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Accelerated aging of grape pomace vinegar by using additives combined with physical methods

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Abstract

(a) High-quality vinegar is traditionally produced by aging in barrels, but this process is time-consuming and costly. The aim of the present work was to evaluate the effectiveness of three novel aging techniques, including additives combined with ultrasound, with microwave, or with heating. (b) The contents of esters, amino acid nitrogen, and non-volatile acids were evaluated. The effects of aging techniques on the color and aroma components of grape pomace vinegar were also studied. (c) It was found the ester contents significantly increased and more aroma components accumulated under three aging techniques, especially the additives combined with ultrasound. The total ester content of grape pomace vinegar treated by the additives combined with ultrasound increased by 42.2% compared with natural aging at 16°C for 180 days. (d) Therefore, the additives combined with ultrasound can be applied as a reliable technique to accelerate the aging process of grape pomace vinegar.

Practical Application

Grape pomace is the main by-product of the wine and grape juice industry, and contains rich nutrient and bioactive components. Conventional aging methods are time-consuming and unable to maintain the original color and acidity of vinegar. The aging technique of additives (glucose, ethanol, and calcium chloride) combined with ultrasound can accelerate the aging of grape pomace vinegar and enhance the properties. This combined technique theoretically underlies the practical application of grape resources and supports the development of grape pomace vinegar with health care function.

1 | INTRODUCTION

The generation of grape by-products results in wastes, which has been a problem in wine brewing process (Bustamante et al., 2008). Grape pomace, the major by-product of the wine and grape juice industry, consists of grape skins, seeds, and minor fruit stalks (Chikwanha, Raffrenato, Muchenje, Musarurwa, & Mapiye, 2018; Lima et al., 2015). Due to residual bioactive compounds, grape pomace is highly potential for recycling (Amendola, De Faveri, & Spigno, 2010; Arvanitoyannis, Ladas, & Mavromatis, 2006). Pomace contains 285–550 mg/kg polyphenols (Yilmaz & Toledo, 2006), 700 g/kg dietary fibers (Sáyagoayerdi, Brenes, & Goñi, 2009), 6.81–12.39 mg/kg proanthocyanidins (Zhang, Li, & Li, 2015), and 34–102 μg/kg resveratrol (Singh, Siddiqui, El-Abd, Mukhtar, & Ahmad, 2016), which can be used in health-care vinegar production. Grape pomace has been applied to wine production (Pedroza, Carmona, Alonso, Salinas, & Zalacain, 2013; Pedroza, Carmona, Salinas, & Zalacain, 2011) and is a source of natural pigments (Boo et al., 2012), natural antioxidants, or antibacterial additives for food applications (Han et al., 2011; Mildner-Szkudlarz, Zawirska-Wojtasiak, & Gośliński, 2010; Sagdic et al., 2011; Yu & Ahmedna, 2013). Nevertheless, there are few studies on the production of grape pomace vinegar.

Fresh vinegar often has a stiff taste, a pungent smell, and weak aroma. The aging technique, which contributes to the formation of esters, alcohols, and acids, is used as a post-treatment to improve the flavor, prolong fermentation time and promote ripening (De Villiers, Alberts, Tredoux, & Nieuwoudt, 2012). The oak barrel (vinegar liquid storage container) aging technology has been accepted for centuries to urge the aging of vinegar (Morales, Benitez, & Troncoso, 2004; Waterhouse & Towey, 1994). However, this traditional aging technology is time-consuming, unable to maintain the original color and can easily produce unpleasant aromas. In order to accelerate the aging process, researchers focus on the physical aging technology that can improve the wine quality in shorter aging time. At present, the physical aging technology mainly includes ultrasonic wave aging (Chang, 2005; Masuzawa, Ohdaira, & Ide, 2000), gamma rays (Chang, 2003), electric field treatment (Zeng, Yu, Zhang, & Chen, 2008), and microwave aging (Liu et al., 2015). The application of the physical aging technology has been proved to be effective in accelerating wine aging process, but its application in vinegar aging is innovative and attractive. From a chemical perspective, compounds carrying hydroxyl groups can react with organic acids to form esters (Cirlini, Caligiani, & Palla, 2009). However, the application of additives in vinegar aging has not been studied worldwide or reported in international journals.

