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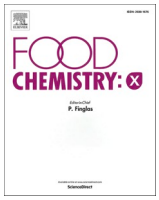

Changes in flavor profile of sauce-flavor baijiu: Perceptual interactions between 1-propanol and aroma compounds

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ABSTRACT

1-Propanol is an important aroma compound in sauce-flavor Baijiu. However, the mechanism by which it affects the aroma of sauce-flavor Baijiu has not been fully investigated. In this study, an instrumental and perceptual analysis was employed to assess the impact of 1-propanol on the flavor and volatile compounds in sauce-flavor Baijiu. The combination of sensory evaluation and electronic tongue analysis revealed that 1-propanol diminished the floral and fruity flavor and increased the bitterness and astringency. According to SHS/GC–MS analysis, 1-propanol mainly inhibited the volatility of ethyl acetate, ethyl butyrate, ethyl valerate, and ethyl hexanoate. Further, the Feller additive model and odor activity value method confirmed the four binary mixtures formed by 1-propanol and four esters had masking effects. Finally, the partition coefficients were calculated to clarify the relationship between volatility and interaction of compounds.

1. Introduction

Higher alcohols, defined as alcohols with more than two carbon atoms, mainly include 1-propanol, isobutyl alcohol, and isoamyl alcohol, were the primary fermentation products in many types of alcoholic beverages (Lachenmeier et al., 2008). Other than ethanol, higher alcohols are the key component of alcohols in alcoholic beverages, with amounts varied depending on the type of beverage. Previous studies showed that the concentration range of higher alcohols in beer was 60–100 mg/L (Zhou, 2005), while those in wine ranged from 300 to 390 mg/L (Qi et al., 2018). Notably, the concentration of higher alcohols in Baijiu was considerably higher, at 600–2500 mg/L (Lv et al., 2024). The higher alcohols are important aroma compounds, and their effect on the flavor of alcoholic beverages has been a prominent area of research. It has been demonstrated that the higher alcohols have the potential to diminish the woody flavor of wine (Ferreira et al., 2016). The appropriate quantity of higher alcohols conferred a full-bodied quality and mellow taste to alcoholic beverages (Ma et al., 2017). However, an excess of higher alcohols exerted a negative impact on the flavor of alcoholic beverages. The presence of higher alcohols in beer at a concentration exceeding 120 mg/L has been associated with the unpleasant odor and the potential for dizziness (Ma et al., 2017), while a concentration in wine above 400 mg/L was sufficient to impart a pungent taste (Arancha et al., 2016). A deficiency of 1-propanol in alcoholic beverages resulted in a loss of flavor complexity (Li et al., 2024). In general, higher alcohols serve as important flavor compounds and are extensively distributed within a wide range of alcoholic beverages, influencing the overall flavor profile.

Baijiu is a type of traditional distilled alcoholic beverage mostly produced by spontaneous solid-state fermentation using sorghum as the primary material, which is a typical representative of Chinese traditional culture (Pang et al., 2020). As one of the important types of Baijiu, sauce-flavor Baijiu had a prominent sauce aroma and mellow taste due to its distinctive production process and brewing conditions (Li et al., 2008). The sauce-flavor Baijiu exhibited a rich variety of higher alcohols. In comparison to other alcoholic beverages, the high concentration

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of 1-propanol represented a distinctive feature of its higher alcohol composition (Zhang et al., 2023). 1-Propanol was an essential aroma compound in sauce-flavor Baijiu, with its flavor being described as alcoholic, vegetal, and musty (Fan et al., 2015). The odor threshold of 1-propanol in a 53 % aqueous ethanol solution was determined to be 223.93 mg/L (Qiao et al., 2023). Studies on alcoholic beverages have shown that 1-propanol has multiple effects on the flavor. It has been demonstrated that 1-propanol had the potential to enhance the aroma of esters, resulting in a richer and more harmonious flavor profile (Zhu et al., 2024). Conversely, 1-propanol also introduced undesirable flavors like spiciness and impacted the balance of flavors (Wang, Yuan, et al., 2022). This phenomenon might be related to the varying content of 1-propanol. Meanwhile, the study has shown that the intoxicating effect of sauce-flavor Baijiu was likely to be caused by 1-propanol (Xie et al., 2018). The impact of 1-propanol concentration on the quality of sauce-flavor Baijiu is currently acknowledged as a key factor in quality assessment. Nevertheless, the effect of 1-propanol on the flavor of sauce-flavor Baijiu remains unclear. Therefore, 1-propanol was chosen as the main research object of this study to further investigate its effect on the flavor of sauce-flavor Baijiu.

The flavor of Baijiu is not contributed by a certain substance, but rather by the complex interactions between substances in Baijiu. The interactions between flavor compounds have been extensively studied. Oleic acid has been demonstrated to inhibit the volatilization of ethyl acetate and ethyl hexanoate, thus masking the aroma of strong-flavor Baijiu (Liu et al., 2024). The investigation into the interaction of 2-furylmethanethiol with eighteen skeletal aroma-active compounds revealed that 2-furylmethanethiol was more likely to produce additive or synergistic effects with esters and to enhance the release of fruity and acidic flavors (Yang et al., 2024). Similarly, the interactions between 1-propanol and the aroma compounds have also been explored. A previous study has demonstrated that the interaction between five higher alcohols, including 1-propanol, and ethyl ester aroma compounds in wine elevated the threshold of fruit aroma, thereby inhibiting fruity flavor (Cameleyre et al., 2015). High concentrations of 1-propanol or 2-phenylethanol could mask the sweaty note of 3-methylbutyric acid in sauce-flavor Baijiu (Niu et al., 2020).

However, there is a lack of comprehensive research investigating the impact of 1-propanol on the aroma compounds in sauce-flavor Baijiu. The principal objectives of this study were to: (1) determine the effect of 1-propanol on the flavor, taste and the main aroma compounds in sauce-flavor Baijiu, (2) investigate the perceptual interactions between 1-propanol and major aroma compounds, and (3) explore and discuss the effect of volatility on interactions of aroma compounds.

2. Materials and methods

2.1. Chemicals

The standard compounds are presented in detail in Table S1. All reagents were of a purity level exceeding 98 %. Sodium chloride and absolute ethanol (≥99.97 % purity) were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). A C8-C40 n-alkane standard mixture (≥99 % purity) was purchased from Sigma-Aldrich (Shanghai, China) and used to determine the retention indices (RIs) of compounds. Three internal standards, including amyl acetate, tert-amyl alcohol and 2-methylhexanoic acid, were purchased from Sigma-Aldrich (Shanghai, China).

2.2. Analysis of aroma compounds in sauce-flavor baijiu

2.2.1. Quantification of higher alcohols by gas chromatography with flame ionization detector (GC-FID)

To comprehensively analyze the content characteristics of higher alcohols in sauce-flavor Baijiu, thirty kinds of commercially available sauce-flavor Baijiu were selected for this study, all of which had an alcohol content of 53 % (*v*/v). The Baijiu samples were diluted to 10 % ethanol by volume with pure water for the following analysis. A solution of Baijiu dilution (2 mL) and sodium chloride (1 g) was added to a 20 mL sample vial. Additionally, 15 μL of tert-amyl alcohol was added as an internal standard, resulting in a final concentration of 60.38 mg/L.

