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Comparative characterization of key compounds of Sauce-flavored rounded-Baijiu in northern and southern China and the potential possibility of similar quality of their combined products

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A R T I C L E   I N F O

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A B S T R A C T

The flavor characteristics and sensory attributes of northern Sauce-flavored rounded-Baijiu (NR) and southern Sauce-flavored rounded-Baijiu (SR) were systematically studied. A total of 56 major flavor compounds were quantitatively detected through Gas chromatography-flame ionization detection (GC-FID). Besides, 20 kinds of key aroma compounds and 7 significant differential compounds were identified by calculating the odor activity values (OAVs) and orthogonal partial least squares discriminant analysis (OPLS-DA) of flavor compounds in NR and SR. And then, the relationship between sensory attributes and key aroma compounds were investigated by network analysis. Furthermore, A group of combined-Baijiu (the ratios of the first to the fifth round of Sauce-flavored Baijiu in the north (NR1–5) were 5 % NR1, 35 % NR2, 30 % NR3, 20 % NR4, 10 % NR5) was closer to the style known for blending different proportions of SR into the combined-Baijiu. This study provided a guiding significance for the standardized production of sauce-flavored Baijiu.

1. Introduction

Chinese baijiu had been proven to be one of the oldest distillates in the world, and it had a long history of being produced by a unique fermentation method and abundant flavor (Liu & Sun, 2018; Wang et al., 2019; Xu et al., 2022). Moreover, Sauce-flavored Baijiu was recognized as one of the most famous Baijiu in China because of its diversity of flavor compounds and characteristic "sauce", "baked", "caramel" and "fruity" aromas (Yan et al., 2020). The style profile of Sauce-flavored Baijiu was prominent "sauce" aroma, elegant and delicate, mellow body, and long-lasting aftertaste (Qin et al., 2024). Besides, in 2023, Chinese Baijiu production exceeded 4 million tons, and the market value exceeded a trillion dollars. Among them, the production capacity of Sauce-flavored Baijiu had reached about 0.75 million tons, and the sales revenue had exceeded 31.6 billion dollars (Shui et al., 2024). According to the comparison of the above data, Chinese Sauce-flavored Baijiu will account for about 11.9 % of the overall production capacity and sales revenue of about 30.4 % in the Baijiu industry in 2023. Thus, Sauce-flavored Baijiu was not only loved by consumers, but also welcomed by manufacturers and enterprises for its unique style and production process.

The production of Chinese Sauce-flavored Baijiu was a spontaneous and repeated batch process, which mainly included the production of Daqu, alcoholic fermentation, solid-state distillation and aging (Kang et al., 2022). "12987" was defined as the production characteristics of southern Sauce-flavored Baijiu (Scheme 1 A), where "1" represented one-year production cycle, "2" indicated two times of sorghum input, "9" represented nine times of steaming, "8" indicated eight times of fermentation, and "7" represented seven times of distillation, and the Baijiu produced by each distillation was called rounded-baijiu (semi-combined-Baijiu) (Duan et al., 2022; Zhao et al., 2024). The rounded-Baijiu in south was called southern Sauce-flavored rounded-Baijiu (SR). At present, the national standard for Sauce-flavored Baijiu (GBT26760–2011) was mainly formulated with reference to the

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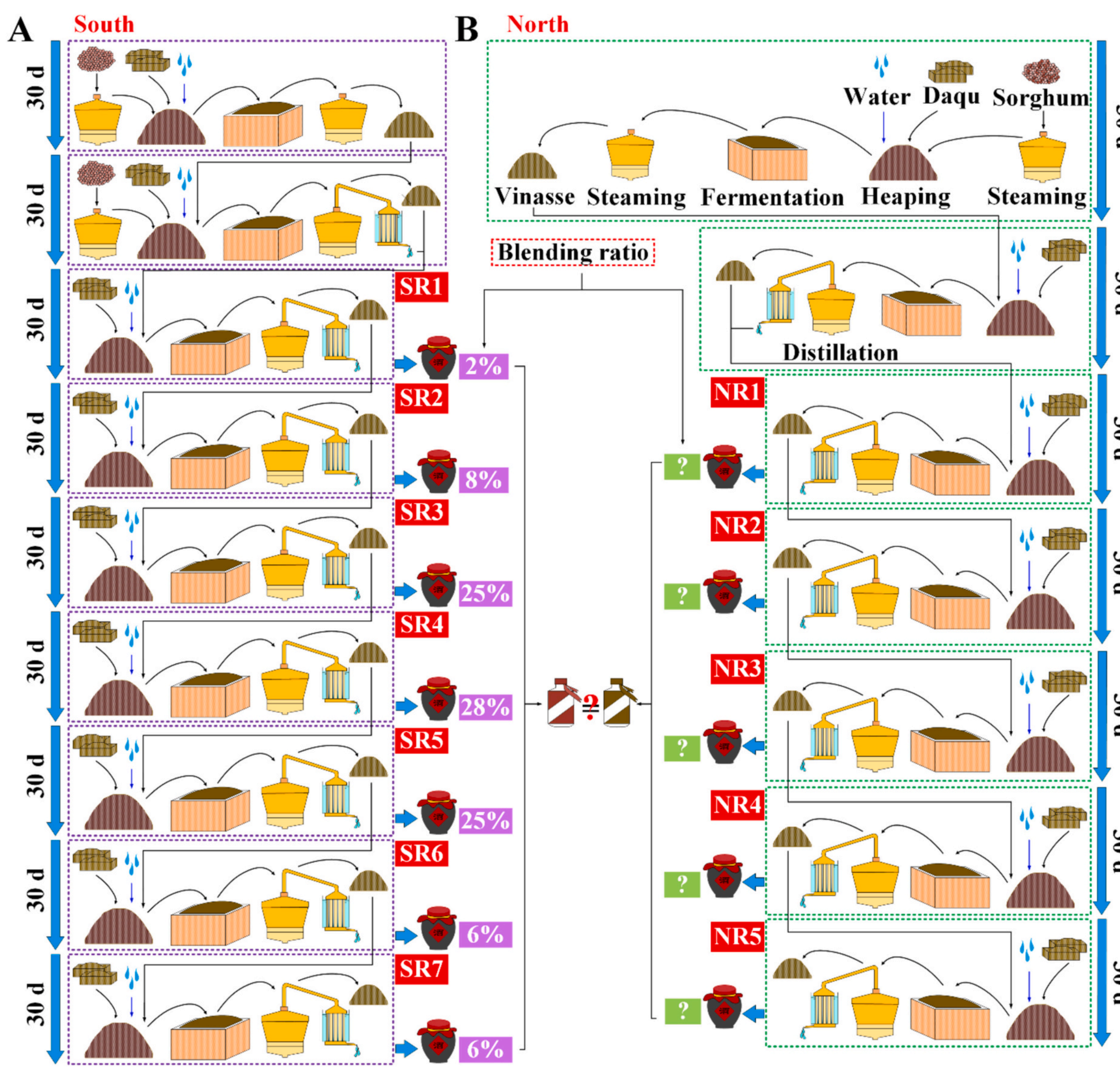
southern sauce-flavored Baijiu, but with the continuous upgrading of consumer demand, the production enterprises of Sauce-flavored Baijiu have been distributed in most parts of China, such as North China. However, the production process of Sauce-flavored Baijiu was greatly affected by regional characteristics and geographical environment, which led to different production processes in southern and northern China (Ren et al., 2024). Therefore, the production characteristics of northern Sauce-flavored Baijiu was summarized as "11765" (Scheme 1B), which the first "1" indicated one-year production cycle, the second "1" represented one time of sorghum input, "7" indicated seven times of steaming, "6" represented six times of fermentation, and "5" indicated five times of distillation, which produced five rounded-Baijiu (semi-combined-Baijiu). The rounded-Baijiu in north was called northern Sauce-flavored rounded-Baijiu (NR). Moreover, each rounded-Baijiu was blended into product the combined-Baijiu in different ratios.

In addition, previous studies had shown that the sensory characteristics of sauce-flavored baijiu produced in different regions were different, which mainly due to the differences in flavor compound content and each round of fermentation (Wei et al., 2020). Moreover, from the first round to the seventh round of southern Sauce-flavored Baijiu (SR1–7) had different style characteristics. The alcohol content of SR1 was significantly higher than that other rounds, and the "grain" aroma, "sour" and "spicy" of SR1 and SR2 were more prominent than SR3–7, which may be caused by a large number of microbial fermentation and the high utilization of starch in raw materials (Zou et al., 2024). Besides, microorganisms metabolize pyrazines and participate in Maillard and caramelization reactions of SR3–5, so they had more "sauce" aroma than other rounds of Baijiu (Chen et al., 2024; Ge et al., 2024). The high content of aldehydes of SR6 and SR7 led to a more prominent burning taste than SR1–5. However, the flavor and sensory characteristics of NR and its differences from SR were not clearly reported. Furthermore, the different proportions of SR that are known to be in the combined-Baijiu, which included 2 % SR1, 8 % SR2, 25 % SR3, 28 % SR4, 25 % SR5, 6 % SR6 and 6 % SR7. However, there was no recommended combination ratio for the northern combined-Baijiu. Thus, this paper systematically analyzed flavor compounds and sensory profile of NR and SR and tried to come up with a set of combined-Baijiu blended in different proportions of NR, and its stylistic characteristics were close to those of the south.