This work is aimed to characterize the changes of grape pomace vinegar under different aging techniques. We investigated the influence of three additives (glucose, ethanol, and calcium chloride) combined with physical methods (ultrasound, microwave, and heating) on grape pomace vinegars. The contents of esters, amino acid nitrogen, non-volatile acid, and volatile substances as well as the color of vinegars after treatment were analyzed. The results are expected to be valuable for finding an efficient aging method of grape pomace vinegar and to provide ideas for further industrial production.

2 | MATERIALS AND METHODS

2.1 | Materials

The grape pomace used in the experiments was produced from red grapes and supplied by Food Processing Laboratory of Changchun Normal University (Jilin, China). Acetic acid bacteria powder was Shanghai brewing 1.01 (Shanghai Institute of Brewing Science, Shanghai, China). Glucose, ethanol, and calcium chloride were purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). All other chemicals used in this study were of analytical grade and bought from Beijing Chemical Works (Beijing, China).

2.2 | Methods

2.2.1 | Preparation of grape pomace samples

Grape pomace was mixed with deionized water at a ratio of 3:5 (w/v) for 30 s and treated by an ultrasonic-microwave combined reaction system (Nanjing Shunliu Instruments Co. Ltd., Jiangsu, China). The mixture was pretreated with ultrasound at the power of 125 W for 20 min, and with microwave at the power of 281 W for 2 min. Then the samples were cooled to room temperature and stored until subsequent analysis.

2.2.2 | Bacterial activation

The culture medium containing 1% (w/w) yeast extract and 1% (w/w) glucose (pH 4.5) was put into 250 ml Erlenmeyer flasks (each with 100 ml), which were sealed with gauze (four layers) and cotton plug. Then the flasks were sterilized at 121°C for 30 min, and cooled to room temperature. After that, the sterilized medium was adjusted to 4% alcohol and added with 0.5% of acetic acid bacterial powder. The flasks were sealed with four layers of sterile gauze, and the bacteria were mixed with the culture medium. Then the bacteria were activated in a constant-temperature incubator (Shanghai Yiheng Technology Co. Ltd., Shanghai, China) at 30°C and a rotation speed of 140 r/min for 24–36 hr.

2.2.3 | Acetic acid fermentation

The treated grape pomace homogenate was adjusted to 7.5% alcohol (v/w) and then added with 12.3% (v/w) activated acetic acid bacteria. The grape pomace homogenate was added into a 250 ml Erlenmeyer flask which was sealed with sterile gauzes per 100 ml, and then fermented at 140 r/min and 30°C for 9 days. The grape pomace vinegar was centrifuged at 4000 r/min for 10 min, and the acidity of the clarified mixture was 5.52 g/100 ml.

2.2.4 | Aging conditions of grape pomace vinegar

The grape pomace vinegar was treated by additives combined with ultrasound (ACU), additives combined with microwave (ACM) or additives combined with heating (ACH). Then esters were determined to investigate the accelerating aging of grape pomace vinegar. The specific test program was shown in Table 1.

2.2.5 | Analysis of aging effect and quality

Vinegar samples were put into 10 ml centrifuge tubes covered with sealing films. The tubes were labeled as sample 1 (ACU), sample 2 (ACM), sample 3 (ACH), sample 4 (storage at 16°C for 180 days), sample 5 (storage at 4°C for 180 days), and sample 6 (fresh vinegar). The contents of esters, amino acid nitrogen, non-volatile acid, and volatile substances as well as the color of vinegars after treated by each aging technique were measured.

2.2.6 | Determination of esters

The ester contents were determined according to GB/T 19777-2013. Specifically, the vinegar sample (20 ml) was added into a 250 ml

TABLE 1 Test design table

Sample	Experimental factors		
	Ethanol (v/v) (%)	Glucose (w/v) (%)	Calcium chloride (w/v) (%)
US-et	0.1, 0.2, 0.3, 0.4, 0.5	–	–
US-gl	–	1, 2, 3, 4, 5	–
US-eg	0.1, 0.2, 0.3, 0.4, 0.5	4	–
US-egc	0.3	4	0.1, 0.2, 0.3, 0.4, 0.5
MW-et	0.1, 0.2, 0.3, 0.4, 0.5	–	–
MW-gl	–	2, 3, 4, 5, 6	–
MW-eg	0.1, 0.2, 0.3, 0.4, 0.5	5	–
MW-egc	0.4	5	0.1, 0.2, 0.3, 0.4, 0.5
HE-et	0.1, 0.2, 0.3, 0.4, 0.5	–	–
HE-gl	–	1, 2, 3, 4, 5	–
HE-eg	0.1, 0.2, 0.3, 0.4, 0.5	4	–
HE-egc	0.4	4	0.1, 0.2, 0.3, 0.4, 0.5