The higher alcohols, including 1-pentanol, 1-butanol, 1-propanol, isoamyl alcohol, and isobutyl alcohol, were quantified on a 7890B GC equipped with a flame ionization detector (FID) (Agilent Technologies, Santa Clara, CA, USA). Samples were analyzed using a DB-WAX column (60 m × 0.25 mm × 0.25 μm, Agilent Technologies, Santa Clara, CA, USA). Helium was utilized as the carrier gas at a constant flow rate of 1.0 mL/min. The inlet temperature was 230 °C, and the detector temperature was 250 °C. Injection quantity was 1 μL, shunt ratio was 40:1. The temperature program was set as follows: the initial temperature was held at 40 °C for 3 min, then increased to 120 °C at a rate of 5 °C/min and held for 4 min. Finally, the temperature was increased to 220 °C at a rate of 15 °C/min. Heatmap was drawn using GraphPad Prism8 and the standard curves of five higher alcohols were shown in Fig. S1. The method validation results of 1-propanol were shown in Table S2.

2.2.2. Quantification of other volatile compounds by gas chromatography–mass spectrometry combined with headspace solid-phase microextraction (HS-SPME/GC–MS)

The method for the determination of aroma compounds was according to previous research (Liu et al., 2023). The Baijiu sample (1 mL) was added to a 20 mL headspace sample vial, and 5 mL of saturated sodium chloride solution was added to obtain a Baijiu dilution with an alcohol content of 9 % (*v*/v). Subsequently, 10 μL of each of three internal standards, including tert-amyl alcohol, 2-methylhexanoic acid, and amyl acetate, were added to the sample vial, resulting in final concentrations of 80.50 mg/L, 140.00 mg/L, and 103.80 mg/L, respectively. The SPME fiber (thickness 80 μm, length 10 mm, CAR/PDMS/DVB) was used to extract the aroma compounds. All samples were equilibrated at 45 °C for 5 min and then adsorbed at 50 °C for 40 min at 350 r/min. Finally, the samples were desorbed at the injection port for 5 min at 250 °C to complete the injection.

The aroma compounds of Baijiu were identified on a 7890B gas chromatography combined with a 5977B mass selective detector (MSD) and equipped with an DB-WAX column (30 m × 0.25 mm × 0.25 μm, Agilent Technologies, Santa Clara, CA, USA). The carrier gas (helium, 99.999 %) was maintained at a flow rate of 1.0 mL/min, and the split ratio was set at 20:1. The heating program was set as follows: the initial temperature was kept at 50 °C for 2 min, increased to 145 °C at 3 °C /min, then heated up to 230 °C at 15 °C /min and maintained for 3 min. The MS was operated at an electronic ionization energy of 70 eV, and the range of full scan mode was *m/z* 35–350. The temperatures of transfer line and ion source were set to 230 °C and 260 °C, respectively. The identification of volatile compounds was based on mass spectrometry data from the NIST11 database. Retention indices (RIs) and authentic standards were used for further validation. The RIs of the compounds were calculated with the C8-C40 n-alkane standard mixture under the same chromatographic conditions as the samples. The standard curves of volatile compounds were shown in Table S3, and the chromatogram was shown in Fig. S2.

2.3. Effect of 1-propanol on aroma profiles by sensory analysis

2.3.1. Sensory panel and experimental conditions

The sensory analysis was referred to the previous study by Martin et al. (Martin & Revel, 1999) and the standard sensory method (ISO, 2012). Sensory panel consisted of fifteen judges (nine females and six males, aged 22–25 years old), all of whom were from Jiangnan University and were in good health with no history of smoking. Prior to the commencement of the experiment, the judges participated in training program three times a week for a month. During this period, they were trained to accurately identify the typical aroma characteristics

associated with the samples and to precisely determine the intensity of these aromas. This involved exposure to a wide range of reference samples with known aroma profiles and intensities, allowing the judges to calibrate their senses and become familiar with the rating scales used. Prior to participation in this study, each subject was informed of the nature of the study and gave consent. This study was reviewed and approved by the Jiangnan University.

All sensory analysis experiments were conducted in the 53 % ethanol-water solution. The 20 mL sample was poured into a brown bottle, coded with a 4-digit random identifier. At the initiation of each experiment, the bottle was placed at a room temperature of 20 ± 2 °C for 10 min to allow for equilibration. All experiments were conducted in triplicate.

2.3.2. Descriptive analysis of the aroma profiles

The analysis of the aroma profiles was carried out according to previous literature (Dong et al., 2019). At the first step, panelists were requested to conduct a sensory assessment based on their subjective senses and to record descriptions of aroma attributes that they could actually perceive in the sample. To enhance the accuracy of their descriptions, they were provided with a list of commonly encountered aroma descriptors as a reference. Then, all panelists engaged in a discussion and screening of the descriptors. Eight aroma sensory descriptors, which included “sweet”, “green”, “roasted”, “floral”, “acidic”, “sauced”, “fatty”, and “fruity”, were selected and employed as a reference for the subsequent quantitative descriptions. Finally, the sensory panelists evaluated the intensity of the aforementioned eight aromatic descriptors on a scale of 0 to 10, with 0 representing no perceivable intensity and 10 representing strong intensity. Radar plots of the intensity of the eight aroma descriptors were drawn using ChiPlot (https://www.chiplot.online/).

2.4. Effect of 1-propanol on the taste using electronic tongue

The taste attributes of Baijiu were determined using an electronic tongue system (SA402B, Intelligent Sensor Technologies, Inc., Kanagawa-Pref., Japan). The six sensors and the corresponding characteristics were as follows: AAE (umami), CT0 (saltiness), CAO (sourness), C00 (bitterness), AE1 (astringency), and GL1 (sweetness). It is necessary that the sensors be activated in advance of any determination. The five basic taste sensors, comprising AAE, CT0, CAO, C00, and AE1, were soaked in the reference solution (30 mmol/L KCl solution containing 0.3 mmol/L *L*-tartaric acid) for at least 24 h. The GL1 sensor was pretreated in the sweet soaking solution at least 48 h before analysis. The Baijiu sample was diluted five times with deionized water and 40 mL of the dilution was transferred to the measuring cup. The sensors were washed in positive and negative cleaning solution for 90 s, followed by washing in the reference solution for 120 s to equilibrium to obtain the reference solution potential. Finally, the sensor was immersed in each sample for 30 s to obtain the potential of the sample. The potential difference between the sample solution and the reference solution in each sensor was converted into taste value using taste analysis software. Each sample was repeated four times after the cleaning and calibration operations, and the average value was taken as the final test result.