In this study, GC-FID technology was used to analyze the differences between southern and northern flavor compounds. Moreover, the key aroma compounds of NR and SR were identified with the help of odor



Scheme 1. The production process of sauce-flavored baijiu in south (A) and north (B). South: southern sauce-flavored round-baijiu. North: northern sauce-flavored round-baijiu. SR1–7 indicated the first to the seventh round of sauce-flavored baijiu in the south. NR1–5 represented the first to the fifth round of sauce-flavored baijiu in the north.

activity values (OAVs), and the significantly differential aroma compounds were further identified by variable importance in projection (VIP) values. Besides, the correlation of key aroma compounds with sensory attributes of NR and SR was summarized. In addition, based on the flavor compounds of VIP > 1, a group of northern five round-Baijiu with different blending ratios were selected to be similar in style to the southern combined-Baijiu. It was of positive significance to the improvement of the body design and production process of northern Sauce-flavor Baijiu. Suggestions can be made on the ratio of NR blending northern Sauce-flavored combined-Baijiu.

2. Materials and methods

2.1. Baijiu samples

A total of 12 samples (500 mL of each) were collected in 2021–2022. 5 samples were collected from 1 to 5 rounds production from the Sauce-flavored Baijiu factory located in northern China, named NR1–5. 7 samples were collected from 1 to 7 rounds production from the Sauce-flavored Baijiu factory located in southern China, named SR1–7, including the Shandong region and the Guizhou region. All the samples were stored at 4 °C until analysis.

2.2. Reagents and chemicals

All chemicals and internal standards (ISs) were high purity grade (GC grade), including n-amyl acetate, 2-octanol, 3-methylbutanoic acid ethyl ester, etc. were purchased from Aladdin (Shanghai, China). Anhydrous ethanol, sodium chloride, sodium hydroxide, concentrated sulfuric acid, potassium hydrogen phthalate, anhydrous sodium carbonate, bromocresol green, methyl red and phenolphthalein (analytically pure) were purchased from Shanghai Yien Chemical Technology Co., Ltd.

2.3. Determination of physical and chemical indexes of Baijiu

Total acid, total ester, alcohol content, and solids of Baijiu were determined according to GBT 10345–2022. The solution preparation referred to GBT 601–2016. The measurements for all indicators were taken three times and then averaged.

2.3.1. Determination of total acid in Baijiu

50.00 mL of the sample and two drops of phenolphthalein indicator were added into 250 mL triangle bottle and then placed on the base of the iron frame. Sodium hydroxide solution (0.10 M) was added dropwise in order to adjust the color of the sample to reddish and the volume of sodium hydroxide solution (V_1) consumed was recorded. Then, the total acid was calculated according to formulae (1)

$$X = \frac{C_1 \times V_1 \times 0.0601 V_2}{V_2} \times 10^6 \quad (1)$$

in the formula:

X: the content of total acid in the sample (calculated as acetic acid), mg / L; C_1: concentration of sodium hydroxide standard titration solution, M; V_1: volume of sodium hydroxide standard titration solution consumed during determination, mL; V_2: volume of sample, mL; 10^6: unit conversion factor.

2.3.2. Determination of total ester in Baijiu

After the total acid in the sample was determined, 25.0 mL of sodium hydroxide solution (0.10 M) was added to the reddish solution and mixed with shaking. The blend solution was boiled for 30 min. The collected solution was placed on the base of the iron frame and adjusted the sample color from reddish to colorless by dropwise adding the sulfuric acid solution (0.10 M) and recorded the volume consumed (V_3). At the same time, a blank test was performed with 50.00 mL of ester-free ethanol solution (40 %, *v*/v) in the same manner as described above, and the volume of the sulfuric acid standard titration solution consumed (V_0) was recorded. The ester-free ethanol solution was prepared by adding 600.0 mL of 95 % ethanol to a 1000 mL beaker. Then 5.00 mL of sodium hydroxide solution (3.50 M) was added, heated and refluxed for saponification for 1 h, then transferred to a retort for re-evaporation, and finally 40 % (*v*/v) ethanol solution was prepared (Wu & Xu, 2013). The concentration of the total ester was then calculated according to formulae (2)

$$X_1 = \frac{C_2 \times (V_0 - V_3) \times 88}{50} \times 10^3 \quad (2)$$

in the formula:

X_1: concentration of total ester in the sample (calculated as ethyl acetate), mg / L; C_2: concentration of sulfuric acid standard titration solution, M; V_3: volume of sulfuric acid standard titration solution consumed, mL; V_0: volume of sulfuric acid standard titration liquid consumed by blank test sample, mL; 88: molar mass of ethyl acetate, g·mol $^{-1}$; 50.0: volume of sample, mL.

2.3.3. Determination of alcohol content in Baijiu

Distilled water was added to the hand-held alcohol meter, washed three times, and then the sample was added to the hand-held alcohol meter and rinsed once, and the data were recorded after equilibration for 5 min after adding the sample.

2.3.4. Determination of solids in Baijiu

The determination method was slightly modified with reference to previous studies (Ma et al., 2024). Take clean glass flat weighing bottle, placed in a 103 °C drying oven, bottle cap diagonally supported on the bottle side, heated for 1.0 h, removed from the cover, placed in a desiccator cooled for 0.5 h, weighing, and repeat the drying to the front and rear of the two mass difference of no more than 2 mg, that is, constant weight. Weighing 50.0 mL sample into the weighing bottle, placed on a boiling water bath and evaporated until dry, transferred to 103 °C drying oven, the cap obliquely supported on the side of the bottle, drying 2–4 h, remove the cover and put it into the desiccator. After cooling for 0.5 h, it was weighed. Then put it into the 103 °C drying oven to dry for about 1 h, take it out, put it into the desiccator to cool for 0.5 h and then weigh it. Repeat the above until the mass difference between the two operations does not exceed 2 mg, i.e., a constant weight. Data processing see formula (3)

$$X_2 = \frac{m - m_1}{50.0} \times 10^6 \quad (3)$$

in the formula:

X_2: the content of solids content in the specimen, mg / L; m: the mass of the weighing bottle and solids, g; m_1: mass of the weighing flask after drying, g; 50.0: the volume of the specimen, mL; 10^6: unit conversion factor.

2.4. Gas chromatography-flame ionization detection (GC-FID) analysis

Baijiu samples were prepared according to previously reported work (Huang et al., 2024) with slight modifications. The sample was prepared by adding 10 μL of internal mixed standard solution (n-amyl acetate, 2-methyl-2-butanol, and 2-ethylbutyric acid) into 1 mL of Baijiu filtered by 0.22 μm microporous filter membrane. The GC-FID analysis was carried out using an Agilent 7890 A (Agilent Technologies, Santa Clara, USA) instrument coupled to an ionization flame detector. The sample was separated by a SH-Rtx-Wax (30 m × 0.25 mm × 0.25 μm) column referred to the previous literature (Gong et al., 2023). The injection temperature was 250 °C and the carrier gas was high-purity nitrogen (99.999 %). The flow rate was adjusted to 1 mL / min, the split ratio was adjusted to 30:1, and the tail blow was adjusted to 30 mL / min. The flow rate of hydrogen was 30 mL / min, the flow rate of air was 300 mL / min, and the detector temperature was 300 °C. The temperature program was maintained at 30 °C for 4 min and heated at 3 °C / min. Then the temperature was raised to 210 °C at 15 °C / min and maintained for 8 min. The standard compound was dissolved in 56 % (*v*/v) ethanol solution prepared by ethanol and ultra-pure water to obtain the mixed standard stock solution. It was then diluted into a series of different concentration gradients. After mixing the standard solution (1.0 mL) with the internal standard solution, the mixed solution was analyzed by GC-FID. The standard curve of each volatile flavor substance was drawn with the mass concentration ratio (x) of the test substance to the internal standard substance as the abscissa and the peak area ratio (y) as the ordinate (Table S6). The content of each volatile flavor substance in the wine sample was quantified by the internal standard method. The concentration in mg / L of the interest compounds (C_i) was calculated in compliance with what reported by the method (Paolini et al., 2022), using the following equation:

$$C_i = \frac{C_{IS}}{k} \times \left(\frac{A_i}{A_{IS}} - b\right) \quad (4)$$

C_{IS}: the concentration of internal standard, mg / L; k: the slope of the calibration curve; A_i: the area of the compound of interest; A_{IS}: the area of internal standard; b: the intercept of the calibration curve.

