

# Comparative Analysis of Volatile Components in Chinese Strong-Aroma Baijiu of Different Ages

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## ABSTRACT

In this study, Chinese strong-flavor Baijiu was used as the research object. Volatile compounds were analyzed in newly distilled liquor samples and strong-flavor Baijiu samples aged in stainless steel tanks for 6, 12, 18, and 24 months, respectively. The results demonstrated that organic acids, higher alcohols, and aromatic compounds were continuously generated and accumulated during the aging process, which contributed to the mellow cellar aroma, honey aroma, and aged aroma of the liquor. In addition, the contents of ethyl butyrate, 1-hexanol and other components exhibited a steady upward trend, while acetaldehyde showed a slight fluctuation followed by an increase. Overall, the aging process was characterized by decreased esters, increased acids and alcohols, and coordinated flavor development, verifying the optimizing effect of aging on the flavor profile of Baijiu. This study provides key data support for elucidating the aging mechanism of strong-flavor Baijiu.

## KEYWORDS

Strong-flavour Baijiu; Vintage; Volatile components; Aging

## 1. INTRODUCTION

Baijiu is one of the traditional distilled spirits in China, with a history of over 2,000 years [1], after thousands of years of development, Chinese Baijiu has gradually formed a style system featuring multiple types of aromas. Different types of Baijiu have significant differences in raw materials for brewing, fermentation methods, and process parameters. Moreover, Baijiu has always played an important role in the national economy [2]. According to data from the National Bureau of Statistics of China, the total output of liquor in China will reach 35.49 million tons in 2025. Among them, strong-flavored liquor is highly favored due to its unique flavor and taste. Strong-flavored liquor is produced through processes such as solid-state fermentation, distillation, aging, and blending. Among these, aging is an important process during the fermentation of strong-flavored liquor, which imparts an aged aroma to the liquor and enhances its overall aroma. This process involves complex physical and chemical changes, including esterification-hydration reactions [3], during this process, the concentration of esters in strong-flavored Chinese liquor gradually decreases as the storage time increases, while the total acid content increases particularly in the early stage of aging and then stabilizes [4]. In addition, it will also strengthen the hydrogen bond structure between water and ethanol, thereby enabling a close binding between the water and ethanol molecules [5].

People have been actively exploring the principles of aging for a long time, including the analysis of the changes in the content of volatile and non-volatile substances in different years of aged Chinese liquor. Most of Chinese liquor is composed of water and ethanol, and the content of trace components that determine the flavor differences is extremely low. The core advantage of gas chromatography-

mass spectrometry (GC-MS) in the analysis of Chinese liquor lies in its excellent separation ability and precise structural identification capability. The matrix of strong-flavor Chinese liquor is complex, with hundreds of volatile components and significant variations in their contents. GC-MS achieves efficient separation of complex mixtures through the chromatographic column and combines the characteristic fragment ions provided by the mass spectrometry with the retrieval from the spectral library to determine the structural analysis of trace even traceable flavor components. At the same time, its sensitivity can reach ng/L level, meeting the detection requirements for extremely low concentration compounds. Compared with traditional chromatographic methods that rely solely on retention time, GC-MS significantly improves the reliability of qualitative and quantitative results and is a standardized tool for constructing the fingerprint map of volatile components in Chinese liquor and screening differentiated compounds.

In this study, gas chromatography-mass spectrometry (GC-MS) was employed to analyze the new wine samples as well as the aged wine samples stored in stainless steel tanks for 6 months, 12 months, 18 months, and 24 months. The changes in volatile components were analyzed to further understand the variation patterns of volatile components in the aged process of the strong-flavor Chinese liquor.

## **2. MATERIALS AND METHODS**

### **2.1. Materials**

The strong-flavored liquor comes from Jincaishan Liquor Co., Ltd. (Shandong, China). The new wine sample and the strong-flavored liquor samples aged in stainless steel tanks for 6 months, 12 months, 18 months, and 24 months are all samples of the same production process. Methanol, isoctanol, caproic acid, lactic acid, ethyl acetate, caproylethyl acetate, butyl acetate, and ethyl lactate (chromatographic grade,  $\geq 98\%$ ) are all purchased from Guoxun Group Chemical Reagents Co., Ltd. The standard substances for 0.1 mol/L sulfuric acid titration solution and 0.1 mol/L sodium hydroxide titration solution are purchased from Beiyouweiye Metrology Group Co., Ltd.