Notes: (a) US processing parameters: ultrasonic power of 200 W, time of 30 min, temperature of 30°C. (b) MW processing parameters: microwave power of 231 W, time of 1 min. (c) HE processing parameters: heating time of 2 days, temperature of 80°C.
Abbreviations: eg, ethanol and glucose; egc, ethanol, glucose, and calcium chloride; et, ethanol; gl, glucose.

Erlenmeyer flask and adjusted to pH 8.20 with a 0.01 mol/L sodium hydroxide standard solution. The dilution was mixed with 20.00 ml of a 0.01 mol/L sodium hydroxide standard solution to saponify the esters. Then the mixture was transferred into a round-bottomed flask, which was put on a condenser and boiled for about 30 min. After that, the mixture was cooled to room temperature and transferred into a 250 ml beaker, and the flask was washed with 20.00 ml of distilled water. The wash solution was also transferred into the beaker. Finally, the mixture was titrated to pH 8.20 with a 0.01 mol/L sulfuric acid standard solution. The ester content of each sample, X_1, was calculated as follows:

$$X_1 = \frac{(c_1V_1 - c_2V_2) \times 0.088}{\frac{10}{100} \times 20.0} \times 100 \quad (1)$$

where c_1 was the sodium hydroxide concentration (mol/L), V_1 was the volume of the sodium hydroxide solution (ml), c_2 was the sulfuric acid concentration (mol/L), and V_2 was the volume of the sulfuric acid solution (ml).

2.2.7 | Determination of amino acid nitrogen

The amino acid nitrogen contents were determined according to GB 5009. 235-2016. Typically, a diluted sample (20.00 ml) and 60.00 ml of distilled water were put into a 250 ml beaker and adjusted to pH 8.20 with the 0.01 mol/L sodium hydroxide solution. Then 10.00 ml of a formaldehyde solution was added into the beaker. The resulting mixture was adjusted to pH 9.20 with the 0.01 mol/L sodium hydroxide solution. At the same time, 80.00 ml of distilled water was added into another beaker and adjusted to pH 8.20 with the 0.01 mol/L sodium hydroxide solution, followed by the addition of 10.00 ml of the formaldehyde solution. The mixture was adjusted to pH 9.20 with the 0.01 mol/L sodium hydroxide solution and used as a blank control. The amino acid nitrogen content of each sample, X_2, was computed as follows:

$$X_2 = \frac{(V - V_0) \times c \times 0.014}{\frac{10}{100} \times 20.0} \times 100 \quad (2)$$

where V and V_0 were the volume of the sodium hydroxide solution consumed by the sample and by the blank control respectively (ml), and c was the sodium hydroxide concentration (mol/L).

2.2.8 | Determination of non-volatile acids

The amino acid nitrogen contents were determined according to GB 18187-2000. At each time, a diluted sample (10.00 ml) was added into a single-boiling distillation tube, distilled and transferred into a 250 ml beaker when the volume of the distilled liquid reached 180 ml. Then the solution was replenished to 120 ml with distilled water, and was titrated to pH 8.20 with the 0.01 mol/L sodium hydroxide solution. The non-volatile acid content of each sample, X_3, was computed as:

$$X_3 = \frac{(V - V_0) \times c \times 0.090}{2} \times 100 \quad (3)$$

where V and V_0 were the volume of the sodium hydroxide solution consumed by the sample and by the blank control respectively (ml), and c was the sodium hydroxide concentration (mol/L).

2.2.9 | Determination of chromaticity

The color of samples was measured by a WSF spectrophotometer (Shanghai Yidian Physical Optics Instrument Co. Ltd., Shanghai, China). D65 standard illuminant was determined as spectral condition. L^*, a^*, b^* were three components of the Lab color space (Hyman, Gaus, & Foolad, 2004). The total chromatic aberration was calculated by the CIE1976Lab chromatic aberration formula as follows:

$$\Delta E^*_{ab} = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (4)$$

where ΔL^*, Δa^*, and Δb^* were the difference of L^*, a^*, and b^* between samples.