2.5. Odor activity values (OAVs) calculation

In order to evaluate the contribution of various flavor compounds to the aroma of baijiu, we calculated the overall aroma level of baijiu. In the literature, OAVs have been described as the ratio between flavor compounds and sensory detection thresholds. OAVs ≥1.0 showed that flavor compounds contributed to the overall aroma in baijiu. The data processing was described in the formula (5)

$$OAVs = \frac{C}{OT} \quad (5)$$

in the formula:

C: the concentration of flavor substance, mg / L; OT: the olfactory threshold of the aroma component, mg / L.

2.6. Sensory analysis

Sensory analysis was performed by 10 experienced and trained group members (5 females and 5 males, with an average age of 23) in a group room with a temperature of 20 ± 1 °C. *Prior* to the sensory analysis, the team members underwent a one-month training using Le Nez du Vin's standard aroma kit and compound standards. The sensory analysis method was based on the National Standard of the People's Republic of China (GBT 33404–2016) and previous experiments with slight modifications. Sensory indicators include the sensory intensity of scents like "sauce", "ethanol", "Qu", "honey", "fruity", "floral", "baked", "grain", and "caramel" aromas, as well as the taste like "sour", "sweet", "astringent", "acerbity" and "burning" were employed. The NR and SR (20 mL each) were poured into standard Baijiu tasting glasses and coded randomly. Sensory values (SVs) were scored using a 5-point scale ranging from 1 SV (lowest intensity) to 5 SV (highest intensity), compared to the reference intensity. All analyses are performed in triplicate, the average of which serves as the final score of the liquor sample and is visualized in a radar chart.

2.7. Blending ratio of Sauce-flavor rounded-Baijiu in the north

Taking the seven rounds of Sauce-flavored Baijiu blended with different proportions in the south as a reference, the five rounds of Sauce-flavor Baijiu in the north corresponded to the seven rounds of Sauce-flavored Baijiu in the south (Table 1) to design three groups of Sauce-flavored Baijiu in different proportions to blended into combined-Baijiu (Table 2).

2.8. Statistical analysis

All experimental assays were performed at least three times, and data were recorded using Excel. Column and Radar plots were constructed using Origin 2018 software (Origin Lab Inc., Northampton, MA, USA). One-way ANOVA statistical analysis was performed using IBM SPSS Statistics 26 (SPSS, Inc., USA), and comparisons were considered statistically significant if the $P < 0.05$. A heat map of the correlation flavor substances in different rounds of Baijiu were constructed using R Studio (Version 3.2.5). OPLS-DA was carried out with SIMCA version 14.0. Experimental data between key aroma compounds and organoleptic attributes identified by Spearman correlation analysis were visualized with $r > 0.6$ and significance $P < 0.05$ (http://biomediv.cn/).

3. Results and discussion

3.1. Determination of physical and chemical indexes of NR and SR

The physical and chemical indexes (total acid, total ester, alcohol content, and solids) of NR and SR were detected according to the GBT10345–2022 Baijiu analysis method. The total acid content of NR2 (Fig. 1A) and SR2 (Fig. 1B) was the highest in NR and SR, respectively, but the content in the SR2 (5430.00 ± 90.00 mg / L) was more than twice that in the NR2 (2666.67 ± 4.16 mg / L). Besides, the total ester content of NR1 (3797.20 ± 28.35 mg/L) was higher than NR2–5 (Fig. 1A), while SR2 (4716.80 ± 23.10 mg / L) had a higher total ester content than other rounds of Baijiu (Fig. 1B). In addition, the content of alcohol (NR: 51.57 ± 0.06–56.40 ± 0.20 %vol, SR: 51.05 ± 0.05–55.60 ± 0.30 %vol) and solids (NR: 1.80 ± 0.20–8.45 ± 0.35 mg / L, SR: 1.35 ± 0.15–6.38 ± 0.38 mg / L) in the NR (Fig. 1C) and SR (Fig. 1D) showed a decreasing trend with the increase of rounds.

The total acid content in the SR1 was much higher than that in the NR1. This may be due to the fact that in the early stages of fermentation of SR, the microorganisms were mainly *Lactobacillus* sp. and *Acetobacter* sp., which resulted in a higher total acid content (Wu, Duan, et al., 2023). Moreover, the total ester content in the first and second rounds of NR and SR was higher than that of the other rounds of Baijiu, which may be caused by the fact that *lactobacillus* sp. as the dominant bacterial genus promoted yeast metabolism in the fermented grain and produced a large number of esters. Besides, the content of alcohol and solids of NR and SR decreased with the increases of rounds, which was consistent with the previous study (Shen et al., 2022).

Generally, the total acid content in NR and SR increased first and

Table 1
Correspondence between NR and SR.

NR	SR
NR1	SR1 SR2
NR2	SR3
NR3	SR4
NR4	SR5
NR5	SR6 SR7

Notes: NR: northern Sauce-flavored rounded-Baijiu. SR: southern Sauce-flavored rounded-Baijiu. NR1–5 represented the first to the fifth round of Sauce-flavored Baijiu in the north. SR1–7 indicated the first to the seventh round of Sauce-flavored Baijiu in the south.

Table 2
The addition ratio of NR and SR.

Rounds	NR (%)			SR (%)
	N1	N2	N3	S
1	5	10	7	2
2	35	25	30	8
3	30	25	30	25
4	20	20	25	28
5	10	20	8	25
6	–	–	–	6
7	–	–	–	6

Notes: N1, N2, N3: Sauce-flavored rounded-Baijiu in the north, representing three groups of combined-Baijiu with different rounded-Baijiu ratios, respectively. S: Sauce-flavored rounded-Baijiu in the south. 1–7: rounds 1 to 7.

then decreased with the increase of rounds. The alcohol content and solids content decreased with the increase of rounds of NR and SR. However, the total acid and total ester contents of NR were lower than that of SR. Besides, the total ester content of NR and SR in the first and second rounds was higher than that in other rounds of Baijiu.

3.2. Sensory analysis

In order to study the differences of sensory attributes between NR and SR, 14 sensory attributes were selected to evaluate the aroma (9) and taste (5) in Baijiu. The overall flavor sensory description characteristics of NR3 and NR4 showed more "sauce" (Average SV: 3.09), "ethanol" (Average SV: 2.66) and "Qu" (Average SV: 2.77) aromas than NR1–2 and NR5 (Fig. 2A). However, the SR4 and SR5 had high aroma expressions in "sauce" (Average SV: 3.60) "ethanol" (Average SV: 3.31) and "Qu" (Average SV: 2.75) than SR1–3 and SR6–7 (Fig. 2B). This showed that the contribution intensity of the compounds with major aroma contributions in NR and SR was not consistent. It may be the flavor compounds in the SR4 and SR5 that contributed to the "sauce" and "ethanol" aroma were more abundant. Moreover, the NR1 and SR1 had higher scores for the "fruity" (NR1 SV: 2.70, SR1 SV: 3.10), "floral" (NR1 SV: 2.20, SR1 SV: 3.50), "grain" (NR1 SV: 2.93, SR1 SV: 3.13) than that in other rounds of Baijiu (Fig. 2A, B). This was consistent with previous research (Wang, Zhong, et al., 2021). Besides, the "baked" and "caramel" increased of NR and SR with incremental rounds. In addition, "sour" (NR average SV: 2.52, SR average SV: 2.42), "astringent" (NR average SV: 2.12, SR average SV: 1.82), "acerbity" (NR average SV: 2.60, SR average SV: 2.18), and "burning" (NR average SV: 2.18, SR average SV: 2.02) were found to be moderately strong and imparted a pleasantly harmonious taste (Fig. 2C, D), which was consistent with the results of previous studies (Liu et al., 2023). The intensity of sensory attributes of NR and SR changed with the increase of rounds, and the trend was basically the same.

