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RESEARCH ARTICLE

Analysis of Aroma Compounds of Commercial Cider Vinegars with Different Acidities using SPME/GC-MS, Electronic Nose, and Sensory Evaluation

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Abstract Aroma compounds of commercial cider vinegars according to their acidity (low, moderate, double-strength, and triple-strength) were comparatively investigated. SPME/GC-MS analysis revealed 8 types of aroma compounds commonly existing in the vinegar including acetic acid. A cider vinegar with moderate acidity showed the most volatile profiles. Electronic nose analysis suggested that the peak at 0.40 s was a component related to total acidity of vinegar. Principal component analysis enabled e-nose to discriminate easily by presenting the separated position depending on total acidity. Significant differences with the acidity were observed in the pungent odor intensity and odor preference. According to the results, the moderate acidity vinegar was revealed to retain most volatile profiles and sensory odor. Compared to sensory evaluation, the analysis using SPME/GC-MS and e-nose provided more distinct patterns on their volatile compounds depending on their acidity, which might be a potential tool in determining the volatile profiles of cider vinegars.

Keywords: cider vinegar, volatile, SPME/GC-MS, electronic nose, sensory evaluation

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Introduction

Vinegar is one of the most widely used seasonings in the world because of its availability in several different varieties in every country (1). Vinegar is a product resulting from the alcoholic and subsequent acetic fermentation of any cereals or fruits rich in carbohydrates and contains a specific amount of acetic acid. In today's market, there is increasing demand for vinegar as a health food product with multiple uses (2). On the other hand, the varieties vary according to region. Among the fruit vinegars, cider vinegar is the best-selling one in Korea despite wine vinegar being the most widely-marketed products.

The acidity strength of vinegars is measured by the concentration of acetic acid (%) present in the product. All vinegars sold in the United States at the retail level should have at least 4% acidity, as mandated by the U.S. Food & Drug Administration (FDA) (3). Cider and wine vinegars are typically slightly more acidic with approximately 5-6% acidity. Vinegars sold in the Republic of Korea are classified into four groups according to their acidity and purpose; low acidity (4-5%), moderate acidity (6-7%), double-strength acidity (13-14%), and triple-strength acidity (18-19%) according to fermentation progress (4). The final quality and sensory properties of vinegars are determined by the chemical complexity conditioned by the raw material employed and the fermentation process used. The aroma or flavor is certainly one of the most important determinants of food quality and acceptance. The characteristic aroma of vinegar is the result of high quantities of distinguishing volatile compounds including acetic acid (5). Some authors have highlighted the importance of the production process in the final aroma of vinegars and in their organoleptic qualities (6-8). Because the volatile profiles in vinegar represent a fingerprint of the sample, it is reasonable that a classification approach based on an analysis of the volatile components would be an efficient tool for evaluating the vinegar quality. Indeed, a number of studies have described the effective analysis of volatile compounds for the characterization and differentiation of different vinegars (5,6,9-18).

The strategies to mimic the human sense of olfaction and thus measure odors can be divided into three main types, sensory analysis, chemical analysis, and electronic nose (15). All three methods have their own advantages and disadvantages. Sensory analysis applies the principles of experimental design and statistical analysis to the use of human senses for evaluating consumer products. Currently, sensory analysis still provides the key technique in the food industry, but an evaluation by humans has inherent limitations, such as fatigue (19). The most popular chemical analysis method for vinegars is gas chromatography-mass spectrometry (GC-MS) (9-12). Although GC-MS can separate numerous chemical components from a complex mixture of flavor volatiles, there are still a number of shortcomings, such as a time consuming analysis and the poor correlation with the sensory data (20). An electronic nose is a device intended to detect odors without the pretreatment, which was used for some industry-based applications (21). The odor assessment is normally performed by human sensory analysis or GC-MS, and the electronic nose results can be correlated with those obtained from these techniques.