2.2.10 | Electronic nose(e-nose) determination method

E-nose analysis of volatile substances was carried out according to the method of Xu et al. (2019) and Martin, Mallikarjunan, and

TABLE 2 Sensor array of electronic noses

Sensor serial number	Sensor	Types of sensitive aroma
1	SS_1	Aromatic compounds
2	SS_2	Nitrogen compounds
3	SS_3	Sulfides
4	SS_4	Organic acid esters, Terpene
5	SS_5	Biosynthetic terpenoids and esters
6	SS_6	Lenthionine
7	SS_7	Aliphatic oxygen derivatives
8	SS_8	Ammonia
9	SS_9	Hydrogen
10	SS_{10}	Hydrocarbon
11	SS_{11}	Volatile organic compounds
12	SS_{12}	Sulfide (quantitative)
13	SS_{13}	Ethylene (quantitative)
14	SS_{14}	Cooking volatile gases

Zoecklein (2008) with certain modifications. The iNose (Shanghai Ruifen Co., Ltd., Shanghai, China) equipped with 14 metal oxide semiconductor sensors with different properties, and the general description of 14 sensors was shown in Table 2. Grape pomace vinegar (10 ml) was placed into an e-nose sample vial at room temperature for 30 min to allow for headspace enrichment before analysis. The parameters of the e-nose were set as follows: sample preparation time was 10 s, and flow rate was 1 L/min. Then the measurement phase lasted for 120 s, which was long enough for the sensors to reach stable signal values. After each experiment, cleaning gas was pumped into the sample gas path for 300 s, which was long enough to normalize sensor signals. E-nose data obtained were statistically analyzed by principal components analysis (PCA) and linear discriminant analysis (LDA).

2.2.11 | Statistical analysis

All experiments were performed in triplicate and the data were reported as mean value ± *SD*. Statistical significance was declared at $p < .05$ tested by ANOVA. All e-nose data obtained were statistically analyzed by PCA and LDA using SPSS Software (Version 21.0, Chicago, IL). The data of PCA and LDA were analyzed using Origin 8.1 software.

3 | RESULTS AND DISCUSSION

3.1 | Effect of additives used in grape pomace vinegar aging process

Figure 1 showed the changes of ester contents in vinegars treated by ACU, ACM, and ACH under different parameters of additives. Total ester contents in vinegars treated by ACU and ACH maximized when the ethanol content was 0.2%, which was different from that of 0.4% under ACM (Figure 1a). Total ester contents maximized, when glucose content was 4% or 5% (Figure 1b). Cirlini et al. (2009) and Morales, Tesfaye, García-Parrilla, Casas, and Troncoso (2002) also reported that compounds carrying hydroxyl groups can promote the increase in the ester content. Moreover, the optimal glucose to ethanol ratios under ACU, ACM, and ACH were 4:0.3, 5:0.4, and 4:0.4, respectively, and the total ester contents maximized to 6.97, 6.94, and 6.89 g/L, respectively (Figure 1c). The ester content maximized when the calcium chloride content was 0.3% or 0.4% under ACU, ACM, and ACH, respectively (Figure 1d).

The trends of ester contents under different parameters of additives were similar to each other, which basically increased first and then declined (Figure 1). As far as additives were concerned, the effect of calcium chloride on the aging process was significant. The ester content can exceed 7.2 g/L. As a kind of Lewis acid, calcium chloride can accelerate the ester formation (Lei et al., 2016; Liu, Lotero, & James, 2006). The aging effect of the combination of glucose and ethanol was better than that of the single use of ethanol or glucose. The reason was that the polarity of hydroxyl group in combined additives was stronger than that of ethanol or glucose, which can substitute the hydroxyl group in the acid to form esters more easily (Cocchi, Ferrari, Manzini, Marchetti, & Sighinolfi, 2007; Palacios, Valcárcel, Caro, & Pérez, 2002).