2.5. Effect of 1-propanol on the volatility of aroma compounds using gas chromatography–mass spectrometry combined with static headspace (SHS/GC–MS)

The impact of 1-propanol on the volatility of aroma compounds in sauce-flavor Baijiu was investigated by SHS/GC–MS. The initial concentrations of 1-propanol in the Baijiu samples A, B, and C were 237, 217, and 101 mg/L, respectively. The 1-propanol was added to the three Baijiu samples at 0, 1, 2, 3, and 4 times the actual concentration. Volatile compounds were extracted from the headspace using a PAL3 autosampler (CTC Analytics AG, Switzerland) with a hermetic needle. The temperature of the hermetic needle was set at 85 °C. The sample was kept at 35 °C for 30 min in a stationary state, and then 1 mL of gas from the vial was injected into the GC–MS using the hermetic needle. The injection mode was pulsed with no split and a pulse pressure of 25 psi. The condition of the GC–MS is identical to that described in Section 2.2.2. The changes in the peak areas of the aroma compounds indicated the changes in volatility of the aroma compounds at different concentrations of 1-propanol (Liu et al., 2024). This experiment was performed three times, and bubble charts indicating the percentage change in peak area of compounds were performed using the OmicStudio at https://www.omicstudio.cn/tool. The chromatogram showing the peak areas of volatile compounds was shown in Fig. S3.

2.6. Determination of olfactory thresholds

The olfactory threshold was determined by the three-alternative forced choice (3-AFC) method (Czerny et al., 2008). The compounds to be tested were diluted at a ratio of $1:3^n$ (n taken as 1, 2, 3, 4, 5, 6, 7, 8) according to their actual concentration in Baijiu to obtain nine different concentration groups. Each concentration group consisted of two control samples (ethanol solution, 53 %, *v*/v) and one solution of the compound to be tested. All experiments were repeated three times. Trained assessors and general conditions were identical to that described in Section 2.3.1. The result was fitted by the sigmoid curve ($P = 1/(1 + e^{-(X-C)/D})$). P is the corrected probability of detection, and X, C, and D represent the concentrations of aroma compounds, olfactory thresholds, and parametric features, respectively. P was corrected from P = (3*p-1)/2, where p is the probability of selecting correctly in each concentration group. The detection threshold was defined as the concentration at which the probability of detection was 50 % (Cometto-Muniz & Abraham, 2008).

2.7. Perceptional interaction analysis

2.7.1. Feller additive model

The thresholds of single compounds and binary mixtures were determined in ethanol solutions (53 %, v/v) using the 3-AFC method, and the concentration of binary mixtures was determined based on the quantitative concentration of their in individual compounds in Baijiu. Feller’s additive model was used to evaluate the interaction effect of aroma-active compounds (Lytra et al., 2013). The calculation of theoretical thresholds for binary mixtures was based on experimental thresholds for single substances and its calculation formula was as follows: P(AB) = P(A) + P(B) − P(A)* P(B). Define the ratio of the experimental threshold to the theoretical threshold as R. If $R \leq 0.5$, a synergy effect is observed; if $0.5 < R < 1$, it is an additive effect; if $R > 1$, the masking effect occurs; no perceptual interactions are indicated when $R = 1$.

2.7.2. The odor activity value approach

OAVs, defined as the ratio of odorant concentration to odorant threshold, were utilized to determine how much each odorant contributes to the overall aroma (Li et al., 2019) and to evaluate the interaction effects for mixtures. The olfactory thresholds used in this experiment were determined uniformly to ensure the accuracy of the results. The measured OAV of the mixture was calculated based on the concentration of the mixture and the olfactory threshold, while the theoretical OAV of the mixture represents the sum of the components. X is defined as the ratio of the theoretical OAV value to the measured OAV value, if $X \leq 0.5$, it shows a synergistic effect; if $0.5 < X < 1$, it shows an additive effect; if $X = 1$, it shows no effect; if $X > 1$, it shows a masking effect.

2.8. The partition coefficients analysis

Various concentrations of 1-propanol (0, 1, 2, 3, and 4 times

quantitative concentrations) were mixed with four esters, including ethyl acetate, ethyl butyrate, ethyl valerate, and ethyl hexanoate, respectively. Different PRs (Phase ratio) were obtained by adding different volumes of mixed solutions (0.052, 0.069, 0.103, 1.035, 2.070, 10.300, and 14.800 mL) to 20.7 mL headspace vials with PRs of 397, 299, 200, 19, 9, 1, and 0.4, respectively. The effect of various concentrations of 1-propanol on the volatility of the four esters was examined using GC-FID, with the instrumental parameters detailed in Section 2.2.1. Samples were equilibrated for at least 12 h at 20 ± 2 °C before injection and three parallels were set up for each group. Advanced Circos heatmap plot was performed using the OmicStudio tools at https://www.omicstudio.cn/tool.

The distribution coefficient method was proposed by Ettre et al. (Ettre et al., 1993). The partition coefficient was calculated using the following equation: $\frac{1}{A}=\frac{K_{g/m}}{C_i^{liq}*f_i}+\frac{1}{c_i^{liq}*f_i}*\beta$, where A represents the normalized peak area; C_i^{liq} represents the initial concentration; f_i represents the response factor of the detector; β represents the ratio of gas volume to liquid volume in the headspace vial, that is, the PR; $K_{g/m}$ represents the gas-liquid two phase partition coefficient. From the linear relationship between $1/A$ and β ($\frac{1}{A}=a+b\times\beta$), we get $K_{g/m}=a/b$. $K_{g/m}^{c_g}$ is the partition coefficient that 1-propanol was not added and $K_{g/m}^{e_g}$ is the partition coefficient of 1-propanol was added. If $K_{g/m}^{e_g}>K_{g/m}^{c_g}$, 1-propanol promotes the volatilization of esters; if $K_{g/m}^{e_g}<K_{g/m}^{c_g}$, 1-propanol inhibits the release of esters; if $K_{g/m}^{e_g}=K_{g/m}^{c_g}$, there is no effect.

3. Results and discussion

3.1. Quantification of higher alcohols in sauce-flavor baijiu

In this study, a total of 30 samples of commercially available sauce-flavor Baijiu were collected and analyzed using GC-FID with the aim to quantify five higher alcohols, including 1-propanol, isoamyl alcohol, isobutyl alcohol, 1-butanol, and 1-pentanol.

The quantitative results of 30 Baijiu samples were presented in Fig. 1. Among the higher alcohols, 1-propanol exhibited the highest concentration in sauce-flavor Baijiu, ranging from 87.2 to 390.9 mg/L. It has been suggested that the refreshing taste of sauce-flavor Baijiu may be related to relatively high content of 1-propanol (Niu et al., 2020). Isoamyl alcohol content was second only to 1-propanol, which presented astringency in sauce-flavor Baijiu (Wan et al., 2024). The astringency threshold of isoamyl alcohol was determined to be 27.62 mg/L in an aqueous solution at pH 3.8 (Wang, 2018). Among the seven astringent compounds found in sauce-flavor Baijiu, the astringency contribution of isoamyl alcohol was second only to that of 2-phenylethanol and furfural. The remaining three higher alcohols, ranked in descending order of content, were isobutyl alcohol, 1-butanol, and 1-pentanol. Moreover, the mean 1-propanol concentration of 30 Baijiu samples was calculated to be 191.8 mg/L, which was lower than the concentration reported in previous study (Wei et al., 2023). This phenomenon may be attributed to the storage time of the Baijiu. It has been proven that the concentration of 1-propanol increases with the storage time (Qiao et al., 2024). To further investigate the impact of 1-propanol concentration on the flavor of sauce-flavor Baijiu, we selected Baijiu samples A (236.6 mg/L) and B (216.8 mg/L) with 1-propanol concentrations approximating the mean value and sample C (101.0 mg/L) with a significant deviation from the mean value.