As part of basic research for developing high-strength acidity vinegar using fruits, the aroma compounds and volatiles patterns of four commercial cider vinegar products were comparatively analyzed using solid-phase micro-extraction/gas chromatography-mass spectrometry (SPME/GC-MS), electronic nose, and sensory evaluation to assess their quality properties according to different acidities.

Materials and Methods

Materials Commercial cider vinegar products were purchased from a local market and classified into low, moderate, double-strength, and triple-strength according to their acidity (n=3). The main ingredients, manufacturing plant, and expiration date of all the samples were adjusted as similar conditions except the acidity. Concentrated apple juice, water, and alcohol were the main ingredient for vinegar production, and only the triple-strength acidity vinegar contained citric acid (Table 1). Total acidity was measured as acetic acid (%) by titration with sodium hydroxide (0.1 N) to an endpoint pH of 8.35.

Analysis of the aroma compounds by SPME/GC-MS To extract the aroma compounds, 75 μm carboxen/polydimethylsiloxane SPME fibers (Supelco, Bellefonte, PA, USA) was conditioned at 250°C for 5 min prior to use. Each sample (2 mL) was placed in a 20-mL glass vial fitted with a PTFE/silicone septum (Supelco) and a screw cap. The sample was stirred at 70°C for 20 min, and the compounds were extracted from the headspace to the fiber for 30 min (13). The SPME fibers were injected into the SPME injector of the GC-MS system, and the injection was performed for 2 min at 250°C. The aroma compounds were analyzed using a GC system (Agilent 6890, Palo Alto, CA, USA) coupled with a mass selective detector (Agilent 5973N, Palo Alto, CA, USA). A fused silica capillary column (DB-WAX; 60 m×0.25 mm i.d., 0.25 μm film thickness, J&W, Folsom, CA, USA) and helium gas (1 mL/min) were used for detection. The oven temperature was programmed as follows: kept at 32°C for 7 min, ramped at 2°C/min to 70°C, increased at 9°C/min to 200°C and maintained for 2 min. The MS interface temperature was set to 250°C, the MS quadrupole temperature was 150°C, and the transfer line was 280°C. Peak identification was made by matching mass spectra of unknown with those of Wiley 6.0 MS Library (13) with a minimum of 90% of correspondence and aroma properties were supplied from authentic volatile compounds (acetic acid, 1-hexanol, 2-hexenal, ethyl acetate) of Sigma-Aldrich Chemical Co. (Milwaukee, WI, USA).

Electronic nose analysis The volatile compounds of the vinegars were analyzed using a zNose (Electronic Sensor Technology, Newbury Park, CA, USA) equipped with a surface acoustic wave (SAW) sensor. The samples (2 mL) were placed into a 40-mL vial (Supelco) fitted with PTFE/silicone septa (Supelco) and placed at room temperature for 24 h to obtain the headspace equilibrium. The headspace was transferred to a DB-5 capillary column (0.3 mm×1 m, Supelco, Bellafonte, PA, USA) using helium gas (purity

Table 1. Major ingredients and acidity of commercial cider vinegars with different acidity levels

Acidity level	Major ingredients	Total acidity (%)
Low	Concentrated apple juice 13.92% (apple 100%), water, alcohol, nutrient	4.59±0.02[1)]
Moderate	Concentrated apple juice 5.03% (apple 100%), water, alcohol, nutrient, apple essence	6.61±0.04
Double-strength	Concentrated apple juice 5.03% (apple 100%), water, alcohol, nutrient, apple essence	13.41±0.16
Triple-strength	Concentrated apple juice 5.03% (apple 100%), water, alcohol, nutrient, citric acid, apple essence	18.43±0.20

[1)]Mean±SD (n=3).

99.99%). The analysis was performed under the following conditions: SAW sensor temperature of 60ºC, column of 60ºC, valve of 120ºC, inlet of 150ºC, and trap of 220ºC. All samples were analyzed six times, and VaporPrint^{TM} (Microsense 4.88; Electronic Sensor Technology) was used for data analysis. The main peaks were analyzed by principal component analysis (PCA) using SAS program version 8.1 (SAS Institute, Cary, NC, USA) (22).