3.2 | Effects of aging methods on esters, amino acid nitrogen, and non-volatile acid contents

Table 3 listed the contents of esters, amino acid nitrogen, and non-volatile acid under different aging techniques. As for esters and amino acid nitrogen, the contents reached 7.24 g/L and 0.32 g/100 ml respectively under ACU, while the non-volatile acid contents reached 1.34 g/100 ml under ACM or ACH. Compared with the natural aging at 16°C for 180 days, the total ester content under ACU increased by 42.2%. The effect of ultrasound was related to acoustic cavitation, which consisted of formation, growth, and implosive collapse of bubbles (Saterlay & Compton, 2000; Soria & Villamiel, 2010). The acoustic cavitation can induce certain chemical reactions and accelerate reaction rates (Parrilla, Heredia, & Troncoso, 1999), such as oxidation and esterification. The acceleration by microwave was mainly due to the high-frequency polarity change of polar molecules, which accelerated the molecule diffusion and promoted aging reaction. The thermal effect of microwave or heating also promoted the positive movement of Maillard reaction in the aging process.

3.3 | Effects of aging methods on colors

Tables 4 and 5 showed the effects of different aging techniques on the colors of grape pomace vinegar. The L^* and a^* of sample 1 to sample 5 presented lower values compared with sample 6 (Table 4), indicating the aged vinegar was darker than fresh vinegar. The main influence

