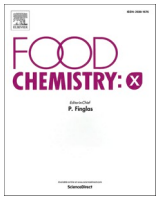
Food Chemistry: X 22 (2024) 101367

Contents lists available at ScienceDirect

Food Chemistry: X

journal homepage: www.sciencedirect.com/journal/food-chemistry-x



# Investigating the impact of various sorghum types on the key aroma compounds of Sichuan Xiaoqu Baijiu through application of the sensomics approach

Yi Ma [a,b,*,1], Ziyun Wei [a,b,1], Xiongjun Xiao [a], Kangjie Yu [a,b], Huiling Huang [a,b], Jianxia Tan [a,b], Yue Wang [a,b], Yong Du [c], Yajun Li [c]

[a] College of Bioengineering, Sichuan University of Science and Engineering, Yibin 644000, China
[b] Engineering Technology Research Center of Special Grain for Wine Making, Yibin 644000, China
[c] Wuliangye Yibin Co., Ltd, China

ARTICLE INFO

*Keywords:*
Baijiu
Sorghum
Sensomics
Key aroma compounds

ABSTRACT:

The aroma of Sichuan Xiaoqu Baijiu (SXB) greatly benefits from the use of sorghum as its primary brewing ingredient. Nevertheless, the impact of different sorghum variety on the primary aroma compounds of SXB has not been thoroughly investigated. Gas chromatography-mass spectrometry (GC-MS) in conjunction with headspace solid phase microextraction (HS-SPME) and liquid-liquid extraction (LLE) were employed in this investigation. Using 5 sorghum varieties as raw materials, five different types of SXB were analysed for their aroma compounds using GC-MS, GC-O, AEDA, aroma recombination, and aroma omission. Key aroma compounds of SXB were successfully identified as ethyl acetate, ethyl 2-methylbutyrate, isoamyl acetate, ethyl hexanoate, ethyl heptanoate, ethyl lactate, ethyl octanoate, ethyl decanoate, phenylethyl acetate, ethyl laurate, ethyl palmitate, isoamyl alcohol, phenylethanol, 1,1-diethoxyethane, 3-hydroxy-2- butanone, furfural, and glacial acetic acid. Glacial acetic acid, ethyl acetate, ethyl lactate, phenylethyl acetate, acetoin, phenylethanol, and ethyl caproate were found to be the seven major aroma compounds that had the biggest impact on the variations of the five SXB aroma properties, according to partial least squares regression (PLS-R) analysis. The collinear network analysis also revealed that the largest positive correlation weight was discovered between the protein and furfural content, tannin content and cereal-like aroma profile while the highest negative correlation weight was found between the moisture and acetoin content. This study is a valuable resource for understanding how raw materials control the directional regulation of the sensory quality of the SXB liquor body.

## 1. Introduction

Sichuan Xiaoqu Baijiu (SXB), a traditional Chinese Baijiu, is esteemed for its elegant aroma. Aroma is a pivotal aspect of Baijiu, influencing consumer acceptance and quality assessment. The brewing climate, technology, and raw materials are the key sources of Baijiu's scent (Qiao, Wang, Wang, Zhang, & Zheng, 2023). Understanding their influence on Baijiu aroma is essential for producing high-quality SXB.

Recent years have witnessed an increasing focus on unraveling the intricate flavor profile of Chinese Baijiu. Open fermentation practices render the Baijiu production process susceptible to environmental microbiological influences. (Pang et al., 2018) illustrated, through an analysis of microbial diversity, that variations in the quantity and flora structure of lactic acid bacteria significantly contribute to the flavor variance in mild-flavor Baijiu. Aging is a pivotal stage in Baijiu production, where physical changes like hydrogen bond association and chemical reactions including redox and esterification processes occur (Jia, Ma, Hu, & Mo, 2023). (Ling, Chen, Xu, & Fan, 2022) employed full two-dimensional gas chromatography-time-of-flight mass spectrometry to examine the volatile components of Gujinggong Baijiu across 42 storage years, highlighting furan, nitrogen, sulfur, lactone, and terpene aroma compounds as primary contributors to scent variations between new and old Baijius. While these studies shed light on the impact of aging technologies and ambient microorganisms on Baijiu aroma, the

* Corresponding author at: College of Bioengineering, Sichuan University of Science and Engineering, Yibin 644000, China.
*E-mail address:* zhangyer2008@suse.edu.cn (Y. Ma).
[1] These authors contributed equally to this work

https://doi.org/10.1016/j.fochx.2024.101367
Received 6 March 2024; Received in revised form 24 March 2024; Accepted 5 April 2024
Available online 8 April 2024

significance of high-quality raw materials in shaping Baijiu aroma remains underexplored. (Guo, Bao, Huang, & Huang, 2018) revealed that variations in sorghum starch structures influence brewing properties, with glutinous sorghum producing higher Baijiu yields due to its greater amylopectin percentage. Additionally, (Sun, Xiong, Du, Qian, & Yao, 2022) investigated the key aroma compounds of two sorghum Xiaoqu Baijiu varieties, finding higher levels of ethyl acetate, ethyl lactate, and organic acids in waxy sorghum Xiaoqu Baijiu compared to japonica sorghum Xiaoqu Baijiu, resulting in stronger grain, sweet, and fruit aromas. (Jiang et al., 2022) summarized the transformation of sorghum proteins into amino acids during fermentation, followed by deamination, decarboxylation, and Maillard reactions, yielding diverse flavoring compounds. While these investigations offer insights into the raw material transformation in Baijiu-making, a comprehensive understanding of their contribution to Baijiu aroma remains elusive.

Current research on Baijiu aroma primarily focuses on characterizing aroma compounds through qualitative and quantitative analyses. Techniques such as GC–MS, gas chromatography-ion mobility spectrometry (GC-IMS), and full two-dimensional gas chromatography-time-of-flight mass spectrometry (GC × GC-TOFMS) have been employed for this purpose. For example, (Zheng et al., 2022) utilized GC × GC-TOFMS combined with solid phase extraction (SPE) to identify around 500 volatile compounds in Wuliangye Baijiu. However, the concentration of volatile compounds alone cannot determine their impact on aroma due to differences in olfactory thresholds (Welke et al., 2021). Sensomics approaches, such as gas chromatography-olfactometry (GC-O), address this issue by considering human olfactory perception. These methods involve sensory evaluation and omission experiments to identify aroma-active compounds contributing significantly to the overall aroma profile. Notably, various industries, including food and beverage, have adopted these approaches extensively (Dach & Schieberle, 2021; Marcq & Schieberle, 2021; Xiao, Luo, Niu, & Wu, 2018). (Hong et al., 2021) identified key aroma compounds in Strong-flavor Baijiu, while (Zhao et al., 2018) successfully screened 9 critical aroma compounds in GJG Baijiu using aroma omission tests. Similarly, (L. Wang et al., 2023) identified 6 key aroma compounds include sotolon, methylthiopropionaldehyde, vanillin, dimethyl trisulfide, and 3-hydroxy-2-butanone in Xiaoqu mild-flavor Baijiu through aroma recombination and omission experiments, shedding light on its distinctive aroma profile. However, research specifically focusing on the application of sensory techniques to characterize essential aroma compounds in SXB or the influence of different sorghum cultivars on these compounds is currently lacking.

Thus, the main content of this study is to characterize the key aroma compounds of SXB brewed by five different varieties of sorghum and explore the influence of sorghum parameter and chemical indexes of sorghum on it: (1) The samples were subjected to pretreatment using LLE and HS-SPME to concentrate the small amounts of aroma compounds present. (2) The volatile substances in the sample were assessed for their qualitative composition using GC–MS, and the FD value of the volatile substances was measured using GC-O/AEDA. (3) The volatile substances with an FD $\geq$ 16 were measured and the OAV value of these substances was calculated. (4) The important aroma compounds in five SXBs were confirmed, selected, and identified using aroma reconstruction and omission studies. (5) A model was created using PLS-R and collinear network analysis to investigate the relationship between the physical and chemical parameter of 5 types of sorghum and the important aroma compounds of SXB.

## 2. Materials and methods

### 2.1. Sorghum and SXB samples

China Xike Agricultural Group procured five sorghum varieties: YNH, SD, XK32, XK35, and XK42, including waxy sorghums (YNH, XK32, XK35, XK42) and japonica sorghum (SD). A total of five SXB test baijiu samples were provided by the Sichuan Brewing Special Grain Engineering Technology Center, all of which complied with Chinese corporate standards and national safety regulations. Among them, four samples were made from Sichuan waxy sorghum (YNH, XK32, XK35, XK42) and one sample was made from Shanxi japonica sorghum (SD). Three replicates of each sample were set up and labeled sequentially (e.g., YNH-1, YNH-2, YNH-3). Alcohol concentration and pH values for the five SXBs are detailed in Table S1 of the supporting information. The production process is depicted in Fig. S1 of the supporting information, and sensory evaluation by a panel of three China national baijiu judges confirmed the representativeness of all samples.

### 2.2. Chemicals

Supporting information Table S2 provides specific information on the source and purity of reference chemicals for qualitative and quantitative purposes. The retention index (RIs) was determined by using C7-C40 n-alkane mixture purchased from Tanmo Quality Inspection TMstandard (Jiangsu, China). Dichloromethane (GC), ethanol (GC), anhydrous sodium sulfate (AR), hydrochloric acid (AR), sulfuric acid (AR), boric acid (AR), potassium sulfate (AR), ammonia (AR), tannic acid (AR), dimethylformamide (AR), ammonium ferric citrate (AR) and sodium hydroxide (AR). Toluene (AR), chloroform (AR), copper sulfate (AR), potassium sodium tartrate (AR), potassium ferrocyanide (AR), glucose (AR) and sodium chloride (AR) were all purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. (Shanghai, China). Bromocresol green indicator, methylene blue indicator and methyl red indicator are all purchased from Beijing Solarbio Technology Co., Ltd. (Beijing, China) and high-purity nitrogen (purity 99.999%) from Dingtai Gas Industry Co., Ltd. (Sichuan, China). All chemicals are analytically pure or higher.