3.2. The effect of 1-propanol on the aroma and taste of sauce-flavor baijiu

We added different amounts of 1-propanol (0, 1, 2, 3, and 4 times the quantitative concentrations) to the selected Baijiu samples to investigate the impact of 1-propanol content on the overall flavor and taste of sauce-flavor Baijiu through sensory evaluation and electronic tongue determination.

First, the sensory panel evaluated the aroma of three original Baijiu samples and determined eight aroma descriptors using QDA methods, including “sweet”, “green”, “roasted”, “floral”, “acidic”, “sauced”, “fatty” and “fruity”. As shown in Fig. 2A, the intensity of eight aroma descriptors of the Baijiu samples was similar. The floral aroma exhibited the highest intensity value with a range of 8 to 10, followed by the fruity aroma with an intensity value of 6 to 8. The roasted and acidic aromas had the same intensity, both in the range of 5 to 6. As the concentration of 1-propanol increased, the aroma intensity of the floral and fruity of the three Baijiu samples showed a tendency to decrease. The lowest intensity of the floral and fruity aromas was observed at a concentration of four times the quantitative amount of 1-propanol was added, at which point the range of floral and fruity aroma intensity was 5–7 and 4–5, respectively. However, other aroma profiles showed fluctuating changes with increasing 1-propanol concentration, and these changes were not obvious. It should be noted that ester compounds mainly present floral and fruity aromas and have a strong aromatic contribution in sauce-flavor Baijiu (Ma et al., 2022). Previous research has indicated that the addition of higher alcohols to wines increases the threshold of ester compounds (Cameleyre et al., 2015). The addition of 1-propanol resulted in a reduction in the intensity of floral and fruity aromas observed in Baijiu. This phenomenon is likely attributed to the interaction between 1-propanol and ester compounds, which serves to elevate the threshold for the release of these aromas.

The electronic tongue is capable of distinguishing the taste characteristics of different foods and quantifying the intensity of taste (Weng et al., 2021). It exhibits higher sensitivity, better repeatability, and a shorter determination time than the human tongue, and consequently is widely employed in studies related to the taste of Baijiu (Zhao et al., 2024). In this study, we evaluated the effect of 1-propanol on the taste of sauce-flavor Baijiu using six taste sensors: sweetness, sourness, bitterness, saltiness, astringency, and umami. In comparison to the original Baijiu samples, the addition of 1-propanol resulted in an increase in response values across all six taste sensors (Fig. 2B). This phenomenon may be associated with the physicochemical characteristics of 1-propanol. The presence of hydroxyl groups in 1-propanol enabled the formation of hydrogen bonds between 1-propanol and polar compounds, such as water and ethanol molecules, which could dissolve each other (Zhu et al., 2018). Meanwhile, 1-propanol exhibited hydrophobic

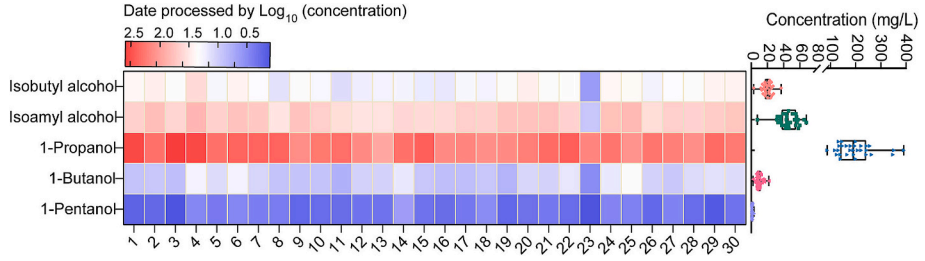

Fig. 1. Quantification of higher alcohols by GC-FID.

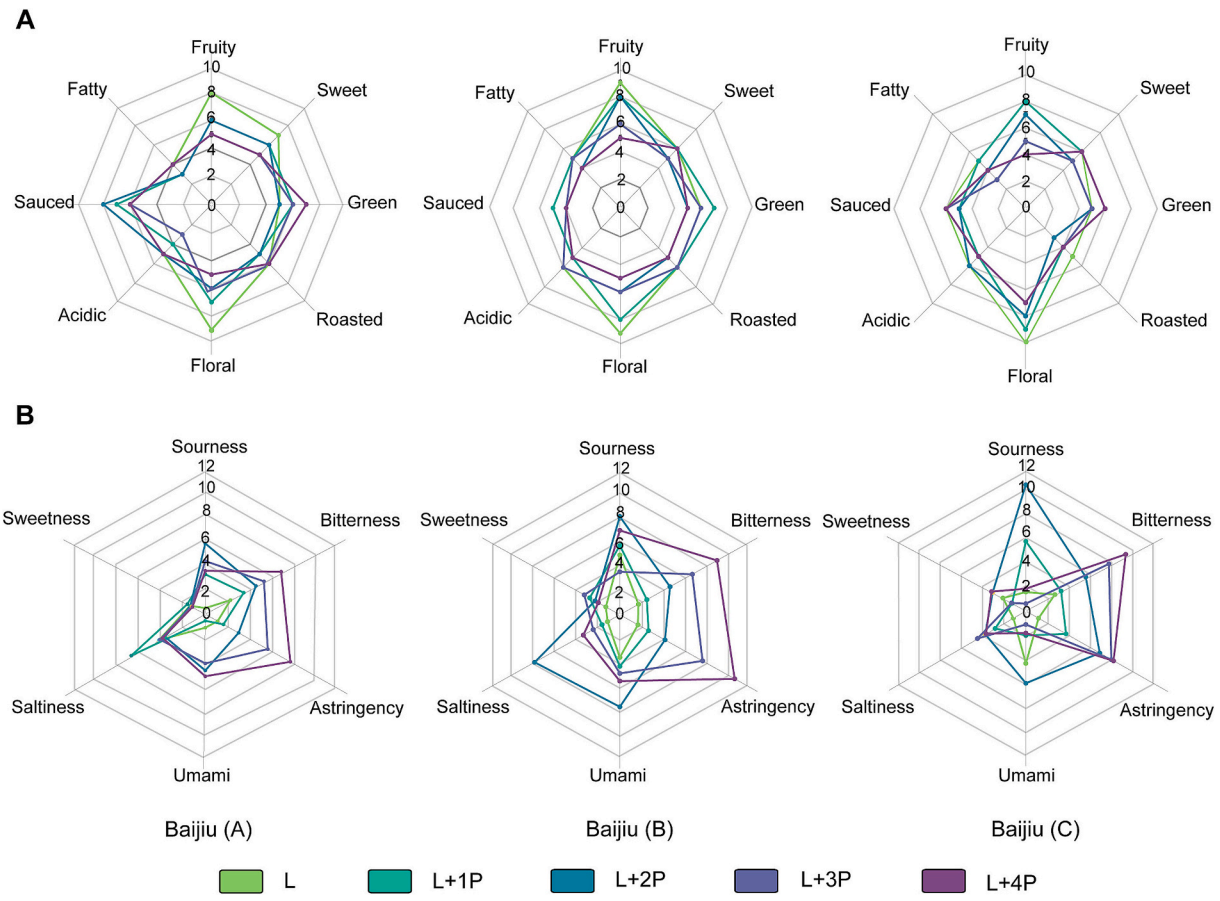

Fig. 2. Changes of aroma and taste of sauce-flavor Baijiu after adding different concentrations of 1-propanol. (A) The aroma profiles of Baijiu; (B) The changes of taste characteristic value of Baijiu. L represents the original Baijiu without adding 1-propanol, and the number in front of the letter represents how many times the quantification of 1-propanol has been added.