Due to the short steaming and fermentation time of NR1 and SR1, the available starch consumption was less and the aroma in Baijiu is not fully released. Therefore, the "grain", "floral" and "fruity" aromas of NR1 and SR1 were more prominent than that of other rounds of Baijiu, which was consistent with previous literatures (Lin et al., 2024). Besides, the "sauce" aroma of NR4 and SR4 was higher than that of other

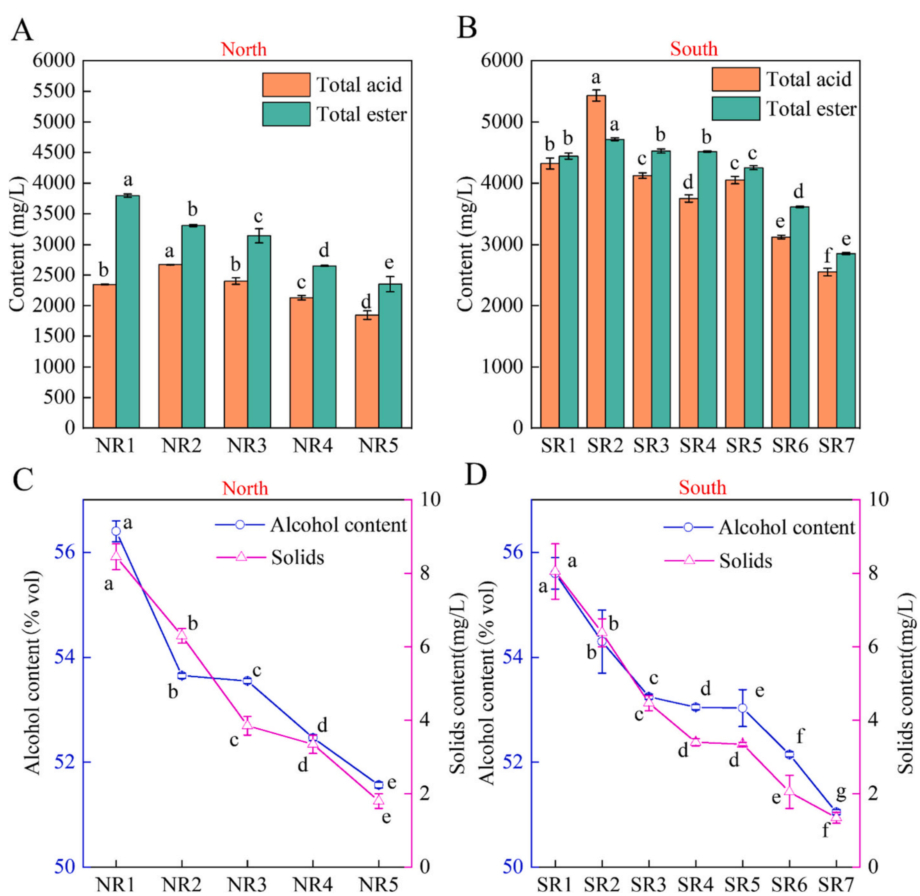


Fig. 1. Analysis of total acid and total ester of NR (A) and SR (B). Analysis of alcohol content and solids of NR (C) and SR (D). North: sauce-flavored rounded-baijiu in the north. South: sauce-flavored rounded-baijiu in the south. NR1–5 represented the first to the fifth round of sauce-flavored baijiu in the north. SR1–7 indicated the first to the seventh round of sauce-flavored baijiu in the south. Error bars in figure indicated standard deviations across three repetitive assays ($n = 3$). Significance: a, b, c, d, e, f, g: There was significant difference ($P < 0.05$) in the contents of compounds with different letters. The average contents with a was the highest and b, c, d, e, f descends sequentially, while the average contents with g was the lowest.

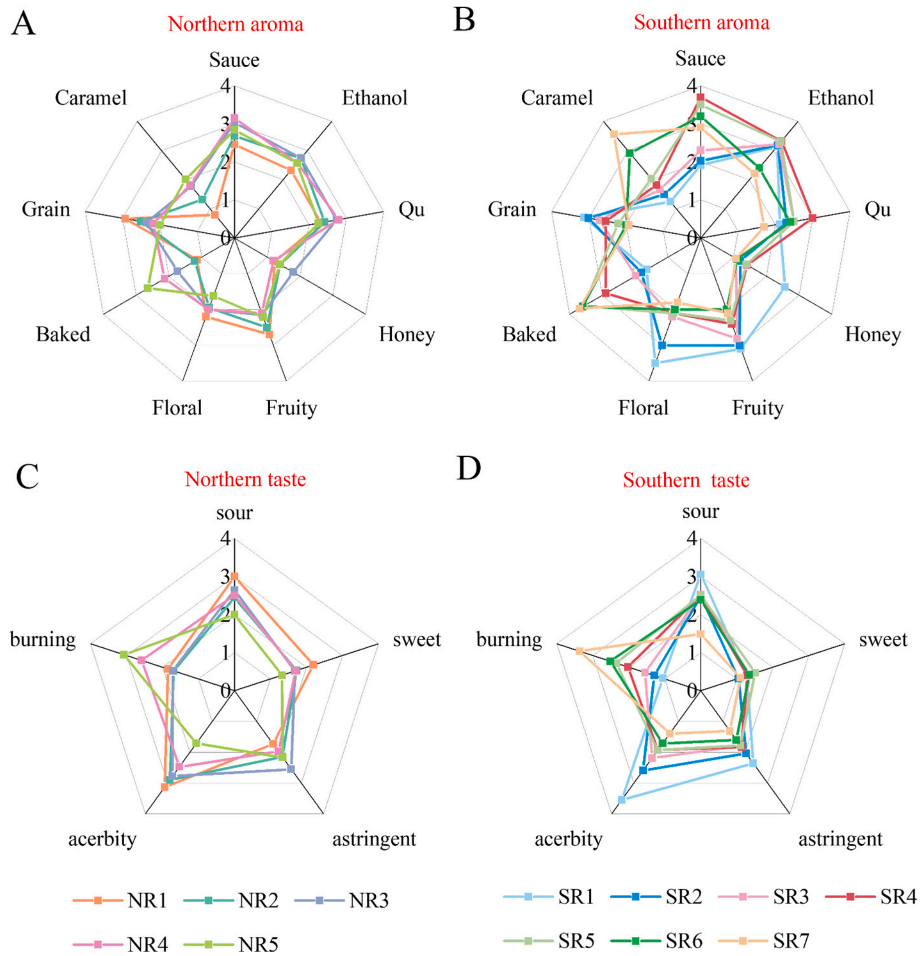


Fig. 2. Sensory analysis of NR and SR. Aroma score plots of NR (A) and SR (B). Taste score plots of NR (C) and SR (D). Northern and southern aroma represented aroma of northern and southern sauce-flavored rounded-baijiu, respectively. Northern and southern taste indicated taste of northern and southern sauce-flavored rounded-baijiu, respectively. NR1–5 represented the first to the fifth round of sauce-flavored baijiu in the north. SR1–7 indicated the first to the seventh round of sauce-flavored baijiu in the south. The content of aroma compounds was expressed as the sum of mean value of triplicate samples in figure.

rounds of baijiu. The reason for this result was that *Bacillus* sp. was abundant in the fourth round as it can survive in extreme environments, it could also secrete proteases and amylases, and forms Sauce-flavored precursors (Wang, Huang, et al., 2021). Moreover, the "sour", "astringent" and "acerbity" with the increase of rounds showed a decreasing trend. Due to the influence of processing parameters, raw material characteristics (tannins and polyphenols) and microbial structure of the fermentation process in the pre-fermentation round of Sauce-flavored Baijiu (Liu & Sun, 2018; Wang, Xu, et al., 2021).

The aroma of Sauce-flavored Baijiu mainly comes from the contribution of flavor compounds. The results revealed that the characteristics of each round of NR were that NR1–2 had more "grain", "floral", "fruity", "sour" and "astringent", and the "sauce" "ethanol" and "Qu" aroma were more prominent in NR3–4, and the "baked" aroma of NR5 were more obvious (Fig. 2A). Moreover, there were significant differences in the aroma intensity of SR1, SR6 and SR7 (Fig. 2B). Besides, sensory characteristics of each round of NR and SR was unique, which closely related to the composition of flavor compounds produced during fermentation. Based on this, the flavor compounds and their differences of NR and SR were further analyzed.

3.3. Flavor compounds profiled of NR and SR

As we all known, Baijiu contained a large number of different kinds of flavor compounds, such as acids, esters and alcohols. These compounds had different sensory characteristics, influencing each other and contributing together to the diversity of Baijiu flavors (Huang et al., 2023). In order to explore the differences in the content of flavor compounds of NR and SR, a total of 56 flavor compounds were quantified by GC-FID, including 14 alcohols, 7 aldehydes, 9 acids, 21 esters, 2 ketones and 3 others (Table S1). The esters were the most abundant in NR and SR, which was mainly generated by the esterification reaction of acid and alcohol in the Baijiu. Moreover, the esters were one of the important aroma components in Sauce-flavored Baijiu.

