 478–486. [CrossRef]
- 17. Chang, Y.J.; Kim, E.M.; Choi, Y.S.; Jeon, K.H.; Kim, Y.B. Development process for decreasing bitterness of doraji (*Platycodon grandiflorum*). *J. Korean Soc. Food Sci. Nutr.* **2015**, *44*, 1550–1557. [CrossRef]
- Su, M.S.; Chien, P.J. Aroma impact components of rabbiteye blueberry (*Vaccinium ashei*) vinegars. *Food Chem.* 2010, 119, 923–928. [CrossRef]

- Lee, S.J.; Lee, K.G. Understanding consumer preferences for rice wines using sensory data. J. Sci. Food Agric. 2008, 88, 690–698. [CrossRef]
- 20. Kwak, H.S.; Ahn, B.H.; Kim, H.R.; Lee, S.Y. Identification of sensory attributes that drive the likeability of Korean rice wines by American panelists. *J. Food Sci.* **2015**, *80*, 161–170. [CrossRef]
- 21. Huh, C.K.; Lee, J.W.; Kim, Y.D. Fermentation and quality characteristics of *yakju* according to different rice varieties. *Korean J. Food Preserv.* **2012**, *19*, 925–932. [CrossRef]
- 22. Siebert, T.E.; Smyth, H.E.; Capone, D.L.; Neuwöhner, C.; Pardon, K.H.; Skouroumounis, G.K.; Herderich, M.J.; Sefton, M.A.; Pollnitz, A.P. Stable isotope dilution analysis of wine fermentation products by HS-SPME-GC-MS. *Anal. Bioanal. Chem.* **2005**, *381*, 937–947. [CrossRef] [PubMed]
- 23. Fan, W.; Qian, M.C. Identification of aroma compounds in Chinese 'Yanghe Daqu' liquor by normal phase chromatography fractionation followed by gas chromatography/olfactometry. *Flavour Fragr. J.* **2006**, *21*, 333–342. [CrossRef]
- 24. Jiang, B.; Xi, Z.; Luo, M.; Zhan, Z. Comparison on aroma compounds in Cabernet Sauvignon and Merlot wines from four wine grape-growing regions in China. *Food Res. Int.* **2013**, *51*, 482–489. [CrossRef]
- 25. Vilanova, M.; Campo, E.; Escudero, A.; Graña, M.; Masa, A.; Cacho, J. Volatile composition and sensory properties of *Vitis vinifera* red cultivars from north west Spain: Correlation between sensory and instrumental analysis. *Anal. Chim. Acta* **2012**, *720*, 104–111. [CrossRef]
- 26. Yang, Y.; Xia, Y.; Wang, G.; Yu, J.; Ai, L. Effect of mixed yeast starter on volatile flavor compounds in Chinese rice wine during different brewing stages. *LWT Food Sci. Technol.* **2017**, *78*, 373–381. [CrossRef]
- 27. In, H.Y.; Lee, T.S.; Lee, D.S.; Noh, B.S. Volatile components and fusel oils of sojues and mashes brewed by Korean traditional method. *Korean J. Food Sci. Technol.* **1995**, 27, 235–240.
- 28. Kim, H.R.; Kwon, Y.H.; Jo, S.J.; Kim, J.H.; Ahn, B.H. Characterization and volatile flavor components in glutinous rice wines prepared with different yeasts of *nuruks*. *Korean J. Food Sci. Technol.* **2009**, *41*, 296–301.
- 29. Mamede, M.E.O.; Cardello, H.M.A.B.; Pastore, G.M. Evaluation of an aroma similar to that of sparkling wine: Sensory and gas chromatography analyses of fermented grape musts. *Food Chem.* **2005**, *89*, 63–68. [CrossRef]
- Niu, Y.; Yao, Z.; Xiao, Z.; Zhu, G.; Zhu, J.; Chen, J. Sensory evaluation of the synergism among ester odorants in light aroma type liquor by odor threshold, aroma intensity and flash GC electronic nose. *Food Res. Int.* 2018, 113, 102–114. [CrossRef]
- 31. Rocha, S.M.; Rodrigues, F.; Coutinho, P.; Delgadillo, I.; Coimbra, M.A. Volatile composition of Baga red wine: Assessment of the identification of the would-be impact odourants. *Anal. Chim. Acta* 2004, 513, 257–262. [CrossRef]
- 32. Apostolopoulou, A.A.; Flouros, A.I.; Demertzis, P.G.; Akrida-Demertzi, K. Differences in concentration of principal volatile constituents in traditional Greek distillates. *Food Control* **2005**, *16*, 157–164. [CrossRef]
- Argyri, A.A.; Mallouchos, A.; Panagou, E.Z.; Nychas, G.J.E. The dynamics of the HS/SPME-GC/MS as a tool to assess the spoilage of minced beef stored under different packaging and temperature conditions. *Int. J. Food Microbiol.* 2015, 193, 51–58. [CrossRef] [PubMed]
- Pino, J.A.; Queris, O. Analysis of volatile compounds of mango wine. *Food Chem.* 2011, 125, 1141–1146. [CrossRef]
- 35. Falqué, E.; Fernández, E.; Dubourdieu, D. Differentiation of white wines by their aromatic index. *Talanta* **2001**, *54*, 271–281. [CrossRef]
- Chang, K.M.; Kim, G.H. Volatile aroma constituents of *gukhwa* (*Chrysanthemum morifolium* R.). *Food Sci. Biotechnol.* 2013, 22, 659–663. [CrossRef]
- 37. Schieberle, P.; Hofmann, T. Evaluation of the character impact odorants in fresh strawberry juice by quantitative measurements and sensory studies on model mixtures. *J. Agric. Food Chem.* **1997**, 45, 227–232. [CrossRef]
- Voon, Y.Y.; Hamid, N.S.A.; Rusul, G.; Osman, A.; Quek, S.Y. Characterisation of Malaysian durian (Durio zibethinus Murr.) cultivars: Relationship of physicochemical and flavour properties with sensory properties. *Food Chem.* 2007, 103, 1217–1227. [CrossRef]
- Lee, H.J.; Cho, I.H.; Lee, K.E.; Kim, Y.S. The compositions of volatiles and aroma-active compounds in dried Omija fruits (Schisandra chinensis Baillon) according to the cultivation areas. *J. Agric. Food Chem.* 2011, 59, 8338–8346. [CrossRef]