properties and was capable of dissolving certain non-polar compounds (Patrik et al., 2017). Therefore, the addition of 1-propanol into Baijiu resulted in an elevated concentration of dissolved compounds, which in turn led to a mostly increasing trend in the response values of the taste sensors. It is notable that the intensity of bitterness and astringency of sauce-flavor Baijiu increased monotonically with the increasing 1-propanol concentration, and the intensity varied significantly. Bitterness intensity increased from 2 to 4 to 6–10, and astringency intensity increased from 0 to 2 to 7–11. 1-Propanol was proved to be one of the main compounds responsible for the bitterness and astringency of Baijiu (Luo et al., 2020). Therefore, the increase in bitterness and astringency of Baijiu may be associated with the taste characteristics of 1-propanol.

3.3. Changes in the release of aromatic compounds following the addition of 1-propanol

The SHS/GC–MS method provided a direct indication of the changes in the volatility of the aroma compounds. In this study, a combination of direct injection and SHS/GC–MS was employed to investigate the influence of 1-propanol content on the volatility of aroma compounds in sauce-flavor Baijiu, as indicated by the change in peak area.

After the addition of 1-propanol at different concentrations, the peak areas of the most identified aroma compounds decreased (Fig. 3). In the three Baijiu samples, the proportion of compounds exhibiting a monotonically decreasing peak area relative to the total volatile compounds with increasing concentration of 1-propanol was 54 %, 46 %, and 31 %, respectively. Conversely, the proportion of compounds displaying a monotonic increase in peak area with the increasing 1-propanol concentration was 7 %. It should be noted that the peak areas of ester compounds generally decreased after the addition of 1-propanol, indicating that 1-propanol inhibited the volatilization of ester compounds, which is consistent with our sensory analysis. Interestingly, the peak area of ethyl lactate increased when a low concentration of 1-propanol was added and decreased with the increase of 1-propanol addition. The amplitude of change was not significant, and all were less than 20 %. The effect of 1-propanol on the volatility of ethyl lactate may be concentration-dependent. The peak areas of ethyl acetate, ethyl butyrate, ethyl valerate, and ethyl hexanoate in the three samples of sauce-flavor Baijiu all decreased after the addition of 1-propanol at different concentrations. The decrease in peak area was more pronounced when the concentration of 1-propanol exceeded three times the original concentration, and all of these decreases were about 30 % (Fig. 3A-B). The amplitude of change in peak area of ethyl hexanoate was the greatest after the addition of 1-propanol, and the higher the concentration of 1-propanol, the more pronounced the decrease in peak area. When four times 1-propanol was added, the peak area of ethyl hexanoate was reduced from 2.38×10^8 to 1.34×10^8, and the peak area reduction was greater than 40 % in all Baijiu samples, which was followed by ethyl butyrate, ethyl acetate, and ethyl valerate, whose peak areas were all reduced by about 30 %. Ester compounds are important aroma components of Baijiu, accounting for more than 60 % of the total volatile components of Baijiu and mostly in the form of ethyl esters. Ethyl acetate, ethyl butyrate, ethyl hexanoate, and ethyl valerate were the main ester compounds in Baijiu, and their presence contributed to enhancing the overall floral, fruity, and sweet aroma of Baijiu, as well as making the taste softer (Xu et al., 2022). Meanwhile, the OAV values of the four ester compounds in the Baijiu samples were found to be greater than 30 (Table S4), indicating that these compounds contributed significantly to

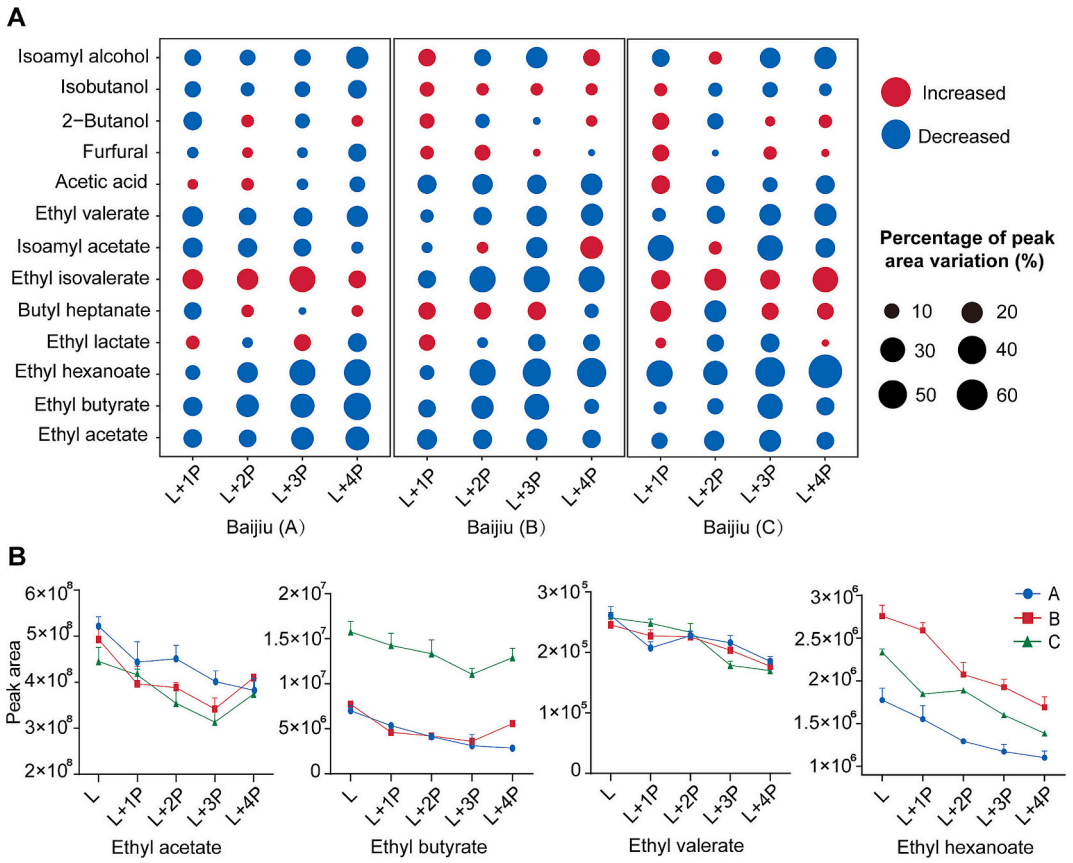

Fig. 3. Changes in peak areas of aroma compounds between the original Baijiu and the addition of 1-propanol. (A) Percentage variation in peak area of 13 compounds; (B) Changes in peak area of ethyl acetate, ethyl butyrate, ethyl hexanoate, and ethyl valerate.

the flavor of the Baijiu. Therefore, ethyl acetate, ethyl butyrate, ethyl valerate, and ethyl hexanoate were chosen as the representative aroma compounds in our subsequent experiments to explore their interactions with 1-propanol.