The results demonstrated that the alcohol content in NR1 and NR2 was higher than that in other rounds of Baijiu (> 4000 mg / L) (Fig. 3A), and SR1 was had the highest content of alcohols in SR (21,178.13 ± 737.90 mg / L) (Fig. 3B). Moreover, aldehydes increased first and then decreased with the increase of rounds of NR, and the highest concentrations of aldehydes reaching 1777.80 ± 0.58 mg / L of NR4 (Fig. 3A). The content of aldehyde compounds showed an overall upward trend of SR and reached the highest of SR7 (959.62 ± 5.48 mg / L) (Fig. 3B). Besides, acid and ester compounds increased first and then decreased with the increase of rounds of NR, with the highest concentrations reaching 2877.12 ± 138.46 mg / L and 7174.26 ± 31.88 mg / L of NR2 (Fig. 3A). The contents of acids and esters showed an overall downward trend of SR and reached the highest of SR1 with the highest values of 4818.34 ± 147.26 mg / L and 10,829.94 ± 427.09 mg / L (Fig. 3B).

Based on this, in order to blend a northern combined-Baijiu that was close to the southern style, the differences in the flavor compounds corresponding to SR1–7 from NR1–5 were further analyzed. Compared with the SR, alcohol compounds of NR1 were much lower than that of

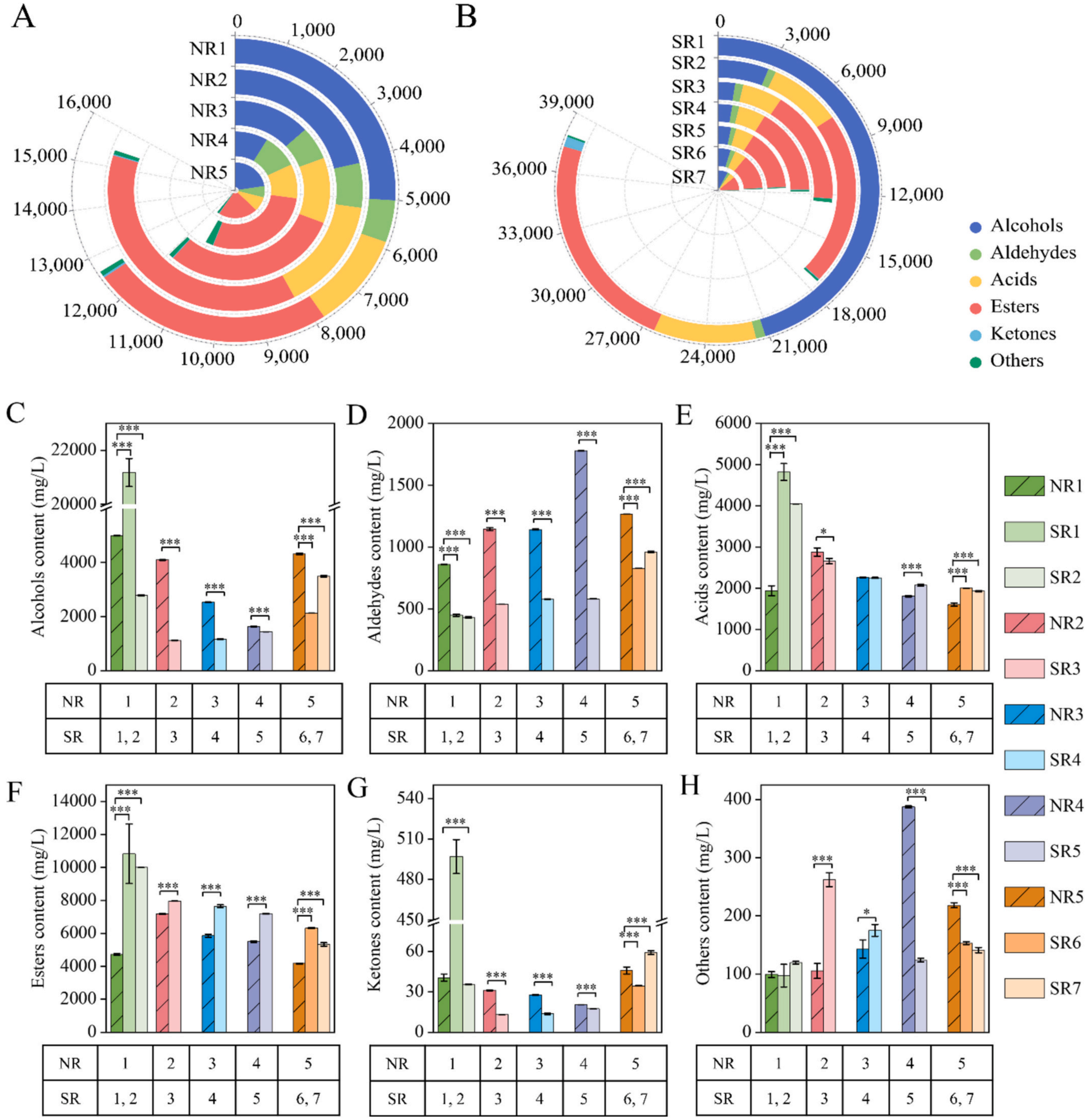


Fig. 3. Analysis of flavor compounds of NR (A) and SR (B). Analysis of alcohol compounds of NR and SR (C). Analysis of aldehyde compounds of NR and SR (D). Analysis of acid compounds of NR and SR (E). Analysis of ester compounds of NR and SR (F). Analysis of ketone compounds of NR and SR (G). Analysis of other compounds of NR and SR (H). NR1–5 represented the first to the fifth round of sauce-flavored baijiu in the north. SR1–7 indicated the first to the seventh round of sauce-flavored baijiu in the south. The content of flavor compounds was expressed as the sum of mean value of triplicate samples (A, B). Error bars in figure indicated standard deviations across three repetitive assays ($n = 3$, C—H). Significant difference of flavor compounds in NR and SR represented by *** ($P < 0.001$), ** ($P < 0.01$) and * ($P < 0.05$), respectively (C—H).

SR1, while the content of alcohol compounds of NR2–5 was higher than that of SR2–7 (Fig. 3C). Particularly, the SR1 contained more 1-propanol (15,816.71 ± 443.37 mg/L) and 2-pentanol (3036.79 ± 66.19 mg/L) than the NR1 (Table S2). Moreover, the aldehyde compounds of NR was higher than that of SR (Fig. 3D), and the contents of acetaldehyde, acetal and furfural were higher than that of other aldehydes (Table S3). Besides, the content of acid compounds of NR1, NR4 and NR5 were lower than that in the corresponding SR, but acid compounds of NR2 and NR3 were slightly higher than that in the corresponding SR (Fig. 3E). Especially, the content of acetic acid glacial in the SR1 was about three times that of NR1 (Table S2). In addition, more ester compounds were found of SR than that of NR (Fig. 3F), and two high-content compounds included ethyl acetate and ethyl lactate (Table S3). The content of ethyl acetate was the highest of NR2 (3334.99 ± 27.63 mg/L) in NR1–5, and was the highest of SR1 (5315.62 ± 61.96 mg/L) in SR1–7. Moreover, the ethyl lactate content of (NR2 2665.58 ± 12.01 mg/L) and SR2 (4874.95 ± 8.25 mg/L) was the highest in NR and SR, respectively. The content of ketone and other compounds did not differ much between the NR (Fig. 3G) and SR (Fig. 3H).

Ethanol was one of the main components of Chinese baijiu. Due to the raw materials and the fermentation environment, the SR1 had a higher ethanol content than other rounds of Baijiu. Excessive levels of higher alcohols had negative impacts on the quality of Baijiu (Fan et al., 2023; Wei et al., 2024). Moreover, compared to ethanol, appropriate amounts of higher alcohols can contribute to f the formation of aromas, with 1-propanol and 2-pentanol having grassy, mellow, and ripe fruit aromas (Jia et al., 2021). When the ratio of higher alcohols was properly balanced, they give the Baijiu a sweet, flavorful, and mellow feel (Wang et al., 2024). Therefore, the rational addition of the first round of Baijiu in the process of Baijiu blending played an important role in the balance and coordination of alcohols in the Baijiu. Besides, aldehydes were mainly derived from the metabolism of yeast and the oxidation of alcohol during fermentation (Sun et al., 2022). Aldehydes are important flavor compounds and have a harmonizing effect on the flavor of Baijiu, and the interaction with acids can affect the flavor of Baijiu and promote the formation of esters (Wu, Chen, et al., 2023). The higher content of aldehydes of NR may be caused by the addition of more auxiliary materials during the fermentation process. Moreover, acetaldehyde, acetal and furfural imparted the undesired aromatic attributes of green apples, fresh-cut grass, walnuts and bitterness (Aguera et al., 2018; Dong et al., 2024). Acid was the precursor of ester compounds, which mainly affected the richness of baijiu, and the right amount of acid can not only balance the flavor but also improve the taste and aftertaste of baijiu (Niu et al., 2024). Acids mainly exhibit cheesy aromas, while esters mainly exhibit floral and fruity aromas, and the ratio between the two will affect the interactive expression of aromas (Hong et al., 2023; Wang et al., 2019). The total acid and total ester content of the first and second rounds were higher than those of the other rounds of Baijiu, and were produced by the use and metabolism of a large number of starch, fat and protein raw materials. Ethyl acetate had the fruity flavor of apple and banana and has a soft taste, while ethyl lactate had a fruity and slightly fatty flavor with a pungent and bitter taste (Xu et al., 2022). They can increase the aroma release of Baijiu and made the flavor of Baijiu harmonized. The content of ketones and other compounds was less and did not differ much of NR and SR.