### 2.3. Determination of physical and chemical indexes of sorghum

The properties of five types of sorghum were examined using the procedures described in ("Determination of moisture in foods", 2016), ("Determination of fat in foods", 2016), ("Sorghum - Determination of tannin content", 2008), ("Determination of starch in foods", 2016), and ("Determination of protein in foods", 2016). The measurements for all indicators were taken three times and then averaged. Detailed experimental methods for each parameter are given in the Supplementary Material.

### 2.4. Sensory evaluation panel

Twenty individuals, evenly split between males and females and aged 22 to 26, were chosen from the Sichuan Brewing Special Grain Engineering Technology Research Center for olfactory experiments and quantitative descriptive analysis (QDA). These participants underwent an 8-week training program, with 30 min of daily practice, to enhance their ability to discern aromas in Chinese Baijiu using sniffing bottles from Beijing Micro-standard Technology Co., Ltd., China, and various Baijiu flavors (Dong et al., 2019). Following ("Sensory analysis - General guidance for the selection, training and monitoring of assessors - Part 2: Expert sensory assessors", 2010) for sensory analysis. Panel A, comprising seven members (3 males, 4 females), was formed based on discrimination, repeatability, and overall consistency tests. Additionally, three national-level Baijiu judges (2 males, 1 female) aged 34–41 from the College of Bioengineering, Sichuan University of Science and Engineering, were recruited as Panel B members. All sensory evaluations were conducted in a constant temperature sensory laboratory maintained at 20 °C. It is important to state that in this study, all participants agreed to participate in the sensory study and to use their information, and that ethical permission was not required by this study.

### 2.5. Establishment of sensory profile of SXB

According to ("Sensory analysis–Identification and selection of descriptors for establishing a sensory profile by a multidimensional approach", 1997). Let all the members of the evaluation team brainstorm to give the aroma terms of SXB initially, then screen the descriptors obtained in the discussion, and delete the pleasant sensation, quantitative and irrelevant terms. After preliminary sorting, use a 9-point scale (1–3 weak, 4–6 average, 7–9 strong), and evaluate the intensity of five SXB by using the descriptors obtained above, and calculate the geometric mean "M" value (Formula 1–1) to evaluate the descriptors.

$$M = \sqrt{P \times R} \quad (1\text{-}1)$$

"P" refers to the ratio of the number of times a descriptor is actually mentioned to the total number of times that the descriptor may be mentioned, and "R" represents the ratio of the strength of a descriptor actually given by the evaluation team to the maximum possible strength of the descriptor.

Following the identification of the sensory descriptor, a 9-point scale (ranging from 1 to 3 for weak, 4 to 6 for medium, and 7 to 9 for strong) was employed to assess the reference sample and its intensity of sensory attributes. Ultimately, each team member assessed five SXB samples in accordance with the reference samples. The 20 mL sample was placed in a glass at a temperature of 20 °C. The experiment was conducted three times, with each repetition needing to be finished within 1 h. Each time, the sample was randomly assigned a 3-digit code and presented. Table S3 provides a detailed list of the specific experimental items.

### 2.6. Isolation of aroma compounds

HS-SPME (Chen, Wang, & Xu, 2013): Put 8 ml of 10% vol (diluted in ultrapure water) SXB sample into a 15 ml headspace sampling vial containing 2 g of sodium chloride. The next extraction conditions were as follows: the sample was equilibrated at 45 °C for 40 min, and then 50/30 m divinylbenzone/carboxene/poly (dimension-ylsiloxane) (DVB/car/PDMS) SPME fiber (2 cm, Supelco, Inc., Bellefonte, PA, U.S.A.). After that, SPME fiber was quickly inserted into the injection port, desorbed at 250 °C for 5 min, and then the samples were analyzed by GC–MS and GC-O techniques.

LLE (Sun et al., 2018): 50 mL SXB sample (diluted to 10%vol with ultra-pure water, and saturated with NaCl) was extracted three times (70 mL × 3) with 210 mL of redistilled dichloromethane in a separatory funnel, and each extraction was treated with a shaking table at 400 r/min for 10 min. After the extraction, the organic phase was collected together. The combined extracts underwent dehydration using anhydrous $Na_2SO_4$ for a duration of 1 day, followed by concentration under a gentle stream of nitrogen until reaching a final volume of 500 μL. Subsequently, the concentrated fractions were stored at −20 °C prior to GC-O analysis.

### 2.7. GC–MS and GC-O analysis

The potential aroma active substances in SXB were identified by GC–MS and GC-O techniques. GC–MS analysis was carried out on the Thermofisher TSQ8000 system equipped with a Thermofisher trace1310 GC mass spectrometry detector. For HS-SPME samples, chemical desorption was carried out in a GC injector at 250 °C for 5 min, and for LLE samples, 1 μL was injected at the injection port at 250 °C. The concentrate obtained by LLE and HS-SPME technology was analyzed on DB-WAX (30 m × 0.25 mm i.d., 0.25 μm film thickness; Agilent Technologies, Inc.) and HP-5 (30 m × 0.25 mm i.d., 0.25 μm film thickness, Agilent Technologies, Inc.) capillary chromatographic column, using non-shunt injection mode, carrier gas was helium (99.99%) and constant flow rate was 2 mL/min. The initial temperature of Oven was 40 °C for 3 min, then increased to 80 °C at 5 °C / min, and finally to 230 °C at 7 °C / min for 10 min. Using electron ionization (EI) mass spectrometry mode, the ionization voltage is 70 eV, MS transmission line and the ion source temperature is 260 °C and 280C, respectively. In full scan mode, the scanning range of MS is 30–550 *m/z*.

GC-O analysis: Agilent 8890B GC system equipped with sniffer ports (ODP 4, Gerstel, Germany) and DB-WAX capillary column was used for GC-O analysis. Panel B evaluators put the nose close to the top of the sniffer mouth to record the retention time and odor characteristics of the smelled smell. The heating program of GC-O column incubator was consistent with that of GC–MS analysis, the temperature of sniffer port was set to 250 °C, and the flow rate of humidifier was 50 mL/min. All analyses were repeated twice by each team member.

### 2.8. Aroma extract dilution analysis (AEDA)

The aroma extract of LLE was gradually diluted with redistilled dichloromethane at the ratio of 1:4. For the HS-SPME sample, The sample diluted to 15%voL with ultra-pure water was diluted with ethanol solution of 15%voL according to the ratio of 1:4, 1:16, 1:64, 1:256 and 1:1024. Under the same conditions in Section 2.7 above, each team member analyzed each diluent by GC-O technology until the aroma could not be detected, and the maximum dilution ratio of the aroma substance smelled was recorded as the flavor dilution value (FD). In order to minimize the experimental error, only substances smelled by at least two or more evaluation experts in the same sample are recorded (Wang et al., 2023).

### 2.9. Quantification of aroma compounds

Because there are many types of aroma active compounds in SXB, their structures are different, and the responses to different pretreatment and analysis methods are not the same, in order to obtain more accurate quantitative results, two quantitative methods were used to analyze the aroma substances with FD $\geq$ 16 in SXB. Some strong polar acids, fatty acids and phenols in samples were quantified by LLE combined with GC–MS (Wang et al., 2020). Most of the volatile alcohols, esters and other trace compounds with low boiling point in samples were quantified by HS-SPME combined with GC–MS (Wang et al., 2020).

LLE- GC–MS quantitative analysis (Li et al., 2019): the LLE sample preparation method is the same as Section 2.6, adding 20 μL mixed internal standard (IS1,2-octanol, concentration: 500 mg/L; IS2, 4-(4-methoxyphenyl)-2-butanone, concentration: 500 mg/L) to the 0.5 mL LLE sample. The GC–MS conditions were the same as those for the identification of aroma compounds by GC–MS and GC-O techniques, and each sample was analyzed in triplicate. The reserve solution is prepared by mixing the standard solution of the substance to be quantified and diluted with dichloromethane, which is then diluted with dichloromethane to a series of concentration gradients (2500, 2000, 1000, 800,640,320,160 and 10 mg/L). IS1, IS2 and IS3 were added to the standard solution (the final concentration was 20 mg/L) and analyzed by GC–MS in the same way as the above samples. Taking the ratio of the concentration of the target compound to the concentration of IS as the Abscissa and the ratio of the response of the target compound to the IS response as the ordinate, the standard curve was drawn. The concentration of the target compound in the Baijiu sample was calculated according to the calibration curve. The correction factor is estimated by the response ratio of the target compound to the internal standard compound at the same concentration.

HS-SPME-GC–MS quantitative analysis (Gong, Ma, Li, Cheng, & Huang, 2023): The 8 mL SXB sample was combined with an internal standard (IS1: 2-octanol, 500 mg/L, 10 μL; IS3: amyl acetate, 500 mg/L, 10 μL). The mixture was then diluted to a volume of 15% with ultra-pure water. The resulting solution was added to a 15 mL headspace bottle and saturated with 1.5 g of NaCl. The compound was subsequently extracted using the same HS-SPME technique as described earlier. The samples

were segregated using a DB-WAX column in a non-shunt mode. The experimental conditions for GC–MS are identical to those mentioned earlier.