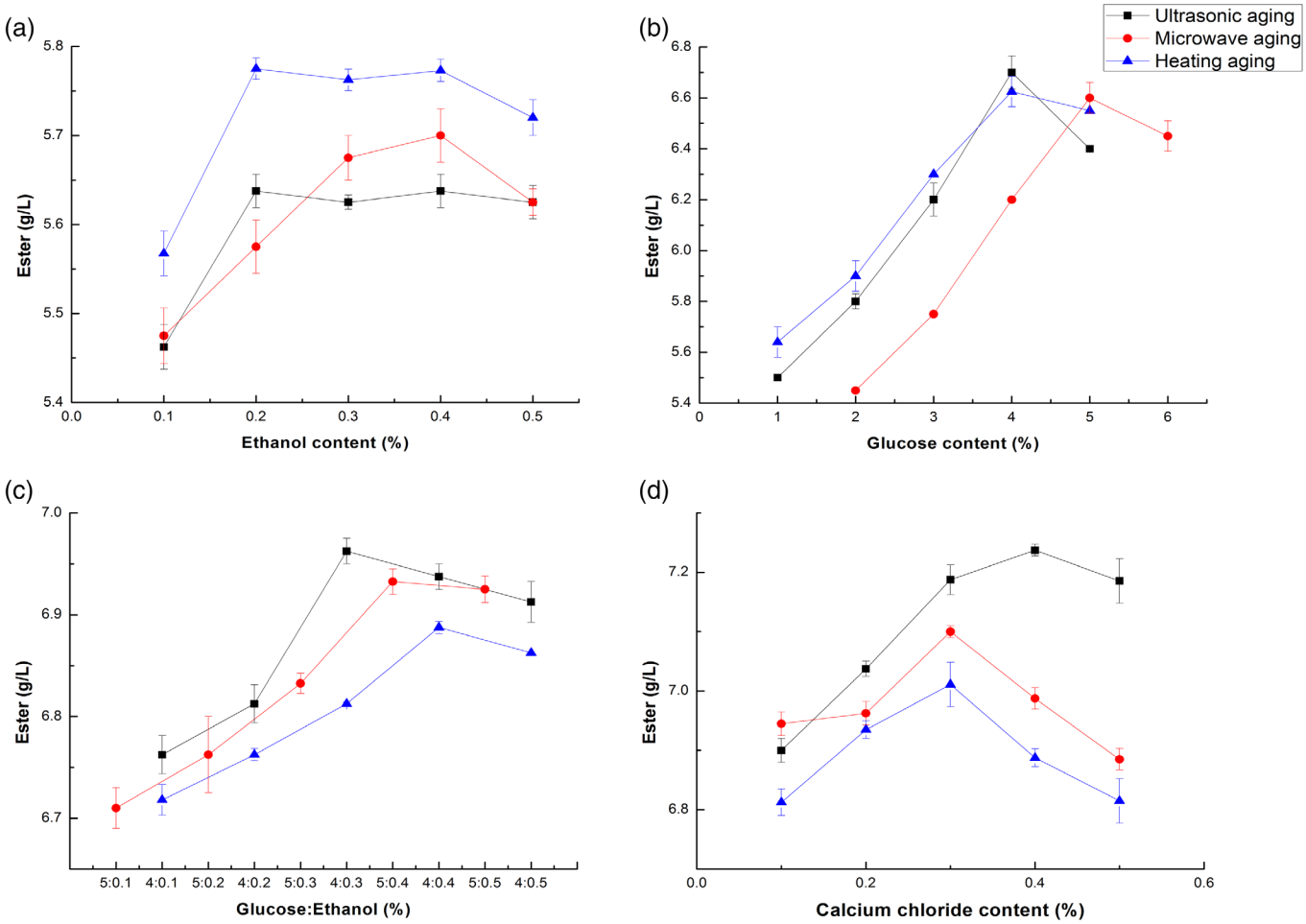

FIGURE 1 The effect of additives on different aging process

TABLE 3 Effects of aging methods on quality of grape pomace vinegar

Sample	Esters (g/L)	Non-volatile acid (g/100 ml)	Amino nitrogen (g/100 ml)
1 (ACU)	7.24 ± 0.11^{a}	1.11 ± 0.14^{a}	0.32 ± 0.04^{a}
2 (ACM)	7.11 ± 0.21^{b}	1.34 ± 0.11^{b}	0.25 ± 0.01^{b}
3 (ACH)	7.01 ± 0.14^{b}	1.34 ± 0.07^{b}	0.14 ± 0.03^{c}
4 (16°C 180d)	5.09 ± 0.13^{c}	1.27 ± 0.12^{ab}	0.30 ± 0.03^{ab}
5 (4°C 180d)	4.76 ± 0.17^{d}	1.24 ± 0.09^{ab}	0.30 ± 0.04^{ab}

Notes: Means and *SD* were determined for triplicate. Superscripts (a–d) indicate significant differences in different treatments ($p < .05$).

TABLE 4 Effects of aging methods on color of grape pomace vinegar

Sample	L^*	a^*	b^*
1 (ACU)	54.29 ± 0.07^{e}	21.81 ± 0.01^{a}	3.75 ± 0.02^{c}
2 (ACM)	56.83 ± 0.02^{c}	19.25 ± 0.02^{d}	3.02 ± 0.03^{e}
3 (ACH)	45.95 ± 0.03^{f}	13.27 ± 0.05^{f}	8.07 ± 0.03^{a}
4 (16°C 180d)	56.65 ± 0.03^{d}	17.18 ± 0.01^{e}	5.75 ± 0.04^{b}
5 (4°C 180d)	58.84 ± 0.04^{b}	20.02 ± 0.03^{c}	3.43 ± 0.03^{d}
6 (fresh vinegar)	60.65 ± 0.02^{a}	21.73 ± 0.02^{b}	2.21 ± 0.01^{f}

Notes: Means and *SD* were determined for triplicate. Superscripts (a–f) indicate significant differences in different treatments ($p < .05$).

factors on the color of grape pomace vinegar were the degradation of anthocyanin and the formation of Maillard reaction products (Xu, Tao, & Ao, 2007; Yıkmış, 2019). As shown in Table 5, the values of total chromatic aberration ΔE^*_{ab} between samples 2 and 3 and between samples 1 and 3 were over 12, indicating that the color had changed obviously. The ΔE^*_{ab} between samples 4 and 5 was more than 3, which indicated discernible color differences. The results imply that storage at 4°C can protect the color of grape pomace vinegar, while storage at 16°C resulted in serious browning. It was because the raw materials contained protein from grape seed and residual sugar. They were more likely to promote Maillard reaction to form melanin, which led to browning of vinegar at higher temperature. Generally, the temperature rise under ACM, ACH, or storage at 16°C promoted the browning process, which influenced the characteristics of vinegar, especially color. In terms of browning and aging time, ACU is more suitable to be applied in the aging of grape pomace vinegar.