In summary, the content of flavor compounds of NR was not significantly different of NR1, NR3–5 and SR3–7, except for the higher content of NR2, SR1 and SR2. Besides, compared with the SR, ester compounds of NR were lower, the content of aldehyde compounds was higher, but aldehydes can promote the formation of esters. Acid, ketone and other compounds were not significantly different. In addition, except for the alcohol compounds of SR1 was significantly higher than NR1, the content of SR2–7 was lower than that in the corresponding NR. Reasons for these results may be the difference between the production process of NR and the SR and the different regional environments. This result suggested that it was possible to adjust the ratio of NR to make it close to the style of the southern combined-Baijiu.

3.4. Relationships between the sensory attributes and key aroma compounds

Odor activity values (OAVs) were used to evaluate the contribution of aroma components to the overall aroma of Baijiu (Wei et al., 2023). To further determine the aroma contribution of flavor compounds in NR and SR, OAV was estimated using previously published aroma active component thresholds. The analysis revealed 20 kinds of key aroma components (Table S4) with OAVs ≥ 1.0 of NR and SR, grouped into 4 categories of aroma compounds were found: alcohols (n = 3), aldehydes ($n = 2$), acids ($n = 5$), esters ($n = 10$). Besides, the R language was used to draw a heat map of the content clusters of the key flavor compounds of NR (Fig. 4A) and SR. The esters (ethyl acetate, ethyl propionate, ethyl butyrate, ethyl isovalerate, isoamyl acetate, ethyl valerate, ethyl caproate, ethyl lactate, ethyl caprylate, ethyl nonanoate) account for 50 % of the total kinds (20) of key aroma compounds in both NR and SR, followed by acids (25 %: butyric acid, isovaleric acid, valeric acid, hexanoic acid, octanoic acid), alcohols (15 %: 1-propanol, 1-butanol, 3-methylbutane), and aldehydes (10 %: isovaleraldehyde, furfural). Moreover, the NR1 was classified into one category alone, and the remaining 4 rounds (NR2–5) were clustered into another category, and NR3, NR4 and NR5 were more similar to each other, among which NR3 and NR4 were more similar to each other (Fig. 4A). Besides, the content of the key aroma compounds was significantly different in SR1, compared to other 6 rounds of Baijiu (SR2–7). At the same time, SR6 and SR7 had similar levels of key aroma compounds, while SR3–5 also show significant similarities (Fig. 4B). This may be related to the difference in regional environments and production processes between the north and the south.

Aroma compounds have been recognized to have aroma-sensing effects and have an enhancing effect on odor intensity positively correlated with them. To further explore the correlation between the sensory attributes of NR and SR and key aroma compounds. The potential correlation between sensory attributes and aroma compounds was investigated using network analysis. The Spearman correlation coefficients between the 20 kinds of key aroma compounds and 14 sensory attributes were calculated. There were 11 aroma compounds with coefficients $P < 0.05$ and $r > 0.6$ of NR, including 1 alcohol (1-propanol), 4 acids (butyric acid, hexanoic acid, isovaleric acid, octanoic acid), 6 esters (ethyl acetate, ethyl caproate, ethyl caprylate, ethyl nonanoate, ethyl valerate, isoamyl acetate) (Fig. 4C). Among them, the sensory attributes "sauce", "ethanol", "Qu", "fruity", "baked", "grain", "caramel", "sweet", "acerbity" and "burning" were significantly correlated with 3, 2, 2, 3, 3, 3, 3, 1, 3 and 4 aroma compounds, respectively. The "sauce" aroma of NR was positively related to 1-propanol, butyric acid and isoamyl acetate. Besides, there were 15 aroma compounds with coefficients $P < 0.05$ and r > 0.6 of SR, including 2 alcohol (1-butanol, 1-propanol), 2 aldehydes (furfural, isovaleraldehyde), 5 acids (butyric acid, hexanoic acid, isovaleric acid, octanoic acid, valeric acid), 6 esters (ethyl acetate, ethyl butyrate, ethyl caproate, ethyl lactate, ethyl nonanoate, isoamyl acetate) (Fig. 4D). Among them, the sensory attributes "sauce", "ethanol", "Qu", "honey", "fruity", "floral", "baked", "grain", "caramel", "sour", "astringent", "acerbity" and "burning" were significantly correlated with 1, 8, 6, 3, 4, 4, 4, 4, 4, 4,4, 4 and 4 aroma compounds, respectively. In addition, most of these aroma components were positively related to the "ethanol" and "Qu" aromas. The "ethanol" aroma of SR was significantly positively related to 1-butanol, butyric acid, ethyl caproate, this was consistent with the results of the studies that have already been reported (Ma et al., 2020). Then, 1-propanol and 3 esters (ethyl butyrate, ethyl lactate, isoamyl acetate) were significantly positively associated with the "Qu" aroma of SR. Moreover, the "fruity" and "floral" aroma of SR were positively related to furfural, isovaleraldehyde, octanoic acid and ethyl acetate. Overall, the flavor compounds that were positively correlated with sensory attributes of SR were more complex than those of NR.

Among the key aroma compounds of NR and SR, esters were the most diverse, mainly formed by esterification of fatty acids during fermentation, and esters also contributed to the creation of pleasant floral, pineapple, apple and banana aromas (Fan & Qian, 2006; Niu et al., 2017). Besides, the results of this study revealed that the "sauce" aroma was positively correlated with 1-propanol, butyric acid and isoamyl acetate. The main aroma components of Sauce-flavored Baijiu were uncertain, and the main "sauce" aroma was a compound of multiple components and odors, which needs further research (Sun et al., 2021). Moreover, previous studies had also shown that some aroma compounds, such as butanoic acid, octanoic acid, ethyl acetate, are related to the long-persistence retronasal aroma of Baijiu (Yu et al., 2023). In addition, it may be that the more complex production process and