The detection limit (LOD) and quantitation limit (LOQ) were defined as the minimum concentrations on the calibration curve where the signal-to-noise ratio reached 3 and 10, respectively. All analyses were conducted in triplicate. Intraday precision was assessed by performing repeated analyses of each compound at a concentration of 1 mg/L (three replicates in a single day). Interday precision was evaluated by repeating the intraday precision analysis on three separate days, with all analyses repeated three times. The precision was calculated using the mean, standard deviation, and relative standard deviation (%) of these values. Detailed information is provided in Table S4.

### 2.10. Determination of odor threshold and calculation of OAV value

In order to calculate the OAV values of the aroma active substances, the olfactory thresholds of each aroma active substance need to be clarified. The odor thresholds of most of the aroma-active substances in this study were obtained from published literature references (measured in ethanol solution or aqueous solution), and for some aroma-active compounds for which the reference thresholds were not found, the olfactory thresholds were determined using the three-alternative forced-choice test according to (Guidelines for threshold determination of Baijiu flavor substances, 2016) and the previously described method (Czerny et al., 2008). The OAV of each substance was calculated as the compound concentration divided by its odor threshold. Compounds with OAV $\geq 1$ are considered significant contributors to SXB's aroma characteristics.

### 2.11. Aroma recombination and aroma omission

Important aroma substances with FD not <16 and OAV not <1 in SXB were selected for aroma reconstruction experiment (Trujillo et al., 2022). The complete recombination model took the mixed solution of water and alcohol of 52%vol as the matrix, added and reconstructed according to the actual concentration of aroma substances, and then balanced at room temperature for 10 min. The sensory differences between the five SXB samples and the complete recombination model were analyzed.

In order to further identify the key aroma substances in three SXB, several aroma loss models were constructed by omitting one or a group of aroma substances selected from the complete recombination model. The deletion experiment was carried out by trigonometric test. The three samples, including two complete recombination models and one aroma loss model, were identified by random three-digit codes and sniffed by panel A and panel B, and the evaluators were asked to identify samples with different odors and record the corresponding numbers. According to the method described (Zheng et al., 2016), the significant difference of aroma between complete reconstruction model and aroma loss model was calculated.

### 2.12. Statistical analysis

The graphics are drawn using Origin Pro 21.0. All significant differences were analyzed using SPSS22.0 (SPSS Inc., Chicago, Illinois) (Duncan test). XLSTAT 2019 (Addin soft, New York, NY) was used for partial least square regression (PLS-R) analysis. The similarity between the original sample and its corresponding complete recombination model is analyzed by Pearson double-tailed test. The co-occurrence network is analyzed based on Spearman correlation coefficient (Spearman $|r| > 0.7$ or $r < -0.7$, $p < 0.01$) and visualized by Gephi (v 0.9.2).

## 3. Results and discussion

### 3.1. Sorghum parameter analysis

Water plays a pivotal role in Baijiu production, with excess moisture increasing the risk of mildew formation and impacting Baijiu quality significantly (Feng et al., 2017). Fig. 1a displays the moisture of five sorghum varieties falling within typical ranges, with SD exhibiting the highest content at 13.48% and YNH the lowest at 10.86%, meeting storage specifications outlined in ("Sorghum", 2007). Microbial degradation of fat during production yields unsaturated fatty acids, aldehydes, and ketones, enhancing flavor, while excessive fat leads to rancid odor development (Niu et al., 2023), as shown in Fig. 1b. SD exhibits significantly higher fat content (7.89%) compared to other samples ($p < 0.05$), with XK32 having the lowest (3.06%). Tannin concentration significantly affects microbial growth and Baijiu production yield (Ji, Wang, & Guo, 2019). Fig. 1c indicates that sample YNH has notably higher tannin content compared to others, with SD being the lowest (1.63% vs. 1.15%). Starch, crucial for alcohol fermentation and sensory attributes, is divided into amylose and amylopectin (Wu et al., 2007). Amylose, less conducive to Baijiu production due to limited water absorption, is highest in SD and lowest in XK42 (Li, Hu, Huang, Gong, & Yu, 2020), as depicted in Fig. 1d. Conversely, amylopectin, promoting microbial proliferation and flavor development, is highest in XK42 and lowest in SD (Zhao et al., 2021), as shown in Fig. 1e. Raw material protein content contributes to Baijiu's distinctive flavor through fermentation, with YNH exhibiting the highest protein content (7.75%) and XK35 the lowest (5.87%) (Luo et al., 2011), as illustrated in Fig. 1f.

### 3.2. Establishment of SXB sensory profile

Based on the collective brainstorming findings of the team members and the ranking using the geometric mean M value calculation, seven sensory descriptors with $M > 0.7$ were chosen: fruity, sweet, floral, alcoholic, fermented grain-like, acidic, and cereal-like. These descriptors were used to create the sensory profile of SXB aroma, as displayed in Table S5. The establishment and strength of the reference sample are shown in Table S6.

Figure 2a displays the sensory profile analysis of the five SXB. In general, the five SXB samples are quite different. Among the five samples, YNH exhibited the most pronounced notes of fruit, grain, sourness, fermentation, and the least prominent alcohol flavor. On the other hand, SD displayed a reasonable level of sweetness and fermentation aroma, with lower intensity in fruit, grain, sourness, and floral aroma, but the highest intensity of alcohol flavor. XK32 exhibited the lowest level of sweetness, although demonstrated elevated floral and sour fragrance. XK35 exhibited a greater level of sweetness, but a lower level of fermented aroma, sour aroma, and grain aroma. On the other hand, XK42 had moderate overall strength of olfactory characteristics. The volatile components in five SXB samples were analyzed using AEDA to elucidate the reasons contributing to the variation in scent.

### 3.3. Results of GC–MS and GC-O

To comprehensively analyze the SXB aroma profile, two complementary pretreatment methods, HS-SPME and LLE, were utilized to extract volatile compounds. This approach addresses the inefficiency of a single pretreatment method in enriching all volatile compounds in Baijiu. The concentrated LLE extract, when applied to filter paper for sensory evaluation, demonstrates SXB's distinctive scent traits, affirming LLE's reliability in extracting volatile components. GC-O technology detects volatile compounds related to SXB, while AEDA verifies their FD values. Fig. S2 illustrates total ion chromatograms of volatile chemicals in five SXB samples, with compound detection and verification conducted using RI, aroma description, MS library comparison, and standard sample confirmation.

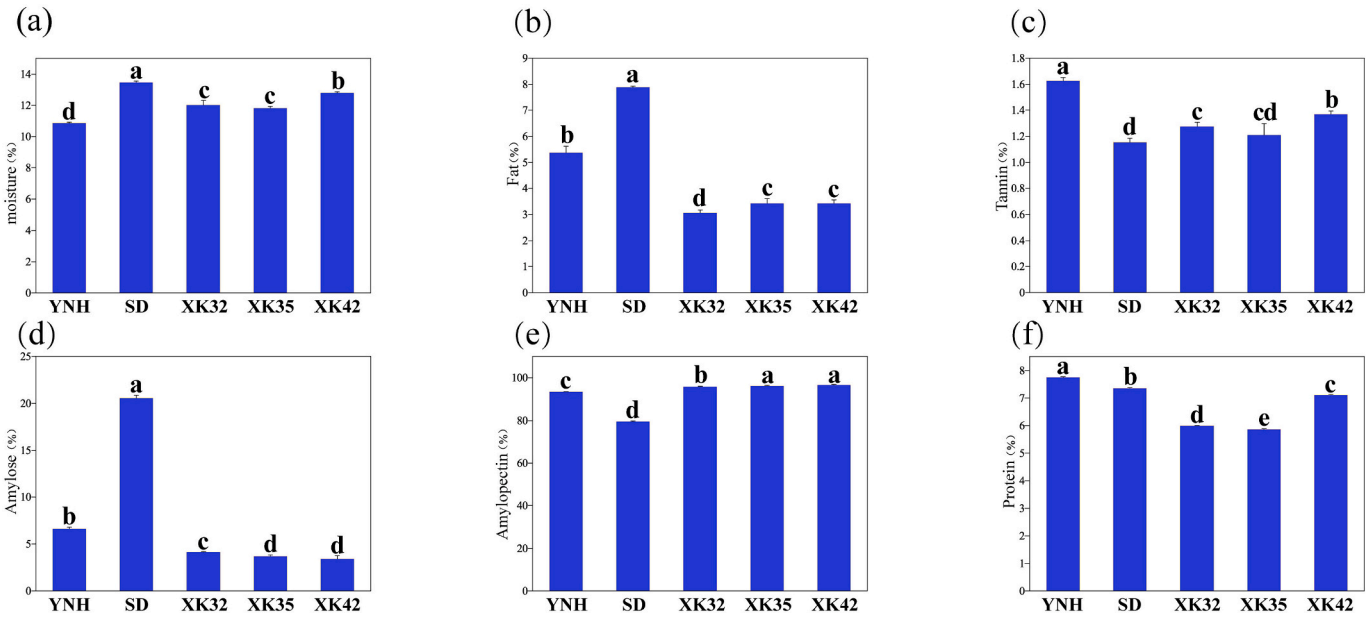



**Fig. 1.** Basic physical and chemical parameter of five kinds of sorghum. (a) Moisture content of sorghums. (b) Fat content of sorghums. (c) Tannin content of sorghums. (d) Amylose content of sorghums. (e) Amylopectin content of sorghums. (f) Protein content of sorghums.