TABLE 5 Analysis of chromatic aberration of grape pomace vinegar after aging

ΔE^*_{ab}	1 (ACU)	2 (ACM)	3 (ACH)	4 (16°C 180d)	5 (4°C 180d)	6 (fresh vinegar)
1 (ACU)	–	3.68 ± 0.03	12.69 ± 0.09	5.56 ± 0.05	4.89 ± 0.08	6.54 ± 0.08
2 (ACM)	3.68 ± 0.03	–	13.40 ± 0.05	3.43 ± 0.04	2.19 ± 0.08	4.63 ± 0.06
3 (ACH)	12.69 ± 0.09	13.40 ± 0.03	–	11.63 ± 0.06	15.27 ± 0.08	17.94 ± 0.06
4 (16°C 180d)	5.57 ± 0.05	3.43 ± 0.03	11.63 ± 0.06	–	4.27 ± 0.02	7.02 ± 0.02
5 (4°C 180d)	4.90 ± 0.08	2.19 ± 0.08	15.27 ± 0.08	4.27 ± 0.02	–	2.77 ± 0.05
6 (fresh vinegar)	6.54 ± 0.08	4.63 ± 0.05	17.94 ± 0.06	7.02 ± 0.02	2.77 ± 0.05	–

Note: Means and *SD* were determined for triplicate.

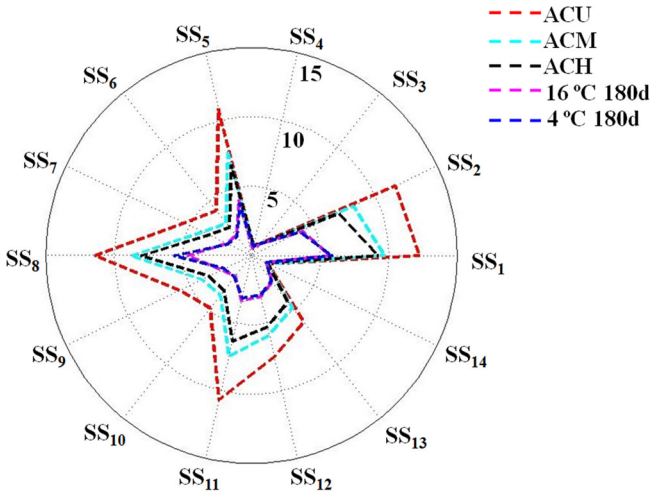

FIGURE 2 Analysis of volatile substances by electronic nose

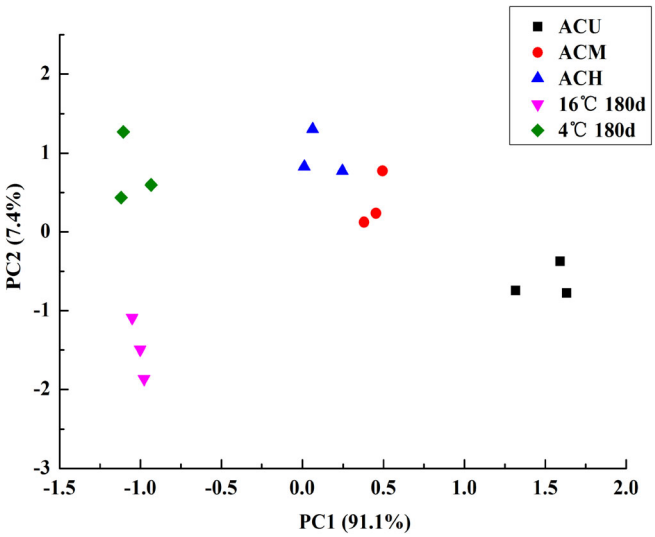

FIGURE 3 Principal component analysis (PCA) of volatile substances

3.4 | Effects of aging methods on volatile substances

3.4.1 | Electronic nose analysis

E-nose response values of volatile substances in grape pomace vinegar were different to some extent (Figure 2). Response values of samples stored at 16°C were nearly the same as those stored at 4°C, indicating temperature was not a key influence factor. The response values of volatile substances in vinegar were similar between ACM and ACH, and were both significantly smaller than those under ACU. The sensor with an obvious difference in response values between ACM and ACH was SS_2. The response values represented the relative content of nitrogen compounds in the grape pomace vinegar, which was related to the more intense Maillard reaction under ACH. During the vinegar aging process, the protein and sugar in grape pomace vinegar will undergo Maillard reaction (Wu et al., 2019). With the Maillard reaction, the protein in vinegar was consumed continuously to form melanin. Heating provided a higher temperature for Maillard reaction, which accounted for the decrease of the nitrogen compounds content (Wang et al., 2017). The results of e-nose showed more aromatic compounds, biosynthetic terpenoids, esters, and volatile organic compounds accumulated during aging, especially in vinegars under ACU. The larger response values of sensors SS_1, SS_5, and SS_{11} were related to the synthesis of flavoring substances. Wang et al. (2017) also reported that the contents of aroma compounds, esters, and volatile substances in vinegar have increased after the ultrasound treatment.