- Escudero, A.; Gogorza, B.; Melus, M.A.; Ortin, N.; Cacho, J.; Ferreira, V. Characterization of the aroma of a wine from Maccabeo. Key role played by compounds with low odor activity value. *J. Agric. Food Chem.* 2004, 52, 3516–3524. [CrossRef]
- 41. Cortes, M.B.; Moreno, J.; Zea, L.; Moyano, L.; Medina, M. Changes in aroma compounds of Sherry wines during their biological aging carried out by Saccharomyces cerevisiae Races bayanus and capensis. *J. Agric. Food Chem.* **1998**, *46*, 2389–2394. [CrossRef]
- 42. Lee, S.M.; Shin, K.J.; Lee, S.J. Exploring Nuruk aroma; identification of volatile compounds in commercial fermentation starters. *Food Sci. Biotechnol.* **2016**, *25*, 393–399. [CrossRef] [PubMed]
- 43. Genovese, A.; Gambuti, A.; Piombino, P.; Moio, L. Sensory properties and aroma compounds of sweet fiano wine. *Food Chem.* **2007**, *103*, 1228–1236. [CrossRef]
- Peinado, R.A.; Mauricio, J.C.; Moreno, J. Aromatic series in sherry wines with gluconic acid subjected to different biological aging conditions by Saccharomyces cerevisiae var. capensis. *Food Chem.* 2006, 94, 232–239. [CrossRef]
- 45. Noguerol-Pato, R.; González-Álvarez, M.; González-Barreiro, C.; Cancho-Grande, B.; Simal-Gándara, J. Evolution of the aromatic profile in Garnacha Tintorera grapes during raisining and comparison with that of the naturally sweet wine obtained. *Food Chem.* 2013, 139, 1052–1061. [CrossRef]
- Butkhup, L.; Jeenphakdee, M.; Jorjong, S.; Samappito, S.; Samappito, W.; Chowtivannakul, S. HS-SPME-GC-MS analysis of volatile aromatic compounds in alcohol related beverages made with mulberry fruits. *Food Sci. Biotechnol.* 2011, 20, 1021–1032. [CrossRef]
- 47. Vanbeneden, N.; Gils, F.; Delvaux, F.; Delvaux, F.R. Formation of 4-vinyl and 4-ethyl derivatives from hydroxycinnamic acids: Occurrence of volatile phenolic flavour compounds in beer and distribution of Pad1-activity among brewing yeast. *Food Chem.* **2008**, *107*, 221–230. [CrossRef]
- 48. Domínguez, C.; Guillén, D.A.; Barroso, C.G. Determination of volatile phenols in fino sherry wines. *Anal. Chim. Acta* **2002**, *458*, 95–102. [CrossRef]
- Ehsani, M.; Fernández, M.R.; Biosca, J.A.; Julien, A.; Dequin, S. Engineering of 2,3-butanediol dehydrogenase to reduce acetoin formation by glycerol-overproducing, low-alcohol *Saccharomyces cerevisiae*. *Appl. Environ*. *Microbiol.* 2009, 75, 3196–3205. [CrossRef]
- 50. Romano, P.; Brandolini, V.; Ansaloni, C.; Menziani, E. The production of 2,3-butanediol as a differentiating character in wine yeast. *World J. Microbiol. Biotechnol.* **1998**, *14*, 649–653. [CrossRef]
- 51. Seo, M.Y.; Lee, J.K.; Ahn, B.H.; Cha, S.K. The changes of microflora during the fermentation of *Takju* and *Yakju*. *Korean J. Food Sci. Technol.* **2005**, *37*, 61–66.
- Masino, F.; Montevecchi, G.; Riponi, C.; Antonelli, A. Composition of some commercial grappas (grape marc spirit): The anomalous presence of 1,1-diethoxy-3-methylbutane: A case study. *Eur. Food Res. Technol.* 2009, 228, 565–569. [CrossRef]
- Ledauphin, J.; Guichard, H.; Sanint-clair, J.F.; Picoche, B.; Barillier, D. Chemical and sensorial aroma characterization of freshly distilled calvados. 2. Identification of volatile compounds and key odorants. *J. Agric. Food Chem.* 2003, *51*, 433–442. [CrossRef] [PubMed]
- 54. Kang, J.E.; Kim, J.W.; Choi, H.S.; Kim, C.W. Effect of the addition of protein and lipid on the quality characteristics of *Yakju. Korean J. Food Preserv.* **2015**, *22*, 361–368. [CrossRef]
- 55. Antonio De León-Rodríguez, A.D.; González-Hernández, L.; Rosa, A.P.B.D.L.; Escalante-Minakata, P.; López, M.G. Characterization of volatile compounds of mezcal, an ethnic alcoholic beverage obtained from *Agave salmiana. J. Agric. Food Chem.* 2006, 54, 1337–1341. [CrossRef]



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