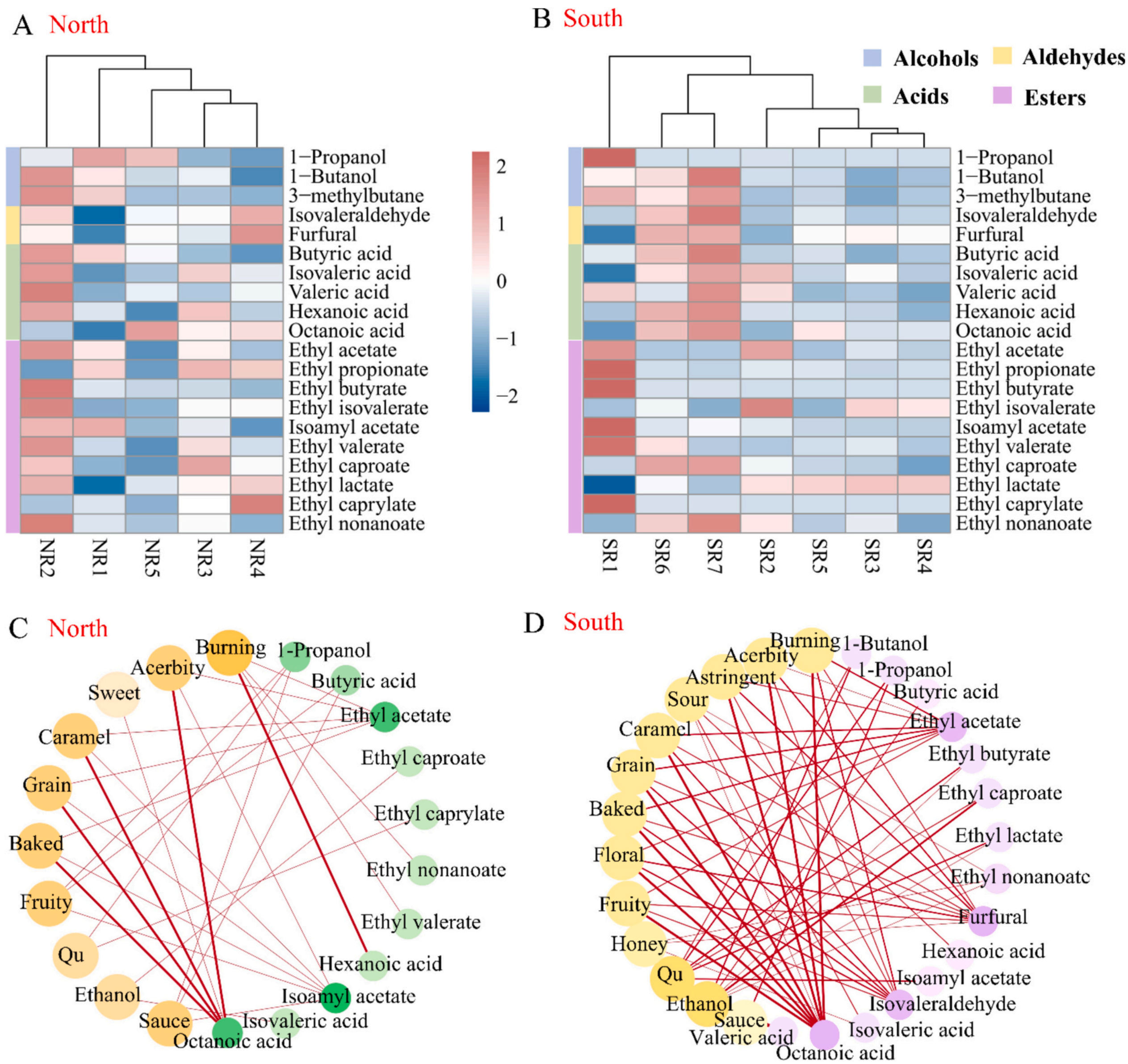


Fig. 4. Analysis of the key aroma compounds of NR and SR. The OAVs of key aroma compounds of NR (A) and SR (B). The network chart of significant correlations with the aroma and taste in the key compounds of NR (C) and SR (D). North: sauce-flavored rounded-baijiu in the north. South: sauce-flavored rounded-baijiu in the south. NR1–5 represented the first to the fifth round of sauce-flavored baijiu in the north. SR1–7 indicated the first to the seventh round of sauce-flavored baijiu in the south. The color (from blue to red) indicated the relative intensity change from low to high (A, B). The orange nodes represented sensory attributes of NR, and the green nodes represented aroma compounds of NR. The yellow nodes indicated sensory attributes of SR, and the purple nodes indicated aroma compounds of SR. The thickness of the lines was proportional to the value of Spearman's correlation ($r > 0.6$, $P < 0.05$). The shade of color was related to the size of the node data. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

special regional environment of SR had led to a richer variety of aroma compounds that were positively correlated with sensory attributes than those of NR.

3.5. Significant difference compounds analysis of NR and SR

OPLS-DA is a supervised discriminant analysis statistical method that mimics the relationship between metabolite expression and sample class. The parameters R2 and Q2 help to judge the accuracy and reliability of the OPLS-DA model (Zhang et al., 2024). OPLS-DA analysis was conducted based on the 20 kinds of key aroma compounds determinations to further explore the differences in aroma characteristics among the NR and SR further (Table S5). Moreover, R2 < 0.3 and the regression intercept of Q2 < 0.5 indicated that the OPLS-DA model has a high degree of stability and reliability. Based on OPLS-DA of the key aroma compounds of NR and SR, it can be seen that the Baijiu of NR (Fig. 5A) and SR (Fig. 5B) can be well separated, indicating that there were differences in key aroma compounds among different rounds. Particularly, there were obvious differences in the key flavor compounds between the three groups of NR1, NR2–4 and NR5 (Fig. 5A). And there were significant differences in the key aroma compounds between the three groups of SR1–2, SR3–5 and SR6–7 (Fig. 5B). This was consistent with the results of the previous grouping (Fig. 3).

The importance of compounds was assessed using variable importance (VIP), and compounds with VIP > 1.0 were considered significant (Wang, Ming, et al., 2021). Besides, compounds with $P \leq 0.05$ were considered statistically significant (Niu et al., 2015). The results showed that 6 kinds of key aroma compounds with VIP >1.0 and $P \leq 0.05$ (Table S5) were found of NR (Fig. 5C) and SR (Fig. 5D), respectively, of which five (1-propanol, ethyl acetate, ethyl lactate, ethyl propionate and

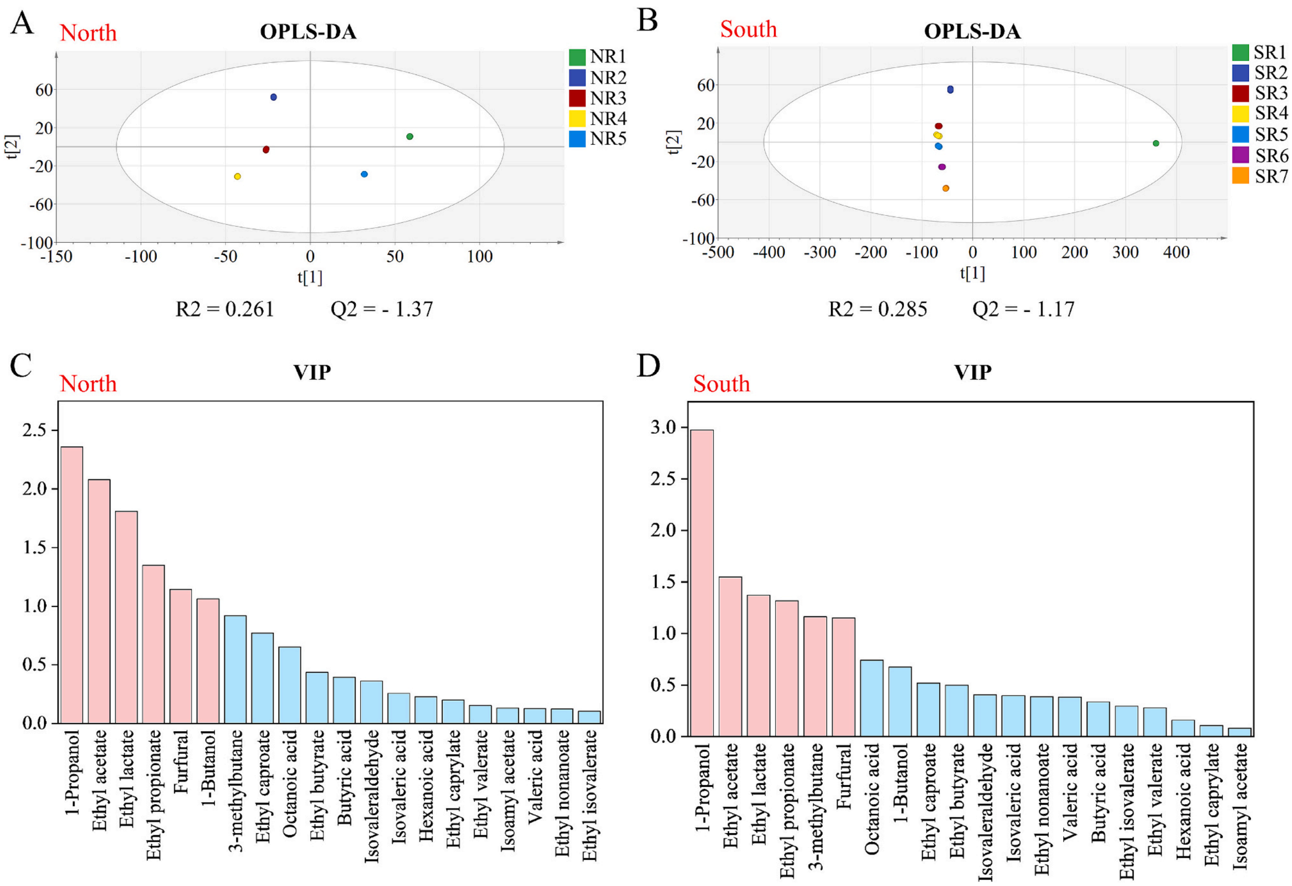


Fig. 5. OPLS-DA analysis of key aroma compounds of NR (A) and SR (B). The VIP of key aroma compounds of NR (C) and SR (D). North: sauce-flavored rounded-baijiu in the north. South: sauce-flavored rounded-baijiu in the south. NR1–5 represented the first to the fifth round of sauce-flavored baijiu in the north. SR1–7 indicated the first to the seventh round of sauce-flavored baijiu in the south. The key compounds with VIP > 1.0 and compounds with VIP < 1.0 were colored red and blue, respectively (C, D). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

furfural) were shared by both. In addition, the key aroma compounds with VIP > 1.0 specific to NR and SR were 1-butanol and 3-methylbutanol, respectively. 1-propanol had the highest VIP value of NR (2.36) and SR (2.98) (Table S5), with the highest content of NR1 (3095.94 ± 5.16 mg / L) and SR1 (15816.71 ± 443.37 mg / L) in NR and SR, while the lowest of NR4 (383.38 ± 0.35 mg / L) and SR4 (250.39 ± 1.10 mg / L) in NR and SR (Table S1). Similarly, Ethyl acetate exhibited the second-highest VIP value of NR (2.08) and SR (1.55) (Table S5), displayed the highest content of NR2 (3334.99 ± 27.63 mg / L) and SR1 (5041.00 ± 71.53 mg / L), while the lowest content was observed of NR5 (1333.02 ± 17.17 mg / L) and SR5 (2000.11 ± 22.83 mg / L) in NR and SR (Table S1). Additionally, 3-methylbutanol was also identified as a significant differential aroma compound, which consistent with previous studies (Wu, Chen, et al., 2023).