Sensory evaluation The sensory evaluation was performed using a 9-pt hedonic scale for both the intensity and preference of cider vinegars. With the exception of color, ten-fold diluted samples were given to the panel for testing on 5 sensory properties. Thirty panelists having wide experience in sensory evaluation scored each attribute on a scale of 1-9 about the intensity and preference, in which 1 was the weakest or most unacceptable and 9 was the strongest or most acceptable (23). The significance of sensory scores obtained was analyzed using a Duncan's multiple range test (22).

Results and Discussion

Comparison of aroma characteristics by SPME/GC-MS The major aroma components of vinegars can be classified into the acids, alcohols, aldehydes, and esters, which are transferred from the raw materials or produced during their fermentation. The headspaces of four cider vinegars were analyzed by SPME/GC-MS and the aroma compounds were grouped according to the appropriate class. Table 2 lists the relative peak area and the sensory odor description. Sixteen aroma compounds were identified in the moderate acidity vinegar, whereas 9 to 12 kinds were detected in others. Eight types of compounds were commonly observed throughout all the samples; ethyl acetate, isoamyl acetate, hexyl acetate, acetic acid, ethyl alcohol, 1-hexanol, furfural, and benzaldehyde. All vinegars were characterized by the highest acetic acid content, which was generated by the acetic fermentation of ethanol and is known for the pungent odor of vinegars (6,16). The triple-strength acidity vinegar showed the highest proportion (84.30%) of acetic

Table 2. Comparison of the aroma compounds analyzed by SPME/GC-MS in commercial cider vinegars with different acidity levels

(unit: peak area, %)

RI[1)]	Component	Acidity level				Odor description (ODE)
		Low	Moderate	Double	Triple	
	Acids					
1450	Acetic acid	49.92±5.54[2)]	58.98±4.53	78.02±0.33	84.30±6.15	Pungent
	Alcohols					
971	Ethyl alcohol	3.30±0.29	1.81±0.17	1.06±0.10	0.42±0.04	Pleasant
1150	2-Aminopropanol	ND	0.10±0.02	0.08±0.01	ND	
1426	1-Hexanol	0.34±0.02	2.29±0.71	0.77±0.06	0.44±0.18	Herbaceous
1432	Cyclohexanol	ND	0.19±0.02	ND	ND	Camphor
	Aldehydes					
1202	Hexanal	ND	1.61±0.20	0.33±0.06	0.22±0.11	
1360	2-Hexenal	ND	2.59±0.38	0.96±0.03	0.51±0.21	Sweet almond, Fruity
1476	Furfural	2.28±0.60	0.08±0.02	0.05±0.02	0.08±0.02	Caramel
1544	Benzaldehyde	0.21±0.02	0.05±0.01	0.04±0.02	0.04±0.01	Bitter almond
	Esters					
929	Ethyl acetate	30.86±2.14	21.02±0.98	12.74±1.73	7.73±2.41	Fruity
982	Ethyl propionate	ND	0.07±0.01	ND	ND	Fruity
1104	Ethyl butyrate	ND	0.47±0.01	0.07±0.02	ND	Pineapple
1162	n-Butyl acetate	2.35±0.12	0.06±0.02	ND[2)]	ND	
1182	Isobutyl acetate	0.67±0.08	ND	ND	ND	
1235	Isoamyl acetate	0.60±0.08	1.13±0.25	0.36±0.07	0.34±0.18	Pear-like
1402	Hexyl acetate	2.27±0.73	4.33±1.57	2.60±0.74	2.82±1.62	Fruity, Green, Fresh
	Others					
1348	2-Pentene	ND	0.16±0.01	ND	ND	

1)Retention indices were determined using C8-C40 as external standards; Mean values
2)Mean±SD (n=2); ND: Not detected
3)Odor description reported in Refs (11,22,23,25).