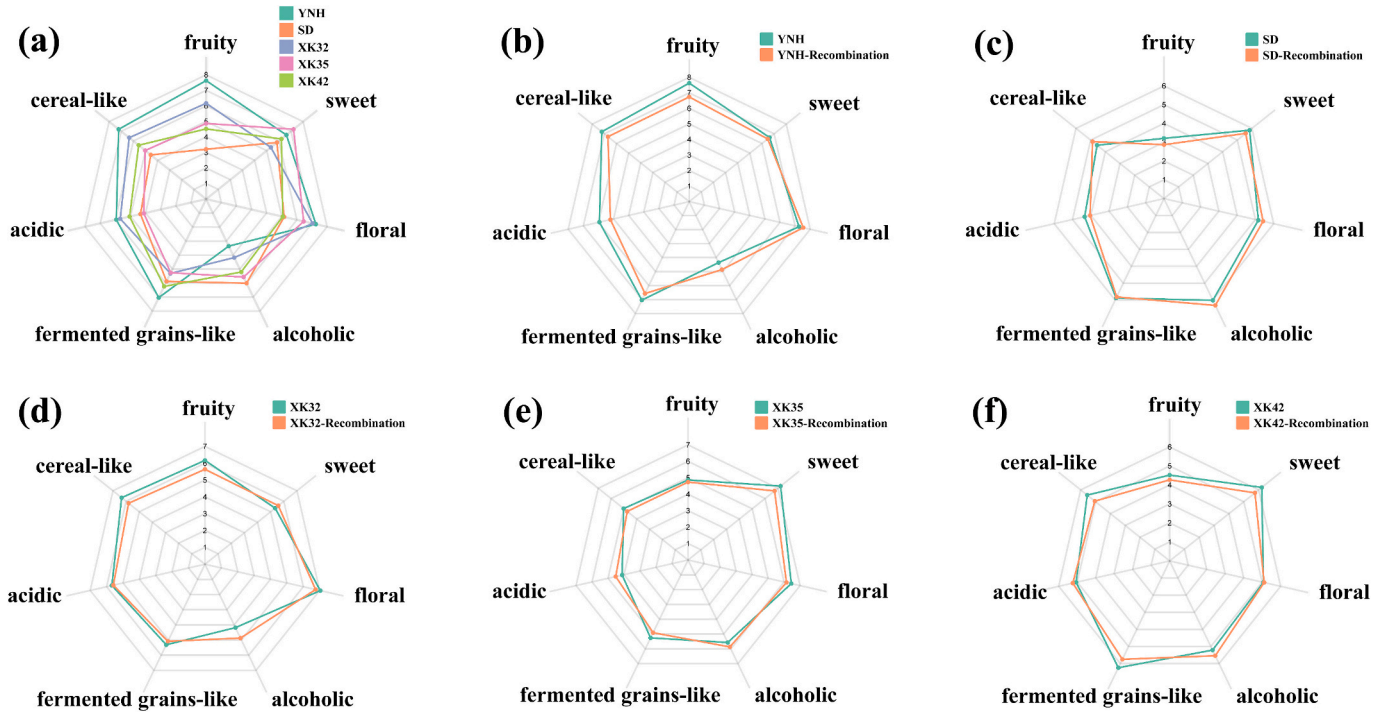



**Fig. 2.** Sensory profiles of five kinds of SXB. (a) Sensory profiles of five kinds of SXB. (b) Aroma recombination model of YNH. (c) Aroma recombination model of SD. (d) Aroma recombination model of XK32. (e) Aroma recombination model of XK35. (f) Aroma recombination model of XK42.

As depicted in Fig. 3a, 87 volatile compounds were detected in the five SXB samples, including esters, alcohols, aldehydes, ketones, acids, phenols, olefins, and other compounds. Among these, 47, 38, 48, 36, and 37 compounds with FD $\geq$ 4 were found in the five SXB samples, respectively, potentially contributing to SXB's aroma characteristics. Notably, differences exist in the variety distribution of these compounds among samples. YNH and XK32 exhibited more esters and alcohols with FD $\geq$ 4, while XK32 had the most ketones and olefins with FD $\geq$ 4. YNH had the most aldehydes and phenols, whereas SD had the most acids. XK35 and XK42 displayed a low number of many compounds but had more acetals, lactones, and other compounds. However, variations in aroma compound distribution alone do not fully explain the differences in SXB aroma properties.

Table S7 details aroma compound identification, FD values, and sample pretreatment methods for the five SXB types. Comparison of FDs revealed similar skeleton aroma compounds (FD $\geq$ 256) across all samples. Some compounds, such as isoamyl alcohol, ethyl lactate, ethyl octanoate, and phenylethanol, had the highest FDs, contributing to shared aroma characteristics. Additionally, differentiated important aroma compounds (FD $\geq$ 16) were identified in all SXB samples, such as

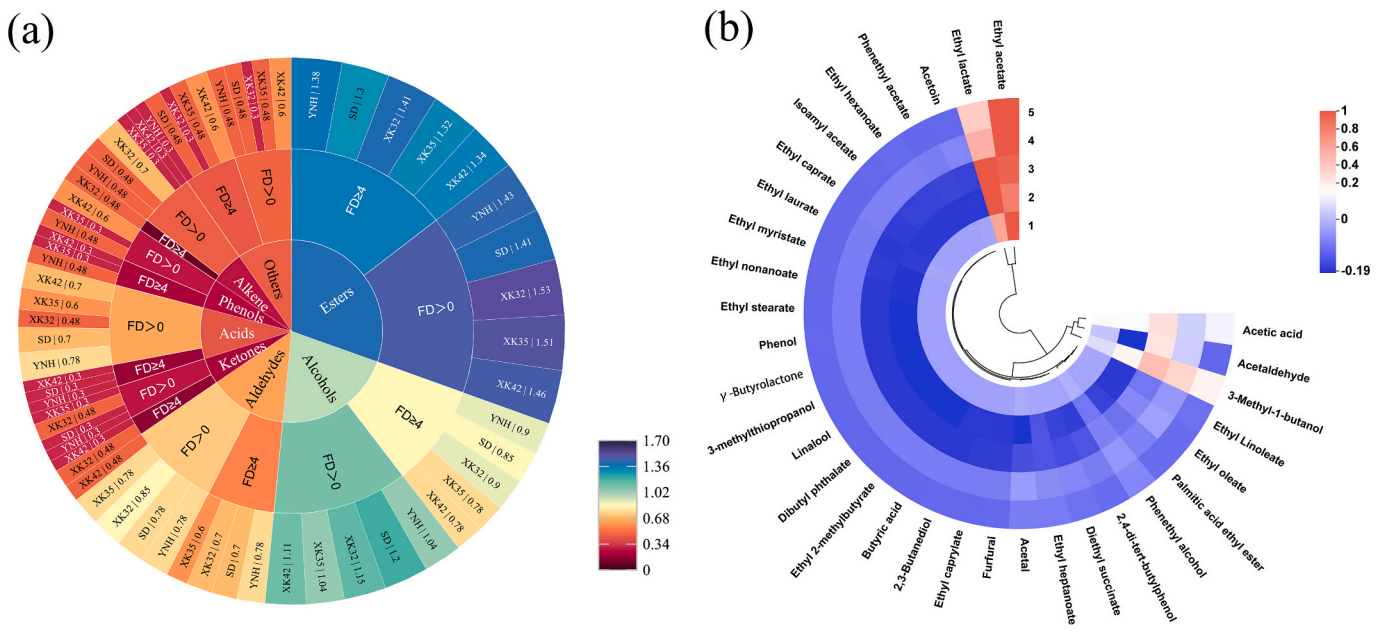



**Fig. 3.** (a) FD distribution of volatile compounds in five SXB samples. (b) 5 Concentration distributions of 32 aroma active compounds in 5 kinds of SXB.

acetal and isoamyl acetate et al. Terpenes detected in only a few samples, like β-caryophyllene, δ-juniene, and cedarene, likely contribute to differences in SXB aroma.

### 3.4. Quantification of aroma compounds

All 32 important aroma active compounds with FDs greater than or equal to 16 in five SXB samples were further quantified to clarify the differences in sensory profiles among the five SXB and to explore the contribution of aroma compounds to the typical aroma characteristics of SXB. Table 1 lists the internal standard, standard curve, R2, content and quantitative method of five SXB aroma compounds. As shown in Fig. 3b, the concentrations of ethyl acetate (1319 mg/L, range: 3038 mg/L) and ethyl lactate (894 mg/L, range: 468–1897 mg/L) were the highest among the five SXB, followed by isoamyl alcohol (320 mg/L, range: 192–517 mg/L) and glacial acetic acid (245 mg/L, range: 150–475 mg/L), and linalool was the lowest (0.026 mg/L, range: 0.026–0.027 mg/L). And only detected in YNH and XK32). Compared with the previous research results (Hong et al., 2021; Sun et al., 2022; L Wang, Kan, et al., 2023; Z. Wang et al., 2021), the concentration of glacial acetic acid is generally lower than that of other typical types of Mild-flavor Baijiu, while the concentration of ethyl lactate, ethyl acetate and isoamyl alcohol is close to that of traditional Mild-flavor Baijiu, which may be one of the reasons why the typical aroma characteristics of SXB are different from other Mild-flavor Baijius. In addition, the concentrations of aroma active compounds of five kinds of SXB were also different. YNH has the highest concentration of aroma active compounds (6371 mg/L), and the highest concentrations of esters (5124 mg/L), ketones (197 mg/L) and acids (476 mg/L). It also shows a high level in the concentration of other aroma active compounds. SD has the lowest concentration of aroma active compounds (1313 mg/L), but has a higher proportion of esters (74.22%), second only to YNH (80.43%). XK32 has the highest concentrations of aldehydes (251 mg/L) and phenols (25 mg/L). It is worth noting that XK32 has the lowest proportion of esters (59.42%). And the highest concentration of aldehydes (9.44%) and acids (9.83%), indicating that the aroma configuration of XK32 is quite different from that of other samples, which may also be one of the reasons why the aroma properties of XK32 are different from other samples. XK35 has the highest concentration of alcohols (564 mg/L) and higher concentrations of esters (2307 mg/L) and ketones (152 mg/L), second only to YNH, while XK42 has the lowest concentration of aldehydes (4 mg/L), and the concentrations of other aroma active compounds are also lower. On the whole, the five SXB aroma compounds all showed the content distribution characteristics of ethyl esters as the main aroma compounds, and esters usually showed pleasant tropical fruit and flower aromas, which could well explain the elegant and comfortable fruit and flower aromas in the SXB sensory profile.