The production of 1-propanol, 1-butanol and 3-methylbutanol was closely related to the fermentation process after the protein in the Sauce-flavored Baijiu raw materials was converted into amino acids. They had fruity, floral and grassy aromas, which made the flavor and taste of Baijiu harmonious and increases the sweetness and aftertaste of the Baijiu (Zhang et al., 2009). Besides, ethyl acetate and ethyl lactate in Sauce-flavored Baijiu were mainly acetic acid and lactic acid that can be converted through enzymatic reactions to provide fruity and floral aromas for Baijiu (He et al., 2019). Moreover, furfural was mainly brought by excipients (sorghum husks, rice husks, corn cobs, etc.) and had a bitter almond flavor.

3.6. *The rational design of recipes of NR*

Sauce-flavored rounded-Baijiu was blended by seven rounds of Baijiu according to a certain proportion. The first and second rounds of Baijiu in southern China had more prominent raw grain aroma, which should be added in an appropriate amount during blending. The yield of the third, fourth and fifth rounds of Baijiu in the south was large, the sauce aroma was prominent, the Baijiu body was mellow and full, and the amount of addition can be appropriately enlarged during the blending. The sixth and seventh rounds of Baijiu in the south had burning aroma, with astringent taste, and the addition amount should not be too large. In addition, the specific proportion of blended combined-Baijiu was determined according to the sensory evaluation of the wine taster (S). Due to the influence of regional environment and climate, northern Sauce-flavored Baijiu can only produce five rounds of Baijiu every year. Based on the previous research in this paper, it was found that NR and SR styles were similar.

Based on the results of significant difference compounds analysis of this paper, the proportion of the second, third and fourth rounds Baijiu of blending in the northern combined-Baijiu was calculated (Table S20) by referring to the content of the common five significantly difference compounds in the third, fourth and fifth rounds Baijiu of southern combined-Baijiu (Table S19). Three groups of NR with different proportions (N1, N2, N3) were designed to be blended into combined-Baijiu according to the existing ratio and slightly modified, and the styles of southern combined-Baijiu were compared. The overall aroma profiles of these samples were shown by a radar chart based on the average intensity of the 14 sensory attributes (Fig. 6). The overall flavor sensory

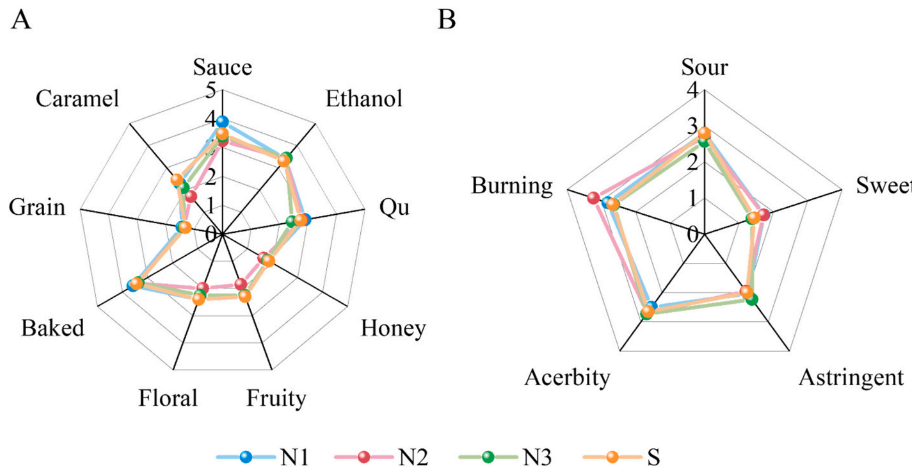


Fig. 6. Sensory analysis of NR and SR with different rounds of blending ratios. Aroma score plots of N1, N2, N3 and S (A). Taste score plots of N1, N2, N3 and S (B). N1, N2, N3 represented norther combined-baijiu, S indicated southern combined-baijiu. The content of aroma compounds was expressed as the sum of mean value of triplicate samples.

description characteristics of the N1 showed more higher aroma expressions in “sauce” (SV: 3.88), “baked” (SV: 3.57), “Qu” (SV: 2.90), “grain” (SV: 1.42) aromas (Fig. 6A) and “burning” (SV: 3.23) (Fig. 6B) than other three groups. Moreover, the aroma scores of “fruity”, “floral”, “caramel” aromas (Fig. 6A) and “sweet” (Fig. 6B) of N1 were higher than those of the other two group of N2 and N3. In addition, the aroma characteristics of the four groups of combined-Baijiu did not differ much in terms of “ethanol”, “astringent” and “acerbity”. The Sauce-flavored Baijiu was characterized as sauce-like, caramel, baked, and fruity flavors, as well as a rich, full-bodied flavor and lingering aftertaste, and its main style was characterized by a prominent sauce aroma (He et al., 2024). Therefore, the results revealed that N1 was closer to southern combined-Baijiu.

In summary, the style of combined-Baijiu blended according to the N1 (NR1: 5 %, NR2: 35 %, NR3: 30 %, NR4: 20 %, NR5: 10 %) combination ratio was closer to the style of southern combined-Baijiu.

4. Conclusion

This study revealed the differences of flavor characteristics and key compounds between NR and SR. High concentrations of alcohol, acid, and ester compounds were respectively detected of NR and SR. The differences of change rule of the species and contents of aroma compounds, and unique sensory profiles of NR and SR were analyzed to improve the quality of northern Sauced-flavored Baijiu. Moreover, combined with the selection of key flavor compounds and significant difference compounds, the significant differences of 1-propanol, ethyl acetate, ethyl lactate, ethyl propionate, furfural, 1-butanol and 3-methylbutanol in NR and SR were further verified. In addition, the NR3, NR4, SR3 and SR4 had a strong “sauce” and “ethanol” aroma, which was speculated that the formation of these flavor characteristics was associated with 1-propanol, 1-butanol, butyric acid, ethyl caproate and ethyl lactate. Furthermore, the NR1 and SR1 had higher “fruity”, “floral” and “grain” aroma than NR2–5 and SR2–7 and it was speculated that the formation of these flavor characteristics was related to 1-propanol, furfural, octanoic acid, isoamyl acetate and ethyl acetate. At the same time, this study revealed that the potential possibility that the quality of northern and southern Sauce-flavored Baijiu was similar. This research provides some groundwork for studying the flavor characteristics and differences between NR and SR and suggests a fresh idea for the blending ratio of different rounds of northern combined-Baijiu.

Ethical approval

All procedures for sensory evaluation were carried out in accordance with relevant laws and institutional guidelines and were approved by the Scientific Research Academic Committee of Guizhou University.

Informed consent

Informed consent was obtained from all individual participants included in the study.

CRediT authorship contribution statement

Hexia Ding: Writing – review & editing, Writing – original draft, Visualization, Data curation, Conceptualization. **Jiekai Yang:** Supervision, Investigation. **Mai Cheng:** Supervision, Investigation. **Xuanchen Li:** Supervision, Investigation. **Maodie Zeng:** Supervision, Investigation. **Wei Yang:** Supervision, Investigation. **Qian Wu:** Supervision, Investigation. **Xiaoye Luo:** Supervision, Investigation. **Juan Zhao:** Data curation. **Xiande Li:** Supervision, Investigation. **Shuyi Qiu:** Supervision, Investigation. **Jianli Zhou:** Writing – review & editing, Visualization, Supervision, Resources, Conceptualization.

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.

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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.2024.101970.

Data availability

The data that has been used is confidential.

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