3.4. Determination of olfactory thresholds

In this study, we determined the olfactory thresholds of 1-propanol and four ester compounds in a 53 % ethanol aqueous solution by the 3-AFC method combined with sigmoid curve fitting to accurately explore the interaction between binary mixtures. To ascertain the olfactory threshold values of the five aroma compounds, an initial concentration was determined by averaging their quantitative concentrations in the three Baijiu samples.

The olfactory thresholds of 1-propanol, ethyl acetate, ethyl butyrate, ethyl valerate, and ethyl hexanoate were determined to be 54.3 mg/L, 23.7 mg/L, 159.8 μg/L, 54.7 μg/L, and 113.1 μg/L, respectively (Fig. 4). The threshold of 1-propanol was higher than four ester compounds, which may be associated with their disparate stability in the ethanol aqueous solution. Comparison of the olfactory thresholds among the four esters revealed that ethyl acetate was much higher than the remaining three esters, which might be related to the length of their carbon chains. It has been reported that esters with shorter carbon chains are more polar and exhibit higher solubility in ethanol aqueous solutions, thereby resulting in higher thresholds. As the chain length increased, their hydrophobicity increased while their water solubility decreased, thus lowering the threshold (Fan & Xu, 2011). Compared with the olfactory thresholds documented in the previous literature, the olfactory thresholds of ethyl butyrate, ethyl valerate, and ethyl hexanoate were all found to be higher (Liu & Sun, 2018). This may be due to the fact that the solution measured in the literature was 46 % ethanol aqueous and the olfactory thresholds of the compounds tend to increase with increasing ethanol concentration in the ethanol-water solution (Yang et al., 2023). However, the thresholds for 1-propanol and ethyl acetate were lower than previously report (Ma et al., 2020). This phenomenon may be attributed to the sensory panelists' capacity to distinguish the aroma of the compounds as well as the distinct experimental environments. In addition, previous study has also shown that these factors can affect the determination of olfactory threshold (Wang, Jing, et al., 2022).

3.5. Perceptional interaction analysis between 1-propanol and ethyl esters

In previous studies, we have known that OAV values of ethyl acetate, ethyl butyrate, ethyl valerate, and ethyl hexanoate were all greater than 30 in three samples of sauce-flavor Baijiu (Table S4), indicating that they were important contributors to the aroma of sauce-flavor Baijiu. Meanwhile, the result of SHS/GC–MS determination showed that 1-propanol inhibited the volatilization of ethyl acetate, ethyl butyrate, ethyl valerate, and ethyl hexanoate, but the mechanism of their interaction was not clear. Therefore, we used the Feller additive model and OAV approach to further investigate the interaction between 1-propanol and four ethyl esters.

3.5.1. Feller additive model

As illustrated in Fig. 5, the experimental thresholds surpassed the theoretical thresholds in all four groups of binary mixtures of 1-propanol and the four esters, indicating that the binary mixtures formed by 1-propanol and the four esters all had different degrees of masking effects. The results showed that 1-propanol had the strongest masking effect on ethyl valerate (R = 12.40), followed by ethyl hexanoate (R = 6.94), ethyl butyrate (R = 6.46), and 1-propanol had the weakest

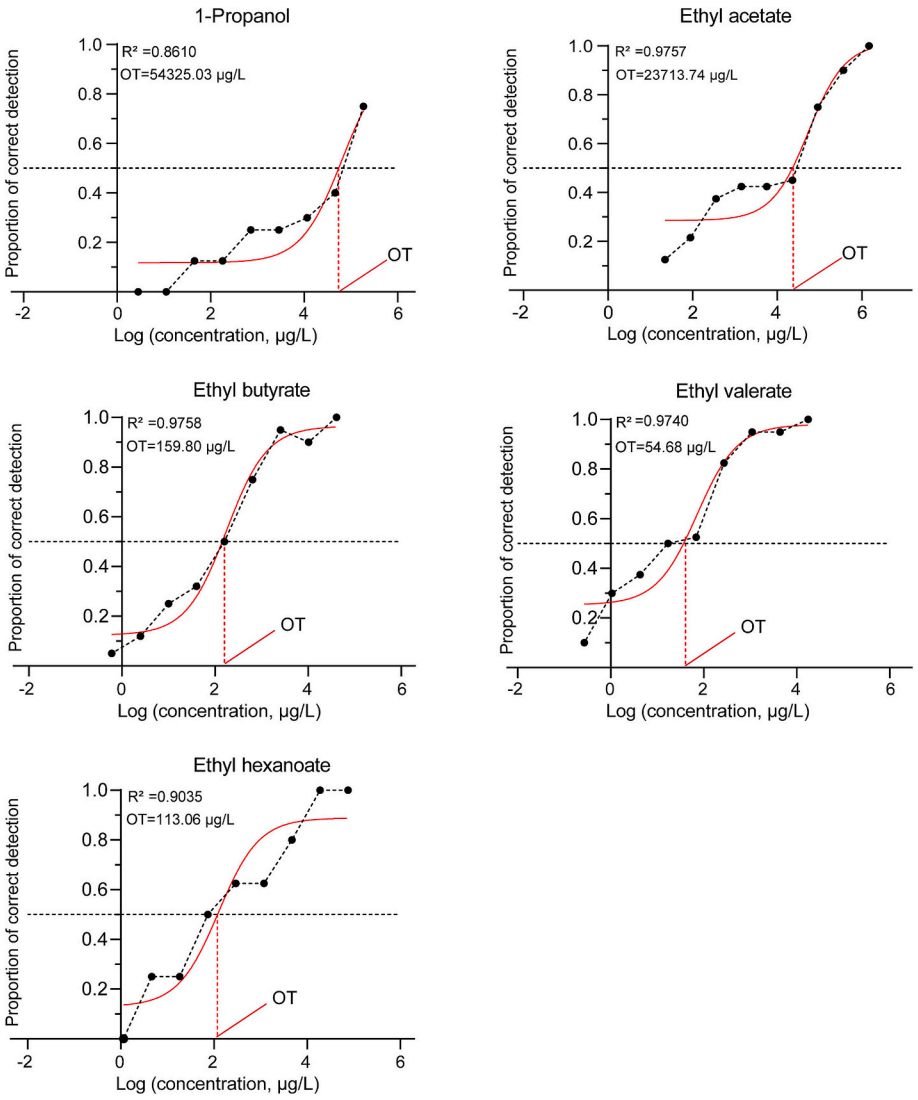

Fig. 4. Olfactory threshold determination results for five compounds in 53 % ethanol aqueous solution. OT, olfactory threshold. The curves are drawn according to a sigmoid function.