### 3.5. OAV analysis

In order to further clarify the contribution of aroma compounds to the typical aroma of SXB, OAV value (the ratio of concentration to aroma threshold) is used to quantify and evaluate the aroma contribution of each aroma compound. It is generally considered that compounds with OAV $\geq$ 1 are important aroma compounds. As shown in Fig. 4a, there are 18 kinds of compounds with OAV $\geq$ 1, including 11 esters, 2 alcohols, 1 aldehyde, 1 acid and 3 others. The same compounds show different OAV values in different SXB, which may be due to the interaction between aroma compounds and volatile and non-volatile compounds in different substrates, resulting in different addition or inhibition of the contribution of aroma compounds, which can to some extent explain that although the five kinds of SXB have similar important aroma compounds, the aroma properties are quite different. The OAV values of ethyl acetate, isoamyl acetate, acetoin, ethyl lactate, furfural, ethyl Laurate, phenylethanol and glacial acetic acid in YNH are the highest among the five SXB samples. Ethyl heptanate in SD has the highest OAV value among five SXB samples. Acetaldehyde, ethyl caprylate, ethyl decanoate, phenylethyl acetate and ethyl palmitate in XK32 have the highest OAV values among the five SXB samples. The acetal, ethyl 2-methyl butyrate, isoamyl alcohol and ethyl caproate of XK35 species have the highest OAV values among the five SXB samples, while XK42 is at a lower level in most of the important aroma compounds OAV.

Although ethyl acetate (average concentration 1319 mg/L) and ethyl lactate (average concentration 894 mg/L) have very high concentrations, their OAV (average OAV:41 of ethyl acetate and average OAV:7 of ethyl lactate) is not high, while acetoin (22, cream, OAV range: 119–759), ethyl octanoate (29, floral aroma, OAV range: 125–337) and ethyl caproate (20, tropical fruits) OAV range: 95–180) there are high OAV values in all samples, which means that they may be the most

**Table 1**
Standard curve and concentration of 32 odor active components in 5 kinds of SXB.

| No.[a] | Compounds | IS[b] | Slope | Intercept | $R^2$ | Concentration (mg/L)[c] | | | | | Quantitative methods | Odor threshold (mg/L)[d] | OAV[i] | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | YNH | SD | XK32 | XK35 | XK42 | | | 1 | 2 | 3 | 4 | 5 |
| 1 | Acetaldehyde | IS1 | 1.9733 | −0.3533 | 0.9999 | 153.11 ± 15.4[b] | – | 247.67 ± 19.2[a] | 144.56 ± 11.2[b] | – | H-SPME-GC–MS | 100[e] | 1.53 | – | 2.48 | 1.45 | – |
| 4 | Acetal | IS2 | 2.4266 | −0.3877 | 0.9998 | 54.62 ± 2.3[a] | – | 20.71 ± 1.4[c] | 54.85 ± 2.1[a] | 26.37 ± 1.1[b] | H-SPME-GC–MS | 2.086[e] | 26.18 | – | 9.93 | 26.29 | 12.64 |
| 5 | Ethyl acetate | IS3 | 1.451 | −0.588 | 0.9975 | 3037.53 ±124[a] | 554.05 ± 35.89[d] | 642.62 ± 41.23[d] | 1335.61 ± 102.3[b] | 1024.86 ± 84.36[c] | H-SPME-GC–MS | 32.55[e] | 93.46 | 17.05 | 19.77 | 41.10 | 31.53 |
| 8 | 2,3-butanedione | IS2 | 0.6651 | 0.1707 | 0.9982 | 13.81 ± 1.02[a] | 2.99 ± 1.35[a] | 0.25 ± 0.06[c] | 1.55 ± 0.24[c] | 4.58 ± 0.94[b] | LLE-GC–MS | 32.612[e] | 0.42 | 0.09 | 0.01 | 0.05 | 0.14 |
| 13 | Ethyl 2-methylbutyrate | IS2 | 0.5549 | 0.04322 | 0.9997 | – | – | 0.19 ± 0.02[b] | 0.31 ± 0.03[a] | – | H-SPME-GC–MS | 0.018[e] | – | – | 10.39 | 17.37 | – |
| 16 | Isoamyl acetate | IS3 | 0.87476 | −0.01976 | 0.9995 | 10.48 ± 1.2[a] | 6.2 ± 1.8[b] | 7.89 ± 1.6[ab] | 8.34 ± 1.7[ab] | 0.86 ± 0.07[c] | H-SPME-GC–MS | 0.094[e] | 111.44 | 65.92 | 83.93 | 88.72 | 9.19 |
| 18 | 3-methyl-1-butanol | IS3 | 0.2507 | −0.1025 | 0.9995 | 258.43 ± 23.4[c] | 191.76 ± 12.7[d] | 371.24 ± 27.3[b] | 517.04 ± 42.3[a] | 263.24 ± 12.4[c] | H-SPME-GC–MS | 179.2[e] | 1.44 | 1.07 | 2.07 | 2.89 | 1.47 |
| 20 | Ethyl hexanoate | IS2 | 0.603 | −0.674 | 0.9994 | 9.46 ± 1.4[a] | 5.93 ± 0.8[b] | 8.54 ± 1.1[a] | 9.91 ± 1.5[a] | 5.24 ± 0.31[b] | H-SPME-GC–MS | 0.055[e] | 171.92 | 107.75 | 155.31 | 180.26 | 95.36 |
| 22 | Acetoin | IS3 | 0.9508 | 0.1757 | 0.9983 | 19.66 ± 1.53[a] | 3.08 ± 0.18[d] | 8.55 ± 0.42[c] | 15.22 ± 1.03[b] | 4.05 ± 0.24[d] | H-SPME-GC–MS | 0.259[e] | 759.00 | 118.82 | 330.30 | 587.77 | 156.55 |
| 24 | Ethyl heptanoate | IS2 | 0.9928 | −0.5895 | 0.9991 | 26.56 ± 1.2[ab] | 27.43 ± 1.3[a] | 24.68 ± 1.2[bc] | 24.3±1[c] | 25.18 ± 0.91[bc] | H-SPME-GC–MS | 13.153[e] | 2.02 | 2.09 | 1.88 | 1.85 | 1.91 |
| 25 | Ethyl lactate | IS3 | 1.9177 | −0.01939 | 0.9992 | 1897.08 ± 153.2[a] | 652.23 ± 54.3[b] | 676.6 ± 31.2[b] | 774.93 ± 36.6[b] | 468.33 ± 13.8[c] | LLE-GC–MS | 128[e] | 14.82 | 5.10 | 5.29 | 6.05 | 3.66 |
| 29 | Ethyl caprylate | IS3 | 0.892 | −0.2526 | 0.9983 | 3.13 ± 0.26[b] | 4.2 ± 0.35[a] | 4.34 ± 0.24[a] | 4.14 ± 0.18[a] | 1.61 ± 0.11[c] | H-SPME-GC–MS | 0.01287[e] | 243.22 | 326.43 | 336.90 | 321.64 | 125.24 |
| 32 | Furfural | IS1 | 0.697 | −0.5421 | 0.9943 | 6.09 ± 0.24[a] | 5.28 ± 0.32[b] | 3.32 ± 0.12[d] | 3.18 ± 0.18[d] | 4.32 ± 0.15[c] | LLE-GC–MS | 0.122[e] | 49.91 | 43.25 | 27.23 | 26.03 | 35.40 |
| 35 | Ethyl nonanoate | IS3 | 0.685 | −0.2544 | 0.9947 | – | 1.58 ± 0.07[a] | 1.65 ± 0.09[a] | 1.71 ± 0.14[a] | – | H-SPME-GC–MS | 3.151[e] | – | 0.50 | 0.53 | 0.54 | – |
| 38 | Linalool | IS2 | 0.9441 | −0.04807 | 0.9962 | 0.03 ± 0.002[a] | – | 0.03 ± 0.001[a] | – | – | LLE-GC–MS | 0.03[e] | 0.88 | – | 0.85 | – | – |
| 41 | γ- butyrolactone | IS1 | 1.7082 | −0.05439 | 0.9944 | 0.47 ± 0.02[b] | 0.23 ± 0.02[c] | 0.17 ± 0.01[d] | 0.55 ± 0.03[a] | 0.49 ± 0.03b | LLE-GC–MS | 564.817[e] | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 43 | Ethyl caprate | IS3 | 0.648 | −0.08335 | 0.9961 | 3.34 ± 0.12[c] | 5.94 ± 0.18[ab] | 6.34 ± 0.24[a] | 5.71 ± 0.37[b] | 3.14 ± 0.14[c] | H-SPME-GC–MS | 1.1223[f] | 2.98 | 5.29 | 5.65 | 5.09 | 2.79 |
| 46 | Butyric acid | IS3 | 0.9047 | −0.3399 | 0.9975 | 0.53 ± 0.03[a] | – | 0.26 ± 0.01[c] | 0.29 ± 0.02[b] | 0.21 ± 0.01[d] | LLE-GC–MS | 1[f] | 0.53 | – | 0.26 | 0.29 | 0.21 |
| 53 | Diethyl succinate | IS1 | 0.278 | −0.9447 | 0.9991 | 24.19 ± 1.5[a] | 14.54 ± 0.9[c] | 16.62 ± 0.98[b] | 15.46 ± 1.02[bc] | 14.19 ± 0.72[c] | H-SPME-GC–MS | 353.19325[e] | 0.07 | 0.04 | 0.05 | 0.04 | 0.04 |
| 55 | 3-methylthiopropanol | IS2 | 0.9266 | −0.3877 | 0.9998 | 0.52 ± 0.02[a] | 0.21 ± 0.01[c] | – | 0.26 ± 0.02[b] | 0.2 ± 0.01[c] | LLE-GC–MS | 2.11[f] | 0.25 | 0.10 | – | 0.12 | 0.09 |
| 58 | Phenethyl acetate | IS1 | 0.605 | −0.06576 | 0.9997 | 10.69 ± 1.2[b] | 5.03 ± 0.32[c] | 12.43 ± 1.1[a] | 3.35 ± 0.24[d] | 2.64 ± 0.12[d] | H-SPME-GC–MS | 0.407[f] | 26.26 | 12.35 | 30.54 | 8.24 | 6.49 |
| 60 | Ethyl laurate | IS1 | 0.837 | −0.0921 | 0.9997 | 19.08 ± 1.1[a] | 10.07 ± 0.84[b] | 0.51 ± 0.03[d] | 3.14 ± 0.15[c] | 2.09 ± 0.15[c] | H-SPME-GC–MS | 0.4[e] | 47.71 | 25.19 | 1.27 | 7.84 | 5.24 |
| 61 | Phenethyl alcohol | IS1 | 0.971 | −0.817 | 0.9998 | 69.43 ± 5.2[a] | 55.78 ± 3.2[b] | 63.88 ± 4.9[a] | 44.88 ± 2.6[c] | 41.14 ± 3.1[c] | H-SPME-GC–MS | 28.9[e] | 2.40 | 1.93 | 2.21 | 1.55 | 1.42 |
| 63 | Phenol | IS1 | 0.378 | −0.6097 | 0.9996 | 1.2 ± 0.09[a] | – | – | 1.08 ± 0.1[b] | 0.84 ± 0.05[c] | LLE-GC–MS | 18.9[g] | 0.06 | – | – | 0.06 | 0.04 |
| 65 | Acetic acid | IS1 | 0.03641 | −0.09309 | 0.9959 | 475.16 ± 21.3[a] | 158.86 ± 8.5[cd] | 261.14 ± 14.9[b] | 150.09 ± 8.3[d] | 179.45 ± 8.9[c] | LLE-GC–MS | 160[h] | 2.97 | 0.99 | 1.63 | 0.94 | 1.12 |
| 66 | Ethyl myristate | IS1 | 0.2152 | −0.05172 | 0.9998 | 0.89 ± 0.03[b] | 0.97 ± 0.05[b] | 4.41 ± 0.34[a] | 0.35 ± 0.02[c] | 0.27 ± 0.01[c] | H-SPME-GC–MS | 46.606[g] | 0.02 | 0.02 | 0.09 | 0.01 | 0.01 |