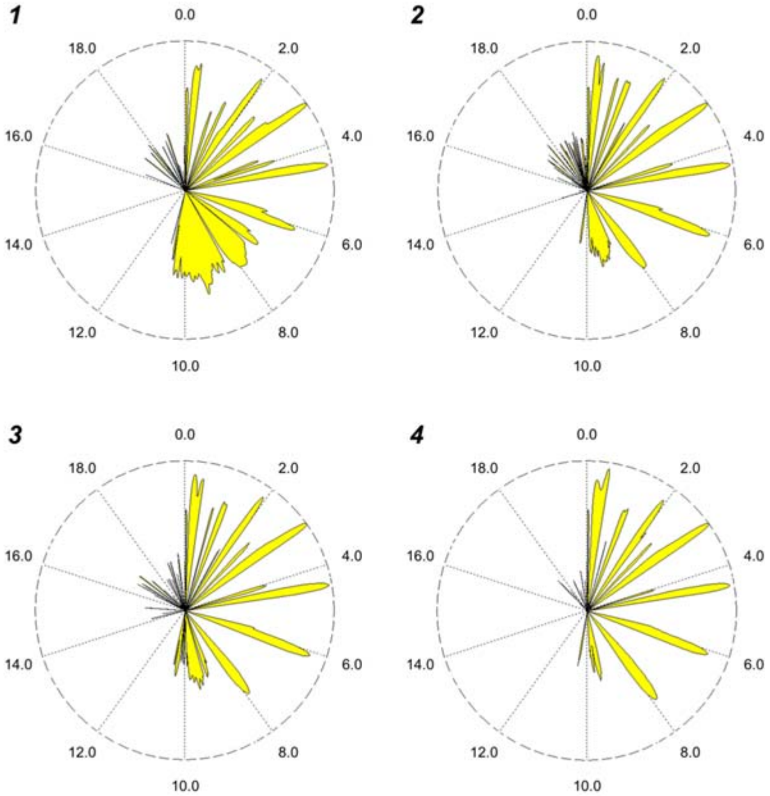

Fig. 1. Comparison of Vaporprint™ on commercial cider vinegars with different acidity levels. Numbers mean retention time (sec). 1: Low, 2: Moderate, 3: Double-strength, 4: Triple-strength

acid which is the main responsibility for the sensory characteristics of the apple vinegar. The most abundant esters in cider vinegars were ethyl acetate, followed by hexyl acetate and isoamyl acetate, which impart a fruity aroma (24). Ethyl acetate produced by the esterification of ethanol and acetic acid (23) was in the range of 7.73-21.02%, which showed the highest proportion in the moderate acidity vinegar. Hexyl acetate gives a fruit type flavor, such as banana peel, apples, and pears (25). 2-Hexenal imparts the flavor of green leafy or fruits and vegetables (25). Isoamyl acetate, ethyl alcohol, benzaldehyde, ethyl propionate, and ethyl butyrate impart pear-like, pleasant, almond oil, fruity, and pineapple flavors, respectively (25). Furfural is a product as a result of the browning reaction of pentose sugar, which is sometimes used to evaluate the sensory properties due to its sweet, almond and caramel aroma (11). Different trends in aroma properties were observed in fruit vinegar products according to their acidity, which were affected particularly by the fruit substrate used and fermentation conditions. In terms of commercial cider vinegars, the products with moderate acidity showed the most volatile profiles and the percentage of acetic acid increased with their acidity. In production of vinegar, industrial process with aerated-stirred fermentation may induce the volatile loss. The high-strength acidity vinegar requires more time and aeration for fermentation; therefore the volatiles will be reduced than others. Lee *et al*. reported that the static culture retains more volatile compounds than agitated culture (26).