3.4.2 | Principal component analysis

PCA was carried out with samples treated by the five aging techniques (Figure 3). From the covariance matrix, PCA extracted two components, which jointly accounted for 98.5% of the whole system variance. The larger the contribution rates of PC1 and PC2 are, the better the main component can reflect the original information. The score plot in Figure 3 showed that PC1 explained most of the variance (91.1%) and allowed the differentiation between two kinds of aging methods: vinegars aged under storage and vinegars aged by additives combined with physical methods. Sample 4 and sample 5 placed in the negative side of PC1, while sample 1 and sample 2 placed in the positive side. The position of sample 3 was close to the zero point. Moreover, sample 2, sample 3, and sample 5 tended to accumulate in the upper area of the score plot, while sample 1 and sample 4 seemed

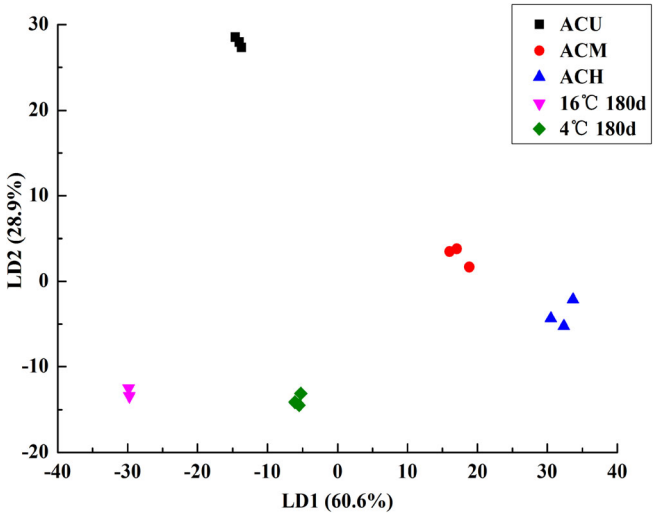

FIGURE 4 Linear discriminant analysis (LDA) of volatile substances

to gather in the lower side. It can be seen that there was no overlap observed in the volatile components of grape pomace vinegar under different aging treatments, which indicates that the five aging techniques can be distinguished by PCA.

3.4.3 | Linear discriminant analysis

Linear discriminant analysis (LDA) is a common classification procedure: the method maximizes the variance between categories and minimizes the variance within categories. Figure 4 showed LDA on volatile substances of samples treated by the five aging techniques. The contribution rates of LD1 and LD2 were 60.6% and 28.9%, respectively, and the total contribution rate was 89.5%. The greater the degree of dispersion between data acquisition points, the better differentiation of the groups. There was no obvious overlap of volatile substances data collection points among different aging techniques. According to the discreteness of the response values, LDA could be used to distinguish the five aging techniques. It could be seen that the position of ACU was in the upper area of the score plot, which was significantly different from other aging techniques.

4 | CONCLUSIONS

The aging technique of additives (glucose, ethanol, and calcium chloride) combined with ultrasound is suitable for the aging of grape pomace vinegar. It can artificially accelerate the aging process and enhance the quality of vinegar. The optimum treatment for vinegar aging was determined to be ultrasonic power 200 W, time 30 min, temperature 30°C, and glucose addition 4% (w/v), ethanol addition 0.3% (v/v), calcium chloride addition 0.4% (w/v). At the optimum condition, the total ester content reached 7.24 g/L. Compared with natural aging at 16°C for 180 days, it increased by 42.2%. In addition, the vinegar colors changed slightly under ACU compared with natural aging. E-nose analysis showed that more aroma components accumulated under ACU. The quality changes of grape pomace vinegar during the aging process can be caused by the increase of certain chemical reaction rates under ACU, such as the esterification reactions, anthocyanin degradation, or the Maillard reactions. Hence, the three additives combined with ultrasound not only significantly shortens the aging time, but also endows grape pomace vinegar with attractive color and aroma.

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CONFLICT OF INTEREST

The authors declare no competing financial interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

ETHICS STATEMENT

Ethics approval was not required for this research.

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