(*continued on next page*)

**Table 1** (*continued*)

| No.[a] | Compounds | IS[b] | Slope | Intercept | $R^2$ | Concentration (mg/L)[c] | | | | | Quantitative methods | Odor threshold (mg/L)[d] | OAV[i] | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | YNH | SD | XK32 | XK35 | XK42 | | | 1 | 2 | 3 | 4 | 5 |
| 74 | 2,4-di-tert-butylphenol | IS1 | 0.536 | −0.273 | 0.9992 | 16.97 ± 1.2[b] | 10.04 ± 1.1[c] | 24.95 ± 1.8[a] | 3.39 ± 0.21[e] | 6.37 ± 0.42[d] | LLE-GC–MS | 34.847[h] | 0.49 | 0.29 | 0.72 | 0.10 | 0.18 |
| 75 | Palmitic acid ethyl ester | IS1 | 0.603 | −0.674 | 0.9994 | 16.27 ± 1.1[b] | 13.52 ± 0.97[c] | 50.95 ± 2.8[a] | 7.68 ± 0.34[d] | 6.3 ± 0.42[d] | H-SPME-GC–MS | 39.299[g] | 0.41 | 0.34 | 1.30 | 0.20 | 0.16 |
| 79 | Ethyl oleate | IS3 | 0.642 | −0.655 | 0.9993 | 33.99 ± 1.9[b] | 5.17 ± 0.28[c] | 67.87 ± 4.8[a] | 62.95 ± 3.9[a] | 9.97 ± 2.8[c] | H-SPME-GC–MS | 130[g] | 0.26 | 0.04 | 0.52 | 0.48 | 0.08 |
| 82 | Ethyl linoleate | IS3 | 0.708 | −0.8841 | 0.9993 | 31.07 ± 1.6[c] | 5.85 ± 0.26[e] | 53.24 ± 4.1[a] | 48.29 ± 2.9[b] | 12.09 ± 0.82[d] | H-SPME-GC–MS | 57.21[h] | 0.54 | 0.10 | 0.93 | 0.84 | 0.21 |
| 83 | Ethyl stearate | IS3 | 0.929 | −0.0484 | 0.9989 | 0.55 ± 0.019[c] | 0.13 ± 0.011[d] | 1.08 ± 0.09[a] | 0.89 ± 0.05[b] | 0.1 ± 0.01[d] | LLE-GC–MS | 4.373[h] | 0.13 | 0.03 | 0.25 | 0.20 | 0.02 |
| 85 | Dibutyl phthalate | IS1 | 0.94845 | −0.9229 | 0.9999 | 0.08 ± 0.008[a] | – | – | 0.05 ± 0.003[b] | 0.04 ± 0.003[c] | LLE-GC–MS | 0.9485[h] | 0.08 | – | – | 0.05 | 0.04 |

[a] Numbers were the same as numbers listed in Table S7.
[b] Quantitative internal standard number.
[c] The concentrations of aroma compounds were represented as mean value of triplicate sample The concentrations of aroma compounds were represented as mean value of triplicate samples ± standard deviation (mean ± SD).
[d] Odor thresholds were taken from reference.
[e] Odor thresholds taken from reference (Z. Wang, Wang, et al., 2021).
[f] Odor thresholds taken from reference (L. Wang, Kan, et al., 2023).
[g] Odor thresholds taken from reference (J. Wang, Ming, et al., 2021).
[h] Odor threshold was measured by three-alternative forced-choice test (3-AFC).
[i] OAVs were calculated by dividing the concentration by the respective odor threshold.

important aroma compounds in SXB. (L. Wang, Kan, et al., 2023) found that ethyl caprylic acid had the second highest OAV value in young Mild-flavor Baijiu, and ethyl caprylic acid had the highest OAV value in Fen Baijiu, Baofeng Baijiu and Caoyuanwang Baijiu (all typical Mild-flavor Baijiu) (Gao, Fan, & Xu, 2014). (J. Wang et al., 2021) found that acetoin had the third highest OAV value in Prairie King Baijiu, and showed higher OAV value in many other typical Mild-flavor Baijius (Sun et al., 2022). Ethyl hexanoate, as the classical key aroma compound of Strong-flavor Baijiu (Hong et al., 2021), also has high OAV values in many typical Mild-flavor Baijius, but all of them are lower than those in SXB, and they all show strong fruity aroma (Z. Wang, Wang, et al., 2021), which shows that ethyl hexanoate also has an important contribution to the aroma characteristics of Mild-flavor Baijiu. Although the content of isoamyl acetate is not high (average 6.75 mg/L), because of its low threshold (0.094 mg/L) and large OAV value (16, banana flavor, OAV range: 9–111), SXB has a pleasant fruit flavor. Furfural (32, baked potato, OAV range: 26–50), ethyl acetate (5, fruit aroma, OAV range: 17–93) and acetal (4, cream, OAV range: 10–26) have higher OAV in five kinds of SXB, so they make a contribution to the composition of SXB aroma that can not be ignored. Furfural is mainly produced by hydrolysis of pentoses from raw materials of Baijiu production to pentalose during distillation and further dehydration and cyclization of pentalose (Zhao et al., 2008). Ethyl Laurate is also the key aroma compounds in Fen Baijiu, Baofeng Baijiu, highland barley Baijiu and CaoyuanwangBaijiu.

Overall, 14 odorants with OAV $\geq$ 1 were present in all five SXB samples, namely, ethyl caprylate, ethyl hexanoate, isoamyl acetate, ethyl acetate, 3-hydroxy-2-butanone, furfural, ethyl laurate, phenethyl acetate, ethyl lactate, ethyl caprylate, ethyl heptanoate, phenylethanol, isoamyl alcohol, and glacial acetic acid, and additionally 4 aroma compounds with OAV $\geq$ 1 in some samples,. They were acetaldehyde (YNH:1; XK32:2; XK35:1), acetal (YNH:26, XK32:10, XK35:26, XK42:13), ethyl 2-methylbutyrate (XK32:10, XK35:17), and ethyl palmitate (XK32:1), which may be one of the causes of the differences in the aroma profiles of the five SXBs, and are therefore also considered as important aroma compounds.