Principal component analysis of volatile compounds by electronic nose Well-trained sensory panelists can identify a significant difference in the strength or blend in samples, but the human nose has the limitation of a fatigue symptom (19). An electronic nose system is a device with a gas sensor system to detect odors. Many studies have also reported the application of the electronic nose to an analysis of the volatile components of different foods (15,16,25-27). Fig. 1 shows the volatile compound pattern of cider vinegars according to their acidity. The VaporPrint™ is a 2-dimensional signal detected by the electronic nose sensor that provides the odor concentration and characteristic shapes (28). This polar format begins at the 0.0 position and follows the dial with retention times increasing in a clockwise direction. The identity and quantity of a substance can be calculated using the software incorporated in the device by measuring the time and amount required for each chemical to reach the sensor (29). Cider vinegars generate unique and easily recognizable images, showing variations in the retention time and amounts of each compound (Fig. 1). The analysis of volatile components by the electronic nose revealed main 16 peaks over an approximately 10 s

Table 3. Comparison of the peak identified by the electronic nose in commercial cider vinegars with different acidity levels
(unit: KCt/sec)

Peak time (sec)	Acidity level			
	Low	Moderate	Double	Triple
0.06	215±16[1)]	ND[2)]	ND	ND
0.30	923±156	3,294±175	3,762±125	2,829±247
0.40	377±201	582±217	2,388±326	4,809±954
0.86	ND	529±28	225±52	ND
1.12	ND	336±11	488±277	114±59
1.20	ND	152±24	77±39	196±53
1.92	3,724±56	4,220±152	3,319±705	2,911±899
2.06	ND	ND	142±52	ND
2.40	360±88	113±22	124±43	69±39
3.02	12,916±478	12,897±374	13,569±592	11,943±934
4.00	135±18	80±22	44±32	ND
4.44	10,032±400	10,721±205	10,545±471	9,198±935
6.08	2,668±683	4,135±228	3,611±593	3,144±689
7.82	196±76	ND	ND	1,687±218
7.90	ND	577±69	1,200±225	ND
9.28	47±52	68±53	51±34	24±25

[1)]Mean±SD (n=6).
[2)]Not detected.

period (Table 3); the rest peaks with very low sensitivity (less than 20 Kct/sec) are exempted from the list. All vinegars showed peaks with high intensity at 0.30, 1.92, 3.02, 4.44, and 6.08 s, whereas high-strength acidity vinegars showed an increasing peak at 0.40 s. Considering that the peak at 0.40 s increased with their acidity significantly (R^2=0.9636), it was estimated that the peak is a component related to total acidity of vinegars. Lee *et al.* (26) reported that the peak within 2 s is acetic acid produced during acetic fermentation, which supports these results. The peak at 0.06 s is observed only in low acidity vinegar, whereas the peaks at 0.86 and 7.90 s are observed only in moderate and double acidity vinegar. The peak at 4.00 s decreased with total acidity (R^2=0.9266). Noh *et al.* (30) reported that the E-noses do not provide information on the quantity of the individual volatile compounds but rather a global and qualitative analysis of the volatile chemical profile, which is the difference with GC-MS system.

PCA is a well-known statistical method to visualize the resemblance and difference by reducing the dimensionality of numerical datasets, and is used widely for analyzing gas-sensing data (28). PCA of the electronic nose data was carried out to obtain an over view of each chemical compound (Fig. 2). This figure shows that the samples are distributed into three groups. The cumulative proportions of eigenvalues on the 1st and 2nd principal components (PCs) through the correlation matrix were 47.32 and 32.17%, respectively. This makes 79.49% of the cumulative

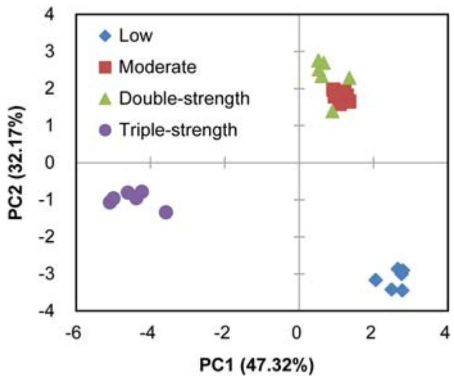

Fig. 2. PCA plot of the main peaks identified by the electronic nose in commercial cider vinegars with different acidity levels.