masking effect on ethyl acetate ($R = 1.74$). The masking effect between 1-propanol and four esters may be related to the olfactory system. When dealing with multiple stimuli, the olfactory system responds to strong ones by overriding the perception of weaker stimuli (Ai et al., 2023). 1-Propanol exhibited a strong irritant odor and was more likely to exert masking effects in interactions with other compounds (Lu et al., 2024). Furthermore, the masking effect among compounds of varying aroma types may be attributed to competition for binding sites on human olfactory receptors. When multiple aroma compounds were present simultaneously, they could compete for these receptors, thereby leading to masking effects between compounds (Cameleyre et al., 2020). Concurrently, research has demonstrated that the interactions between compounds are associated with their volatility, molecular interactions, and functional group structure (Chen et al., 2024). The functional group of 1-propanol is the hydroxyl group, while the ester compounds possess the ester group. The distinction in the structural characteristics of these two functional groups may also be a contributing factor to the masking effect observed in their binary mixtures.

3.5.2. *The odor activity value approach*

The odor activity value (OAV) method was employed to investigate the interaction between 1-propanol and four binary mixtures consisting of four ethyl ester compounds. This approach allowed for the consideration of both aroma component thresholds and concentrations. The degree of interaction between OAV properties in a binary mixture was determined based on its threshold and concentration (Table 1). The three groups of binary mixtures of 1-propanol and ethyl butyrate, ethyl valerate, and ethyl hexanoate exhibited varying degrees of masking effects respectively, consistent with the results of Feller's additive model. The strongest masking effect was observed between 1-propanol and ethyl hexanoate (X = 1.63), while the interaction degrees between 1-propanol and ethyl butyrate (X = 1.01) as well as between 1-propanol and ethyl valerate (X = 1.05) were similar. Nevertheless, the interaction between 1-propanol and ethyl acetate deviated from the analysis of Feller Additive Model. This may be attributed to their X (0.94) being was close to the critical values used for distinguishing different interactions.

Overall, the interactions between 1-propanol and the four ester compounds, as determined by both the OAV method and the Feller's model remained consistent, with masking effect between their binary mixtures. The results were mutually verifiable.

3.6. *The partition coefficients approach*

Different compounds exhibit distinct aromas within the matrix and vary in their odor intensity as well as their sensory interactions. These interactions could impact the release of aroma compounds from the

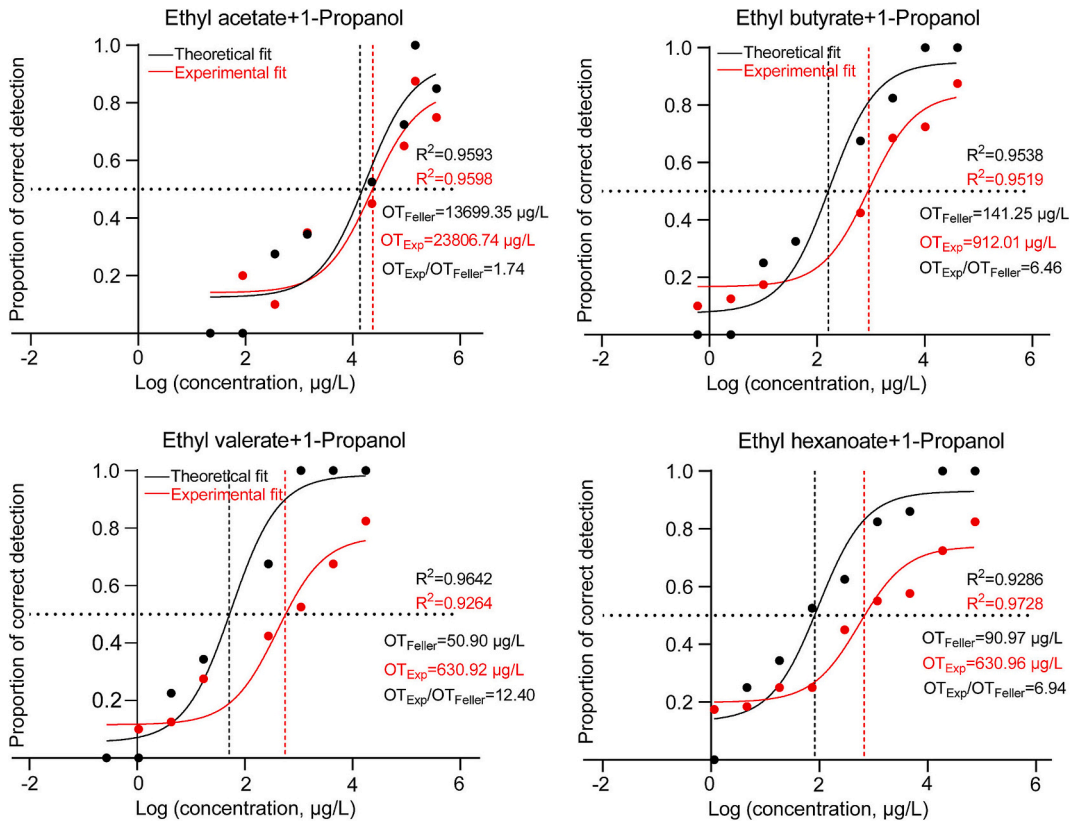

Fig. 5. Experimental results of Feller Additive Model for 1-propanol and four ester compounds in ethanol aqueous solution (53 % *v*/v). OT_{Exp}represents experimentally determined olfactory thresholds, OT_{Feller} represents theoretical olfactory thresholds.

Table 1
Interaction results of 1-propanol and ethyl esters.

Compounds	Compounds concentration[a] (μg/L)	Odor threshold[b] (μg/L)	Theoretical OAVs[c] (compounds OAVs[d])	Measured OAVs[e]	X[f]	Types of interaction[g]
1-Propanol	185,000	54,325.03	3.41	–	–	–
Ethyl acetate	1,455,000	23,713.74	61.36	–	–	–
1-Propanol and ethyl acetate	1,640,000	23,806.74	64.77	68.89	0.94	partial addition
Ethyl butyrate	40,900	159.80	255.94	–	–	–
1-Propanol and ethyl butyrate	225,900	912.01	259.35	247.69	1.05	masking
Ethyl valerate	17,600	54.68	321.87	–	–	–
1-Propanol and ethyl valerate	202,600	630.92	325.28	321.12	1.01	masking
Ethyl hexanoate	75,600	113.06	668.67	–	–	–
1-Propanol and ethyl hexanoate	260,600	630.96	672.08	413.02	1.63	masking

a, the concentrations of compounds was the average of quantitative concentration of three sauce-flavor Baijiu samples; b, determined by this study; c, theoretical OAVs for binary mixtures, which are the sum of the OAVs of the two compounds; d, OAVs of individual compounds, calculated from the compound concentration and olfactory threshold; e, the measured OAVs of the mixture, calculated from the mixture concentration and threshold; f, $X = \sum OAV_i / OAV_{mix}$; g, judging by the value of X.

matrix (Cameleyre et al., 2018). To analyze the interaction between binary mixtures, the release and retention of aroma compounds can be explained by the partition coefficient approach.