### 3.6. Aroma recombination

Aroma recombination experiments were used to verify the criticality of important aroma compounds identified by AEDA and OAV analysis. The recombination model was constructed by mixing 18 compounds with OAV $\geq$ 1 into 52% ethanol solution, and comparing the aroma characteristics of the mixed solution with that of the original sample. As shown in Fig. 2b-f, The seven observed aroma profiles had slightly different aroma intensities between the reconstituted model and the original sample. Although Panel B can distinguish between real Baijiu samples and these models, their aromas are considered to be very similar in quality. This result confirmed that the aroma recombination model was successful in the characterization of SXB.

### 3.7. Aroma omission

In order to further screen and verify the contribution of some types of aroma compounds to the aroma characteristics of SXB, the aroma omission was carried out. In the absence of a single compound or a group of aroma compounds, a total of 94 aroma omission models were established. In addition, the trigonometric test is used to compare the omission model with the complete recombination model (with all the aroma-active volatile chemicals). As shown in Table 2, it was found that 15 ellipsis models showed significant differences ($p < 0.05$) in aroma characteristics, 29 ellipsis models showed highly significant differences in aroma characteristics ($p < 0.01$), 29 ellipsis models showed very highly significant differences ($p < 0.001$), and 45 ellipsis models showed no significant differences in aroma characteristics.

The results showed that esters had an important effect on the overall

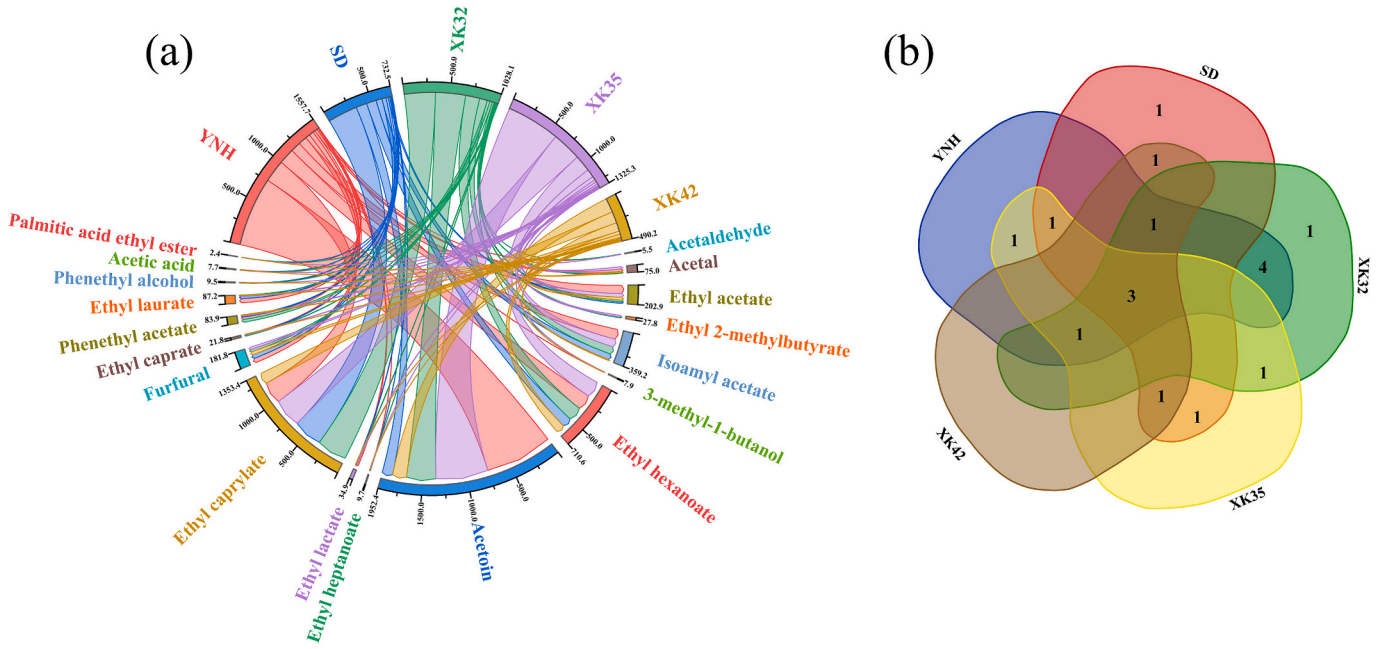



**Fig. 4.** (a) String diagram of 18 important aroma compounds. (b) Wayne diagram of key aroma compounds.

**Table 2**
Omission experiments from the complete model mixture.

| No. | Aroma compounds omitted from the recombinant model | N[a] | | | | |
|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 |
| 1 | All esters | 10*** | 10*** | 10*** | 10*** | 10*** |
| 1–5 | Ethyl acetate | 10*** | 9** | 10*** | 9** | 9** |
| 1–13 | Ethyl 2-methylbutyrate | – | – | 8** | 7* | – |
| 1–16 | Isoamyl acetate | 8** | 6 | 7* | 6 | 5 |
| 1–20 | Ethyl hexanoate | 7* | 6 | 7* | 5 | 6 |
| 1–24 | Ethyl heptanoate | 8** | 7* | 6 | 7* | 6 |
| 1–25 | Ethyl lactate | 6 | 7* | 6 | 5 | 4 |
| 1–29 | Ethyl caprylate | 9** | 8** | 9** | 9** | 8** |
| 1–43 | Ethyl caprate | 6 | 7* | 6 | 5 | 8** |
| 1–58 | Phenethyl acetate | 8** | 5 | 8** | 7* | 7* |
| 1–60 | Ethyl laurate | 8** | 6 | 8** | 5 | 6 |
| 1–75 | Palmitic acid ethyl ester | 3 | 1 | 8** | 3 | 2 |
| 2 | All alcohols | 10*** | 10*** | 10*** | 10*** | 10*** |
| 2–18 | 3-methyl-1-butanol | 5 | 9** | 6 | 8** | 6 |
| 2–61 | Phenethyl alcohol | 9** | 8** | 8** | 9** | 10*** |
| 3–1 | Acetaldehyde | 5 | – | 6 | 5 | – |
| 3–4 | Acetal | 9** | – | 5 | 8** | 6 |
| 3–22 | Acetoin | 9** | 6 | 7* | 5 | 4 |
| 3–32 | Furfural | 6 | 9** | 5 | 7* | 7* |
| 3–65 | Acetic acid | 8** | 8** | 7* | 7* | 8** |

[a] Number of correct judgments in both omission and addition groups from 10 assessors when evaluating the aroma differences by the triangle test.

aroma properties of five SXB. Omission models lacking all esters (models 1 and 2) resulted in highly significant differences in aroma attributes ($p < 0.001$), particularly related to fruit and flower aromas. The absence of ethyl acetate and ethyl octanoate (models 1–5 and 1–29) significantly influenced the overall aroma characteristics of all mild-flavor Xiaoqu Baijius ($p < 0.01$), with >8 evaluators detecting differences. However, specific esters showed varied contributions across samples. For instance, ethyl 2-methyl butyrate (model 1–13) was only recognized by over 7 evaluators in XK32 and XK35 samples, suggesting its impact on flavor properties. Overall, although ester aroma compounds were present in all five SXB types, their contributions to flavor properties varied.

Like most Baijiu products, alcohols are vital constituents of Chinese Baijiu, contributing fruity and alcoholic notes. They are primarily produced as by-products of yeast growth and ethanol fermentation, with their concentration influenced by the fermentation strains and Qu employed (Guo et al., 2020). Removal of phenylethanol (model 2–61) from the complete reconstruction model significantly reduced floral odor intensity, indicating its importance despite low OAV values. Similarly, absence of isoamyl alcohol (model 2–18) led to notable distinctions between SD and XK35 ($p < 0.01$), underscoring its impact on flavor properties.

Most volatile aldehydes and ketones in Baijiu are metabolized by yeast during fermentation. While acetaldehyde (model 3–1) removal did not notably affect aroma properties, furfural (model 3–32) significantly contributed to flavor, as evidenced by distinctions in SD, XK35, and XK42 samples.

Moreover, removal of 1-diethoxyethane (model 3–4) resulted in detectable differences in YNH and XK35 ($p < 0.01$), highlighting its significant contribution to aroma characteristics. Similarly, 3-hydroxy-2-butanone (model 3–22) played a crucial role in flavor properties, distinguished by >7 evaluators in YNH and XK32 samples.

Acids, known to enhance aroma, form aromatic compounds by combining with other components. Glacial acetic acid (models 3–65) was recognized by >7 evaluators across all samples, underscoring its importance in SXB flavor.

In summary, 17 key aroma compounds were identified across the five samples. The Wayne diagram (Fig. 4b) illustrates differences in key aroma compounds among samples, indicating that unique aroma characteristics arise from varying combinations and concentrations of these compounds, rather than differences in compound types alone. Such intricate combinations, possibly involving compound interactions, contribute to the distinct aroma profiles of SXB brewed from different sorghum varieties.