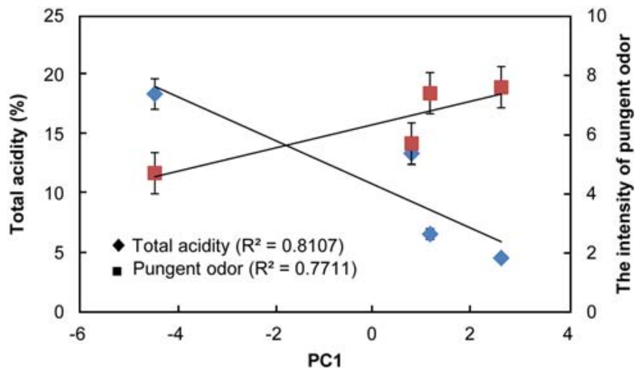

Fig. 3. Relationship between quality attribute (total acidity or pungent odor) and PC1 obtained from the electronic nose.

proportion for the two PCs. The moderate and double-strength acidity vinegars exhibited few overlapped points positioned at the top right side of the plot. On the other hand, these were well separated with low acidity (bottom right side) and triple-strength acidity (bottom left side), providing easy discrimination according to their acidity. The relationship between total acidity and PC1 of the obtained data from the electronic nose showed a good correlation (R^2=0.8107) as shown in Fig. 3. The electronic nose is a very promising tool for characterizing commercial vinegars by analyzing the overall pattern, even though it cannot provide specific information on the components.

Comparison of the sensory properties Table 4 shows the result of the sensory evaluation for the commercial cider vinegars. The sensory score on the intensity of the pungent odor and sour taste increased with the strength of the acidity (p<0.01). No significant difference in pungent odor was observed between the low and moderate acidity vinegars, but significant difference were observed in the double and triple strength acidity vinegars (p<0.05). The relationship between pungent odor and PC1 from the electronic nose also showed a good correlation (R^2=0.7711) (Fig. 3). The sour taste showed a significant difference

Table 4. Comparison of the sensory properties in commercial cider vinegars with different acidity levels

Sensory property		Acidity level				Significance
		Low	Moderate	Double	Triple	
Intensity	Pungent odor	4.70±0.90[1)b]	5.70±1.27[b]	7.40±1.02[a]	7.60±1.11[a]	<0.0001
	Sour taste	4.60±1.11[c]	5.80±1.08[b]	6.40±1.36[ab]	7.10±1.30[a]	0.0009
Preference	Color	4.80±1.47[b]	7.00±1.34[a]	6.50±1.43[a]	7.10±1.30[a]	0.0039
	Odor	6.20±1.66[ab]	7.10±1.37[a]	5.90±1.14[ab]	4.70±1.42[b]	0.0093
	Taste	5.60±1.02[a]	6.10±0.94[a]	6.80±1.33[a]	6.20±1.47[a]	0.2331
	Overall	5.70±1.10[a]	6.60±1.11[a]	6.70±1.35[a]	6.40±1.36[a]	0.3236

[1)]Mean±SD (n=10) using a 9-point scale evaluation.
[a-c)]Values within a row followed by different superscript are significantly different.

among all the samples ($p<0.05$), which shows that the palate is more sensitive than the sense of smell in case of vinegars. In the preference, the sensory color of low acidity vinegar showed a significantly lower score than others ($p<0.01$), which suggests that the brown color of low acidity vinegar in the previous finding (31) had an adverse effect on the preference. In sensory odor, the highest score was observed in moderate acidity vinegar. Lee *et al.* (26) reported that the high pungency of acetic acid limits the sensory results of vinegars, showing a similar tendency to the present results. Although sensory analysis is a widely-used method for obtaining reliable qualitative information, it provides little quantitative data (15). Therefore, it is recommended that objective analysis, such as GC-MS and electronic nose, be used to obtain more accurate information on the chemical species. Desrochers *et al*. (19) have also pointed out sensory analysis and chemical analyses are both powerful tools that can be used in a quality assurance program and become even more powerful when used together.

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