We plotted the heatmap to visualize the changes in the peak areas of the four ester compounds when different concentrations of 1-propanol were added at different phase ratios (PRs). The results showed that in the original Baijiu, the ester compounds had larger peak areas with stronger volatility at PRs of 0.4, 1, 9, and 19. When the PR was greater than 200, the peak areas exhibited a notable decline, accompanied by a reduction in volatility (Fig. 6A). The volatility of the compounds also tended to decrease roughly with increasing PR. When different concentrations of 1-propanol were added to the solutions of the four ester compounds, their volatility was inhibited, especially at PRs of 299 and 399. When the PRs were constant, different concentrations of 1-propanol exerted different effects on the volatility of the four ethyl esters. The higher the concentration of 1-propanol, the stronger the inhibition of volatility of the ester compounds. At a PR of 399, the addition of 4-fold concentration of 1-propanol had the strongest inhibitory effect on the volatilization of the esters.

The $K_{g/m}$ represents the ratio of the concentration of volatile compounds in the gas phase to that of the liquid phase matrix at thermodynamic equilibrium, which can be used to analyze interactions

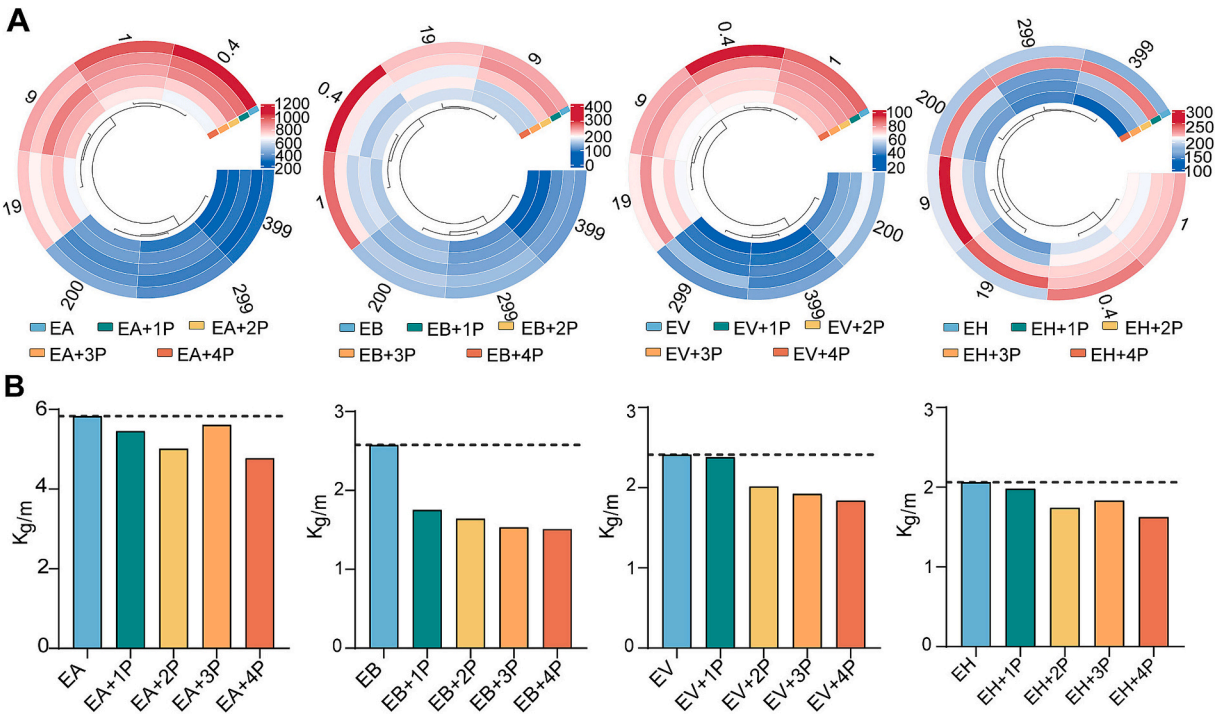

Fig. 6. The effect of 1-propanol on the volatility of four ester compounds. (A) Heatmap of peak area changes of four ester compounds after adding different concentrations of 1-propanol under PRs of 399, 299, 200, 19, 9, 1, and 0.4; (B) Changes in $K_{g/m}$ of four ester compounds when mixed with different concentrations of 1-propanol. (EA) ethyl acetate; (EB) ethyl butyrate; (EV) ethyl valerate; (EH) ethyl hexanoate.

between volatiles, compare the volatility of compounds under different conditions, and correlate it with sensory analysis (Cameleyre et al., 2018). After adding different concentrations of 1-propanol, the $K_{g/m}$ values of the four ester compounds fluctuated, but all were lower than those of the original Baijiu (Fig. 6B), indicating that after adding different multiples of 1-propanol, the molecular weight of the ester compounds in the volatile gas phase decreased and their volatility was suppressed. This was consistent with the regularity mentioned above in our results that the peak areas of four ethyl compounds in Baijiu samples decreased after adding different concentrations of 1-propanol by SHS/GC–MS. Overall, the addition of different multiples of the concentration of 1-propanol led to a decrease in the $K_{g/m}$ values of the ester compounds, suggesting that 1-propanol can inhibit the volatilization of ester compounds, thereby increasing their olfactory threshold and making it easier for binary mixtures to generate masking effects.

4. Conclusion

Through the quantitative analysis of the higher alcohols in 30 samples of sauce-flavor Baijiu, it was found that 1-propanol was the most abundant higher alcohol in sauce-flavor Baijiu, and its content was within the range of 87.2 to 390.9 mg/L. Meanwhile, the sensory evaluation combined with electronic tongue analysis revealed that the increase of 1-propanol concentration would reduce the aroma intensity of floral and fruity in sauce-flavor Baijiu and increase its bitterness and astringency. Furthermore, key compounds affected by the increase in 1-propanol content were screened by using SHS/GC–MS, including ethyl acetate, ethyl butyrate, ethyl valerate, and ethyl hexanoate. The Feller additive model and odor activity value approach revealed the masking effect between the four binary mixtures. Finally, by calculating the partition coefficient, it was further explained that increasing the content of 1-propanol could inhibit the release of four ethyl esters in Baijiu matrix, and high PR and high concentration of 1-propanol had the strongest inhibitory effect. This study provides a new perspective for the study of the influence of 1-propanol on the aroma of sauce-flavor Baijiu. However, further investigation is required to explore the molecular mechanism between 1-propanol and ethyl esters through molecular dynamics simulations.

CRediT authorship contribution statement

Han Zhao: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation. **Li-Juan Chai:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization. **Wei Zhang:** Methodology, Data curation. **Xiao-Juan Zhang:** Writing – review & editing. **Zhen-Ming Lu:** Writing – review & editing. **Song-Tao Wang:** Writing – review & editing. **Cai-Hong Shen:** Resources. **Jin-Song Shi:** Writing – review & editing. **Zheng-Hong Xu:** Writing – review & editing, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2024.102153.

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