### 3.8. Relativity analysis

#### 3.8.1. Correlation between the aroma attributes and the key aroma compounds

The PLS-R model uses 17 key aroma compounds for the X matrix and aroma sensory attributes for the Y matrix. Fig. 5a shows the load diagram, distinguishing among the five SXB sample types. Analysis of YNH samples revealed a correlation between various aroma attributes and crucial aroma compounds like ethyl lactate, ethyl acetate, ethyl laurate, glacial acetic acid, and furfural. These findings align with sensory

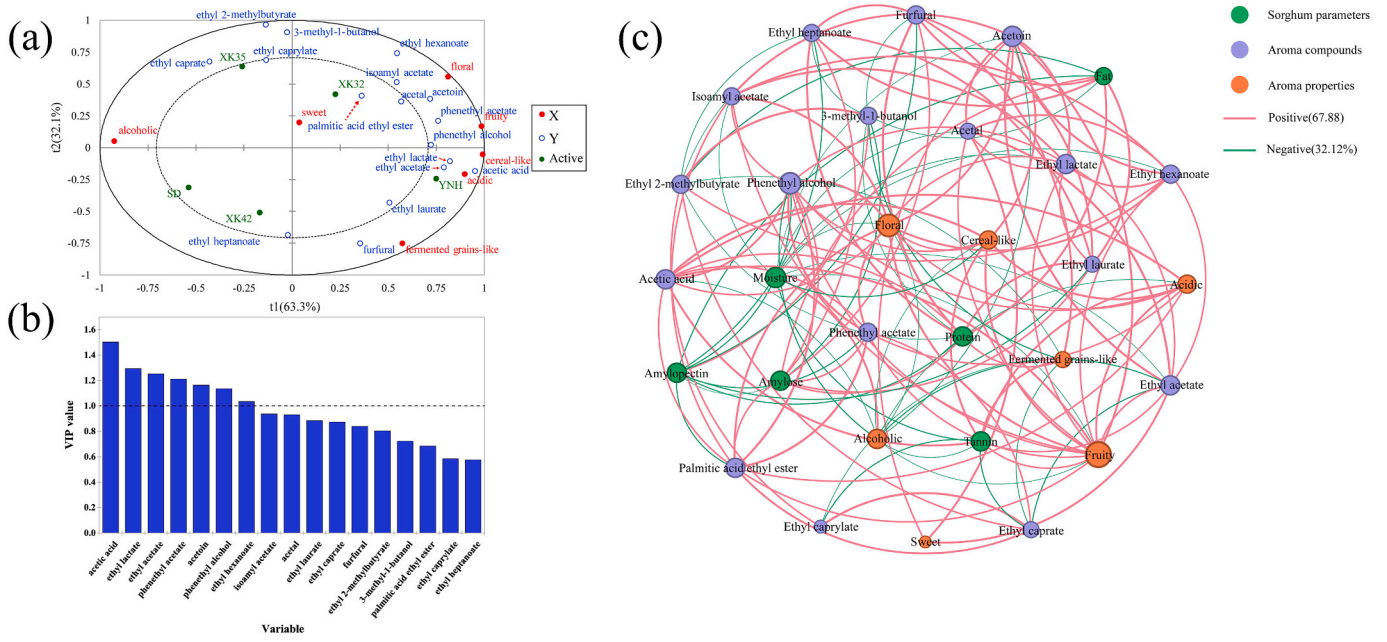



**Fig. 5.** (a) Correlation analysis among SXB samples, aroma properties and key aroma compounds. (b) VIP values of 17 key aroma compounds. (c) Co-occurrence network map of key aroma compounds and aroma attributes based on Spearman correlation coefficient in five kinds of SXB.

evaluations, indicating heightened grain, sour, and fermentation aromas in YNH samples. XK32 samples showed significant correlations between sweetness, flower aroma, fruit aroma, and specific aroma compounds. XK35 samples were mainly associated with alcohol taste, linked to certain aroma molecules. Although SD and XK42 samples were not distinctly differentiated by the model, a connection with ethyl heptanoate was noted. Based on VIP scores above 1, seven key aroma compounds were identified as exerting the greatest influence on the differences in SXB aroma properties.

### *3.8.2. Correlation between the basic physical and chemical indexes of sorghum and the key aroma compounds*

Based on Spearman correlation coefficient ($|r| > 0.7, p < 0.01$), co-occurrence network analysis clarified the interactions between basic physical and chemical parameters and key aroma compounds in SXB brewed from different varieties of alcoholic sorghum. Key aroma compounds influencing the aroma properties of the five SXB types, including glacial acetic acid, ethyl acetate, ethyl lactate, phenylethyl acetate, acetoin, phenylethanol, and ethyl caproate, were identified. Fig. 5c illustrates the findings. Water and alcohol odors exhibited strong negative correlations with several key aroma compounds and aroma attribute factors, consistent with feedback from the evaluation team: samples with stronger alcohol odors tended to receive lower scores for other aroma attributes. Glacial acetic acid, a distinguishing key aroma compound (Guo et al., 2020), showed positive correlations with acid, fruit, grain, and tannin, and weak positive correlations with protein and flower aroma, mainly produced by acetic acid bacteria. Ethyl acetate exhibited positive correlations with sweet aroma and weakly positive correlations with tannin and fruit aroma, in line with sensory descriptors in the AEDA experiment, and tannin can not only inhibit the growth of miscellaneous bacteria, but also give liquor unique aroma in the brewing process (Jiang et al., 2022). Ethyl lactate showed weak positive correlations with flower and fruit aroma and a strong negative correlation with water. Although the five SXB samples had high ethyl lactate content, its contribution to overall aroma was slightly lower due to its high olfactory threshold. Phenylethyl acetate displayed strong positive correlations with flower aroma and weak positive correlations with amylose, fruit, and acid aroma. Acetoin exhibited strong positive correlations with flower, fruit, and grain aroma and a weak positive correlation with sweet aroma. As a key aroma compound with the highest average OAV value, acetoin significantly influenced many aroma properties and exhibited a strong negative correlation with moisture and alcohol odor. Phenylethanol showed strong positive correlations with flower aroma and weak positive correlations with amylose, fruit aroma, acid aroma, and grain aroma, impressing every evaluator with its rich and pleasant scent of roses. Ethyl caproate was positively correlated with flower and fruit aroma and negatively correlated with water, consistent with sensory descriptors from the AEDA experiment.

## 4. Conclusions

The aroma characteristics of SXB brewed from five different sorghum were compared, and 32 kinds of aroma substances were quantified. Sensory study of recombination and omission model confirmed Ethyl acetate, ethyl 2-methyl butyrate, isoamyl acetate, ethyl hexanoate, ethyl heptate, ethyl lactate, ethyl caprylic acid, ethyl decanoate, phenylethyl acetate, ethyl Laurate, ethyl palmitate, isoamyl alcohol, phenylethanol, 1-diethoxy ethane, 3-hydroxy-2-butanone, furfural and glacial acetic acid are the key aroma compounds for the unique aroma of SXB. The results of PLS-R and collinear network analysis showed that glacial acetic acid, ethyl acetate, ethyl lactate, phenylethyl acetate, acetoin, phenylethanol and ethyl caproate were the key aroma compounds which had important influence on the differences of five SXB aroma properties. The results of PLS-R analysis showed that glacial acetic acid, ethyl acetate, ethyl lactate, phenylethyl acetate, acetoin, phenylethanol and ethyl caproate may be the key aroma compounds causing the differences of five SXB aroma properties. The results of collinear network analysis showed that there was the highest positive correlation weight between protein content and furfural content, tannin content and grain aroma property, and the highest negative correlation weight between water content and acetoin content. The results of this study provide a basis for the study of the chemical mechanism of aroma in the raw grain of Baijiu. The study on the specific process of the formation of key aroma compounds in different varieties of Baijiu-making raw materials will guide the breeding policy of raw grain of Baijiu.

## Ethical approval

Sensory evaluation is an important aspect of many research studies, and we recognize the ethical implications associated with conducting such evaluations. We hereby affirm our commitment to protecting the rights and privacy of all participants involved in this study. Throughout the execution of this research, we have implemented appropriate measures to ensure the well-being and autonomy of each participant.

Firstly, participation in this study is completely voluntary, and individuals have the right to decline participation without any negative consequences or coercion. Secondly, we have provided full disclosure regarding the requirements and potential risks associated with the research. Participants are fully informed about the purpose, methods, and expected outcomes of the study, enabling them to make an informed decision about their involvement. Thirdly, written or verbal consent has been obtained from each participant prior to their engagement in the study. This consent ensures that they understand the nature of the research, including any potential uses of their data, and grants permission for their participation. Furthermore, we assure that participant data will be treated with utmost confidentiality and privacy. Under no circumstances will participant-identifying information be published or shared without explicit consent. All data collected will be securely stored and only used for the purposes of this research study. Lastly, we emphasize that participants have the right to withdraw from the study at any time without penalty or explanation. Their decision to discontinue involvement will be respected, and any data collected up until the point of withdrawal will be handled according to the agreed-upon privacy protocols.

We are committed to upholding these ethical principles and ensuring the protection of participants' rights and privacy throughout the entirety of this research study.

## Funding

This work was supported by Talent introduction program of Sichuan University of Science and Engineering (2017RCL24), Sichuan, Tural Science Foundation (2020YJ0402).

## CRediT authorship contribution statement

**Yi Ma:** Project administration, Funding acquisition, Supervision. **Ziyun Wei:** Conceptualization, Data curation, Software, Writing – original draft, Writing – review & editing. **Xiongjun Xiao:** Investigation. **Kangjie Yu:** Formal analysis. **Huiling Huang:** Methodology. **Jianxia Tan:** Methodology. **Yue Wang:** Methodology. **Yong Du:** Resources. **Yajun Li:** Resources.

## 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

The data that has been used is confidential.

## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2024.101367.

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