### **2.2. Measurement of Physical and Chemical Properties of Baijiu**

#### **2.2.1. pH Value Measurement**

Based on the alcohol content measured by the alcohol detector, referring to the national standard analysis method for liquor (GB 5009.225–2023), the pH value of the sample at 25°C was determined using an automatic potentiometric titrator.

#### **2.2.2. Measurement of Total Esters**

$$X = \frac{c \times (V_1 - V_0) \times 88}{50}$$

X represents the total ester concentration, in grams per liter; c is the concentration of the sodium hydroxide standard titrant, in moles per liter; V<sub>0</sub> and V<sub>1</sub> are the volumes of the titration solution consumed by the blank sample and the liquid sample, respectively, which are in milliliters; 88 is the molar mass of ethyl acetate, in grams per mole; 50.0 is the volume of the alcohol sample, which is also in milliliters.

#### **2.2.3. Measurement of Total Acid**

For the total acid test, please refer to the national standard analysis method for liquor GB 12456–2021 to determine the total acidity. The calculation formula for the total acid content in the liquor sample is:

$$X = \frac{c \times (V_1 - V_0) \times 60}{50}$$

X represents the total acid concentration in the liquid sample, measured in grams per liter; c is the actual concentration of the sodium hydroxide standard titrant, measured in moles per liter; V<sub>0</sub> and V<sub>1</sub> are the volumes of the titration solution consumed by the blank sample and the liquid sample, respectively, both measured in milliliters; 60 is the molar mass of acetic acid, measured in grams per mole; 50.0 is the volume of the alcohol sample, also measured in milliliters.

### 2.3. Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

One milliliter of the new wine sample and one milliliter of each of the 6-month, 12-month, 18-month, and 24-month aged wine samples from the stainless steel tank were separately taken, and each was mixed with 4 milliliters of deionized water to form 5 milliliters of samples. The internal standard was selected as a 1 g/L dodecanol solution, and 20 µl of the internal standard was added to each sample.

GC conditions: RTX-WAX capillary chromatographic column (30 m × 0.25 µm × 0.25 mm); carrier gas is high-purity helium; non-flow injection; injection port temperature 250 °C; the column temperature adopts programmed temperature rise: the programmed temperature rise is 40 °C for 3 minutes, then rises at 2 °C/min to 130 °C, then rises at 10 °C/min to 220 °C, and maintains for 4 minutes.

MS conditions: transfer line temperature 250 °C; ion source temperature 230 °C; quadrupole temperature 150 °C; electromagnetic source EI; ion source EI; ionization energy 70 eV; scanning mass range 15 - 500 amu; mass spectrometry scanning mode: full scan (SCAN).

Data processing: The volatile components detected were analyzed qualitatively and quantitatively after being analyzed by mass spectrometry and retrieved from the NIST2020 spectral library; for qualitative analysis, the matching factor was greater than 80; for quantitative analysis, the internal standard method was used for calculation.

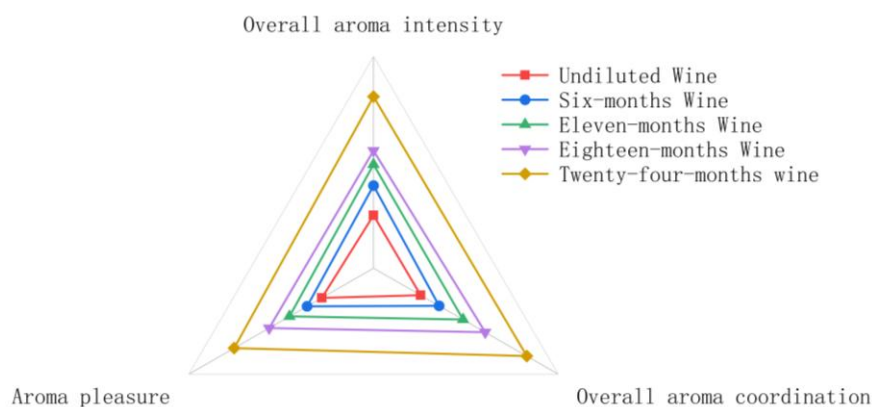
### 2.4. Statistical Analysis

Each wine sample was measured and repeated three times. The error bars represent the standard deviation (SD). The data in the table are presented as the mean ± standard deviation (SD). A simple t-test was conducted, and the asterisks indicate the P value (\* P < 0.05 and \*\* P < 0.01). One-way analysis of variance (ANOVA) and Duncan's multiple range test were performed using SPSS 27.0 software to evaluate the significant differences in the measurements (p < 0.05).

## 3. RESULTS AND ANALYSIS

### 3.1. Sensory Evaluation

The description of the sensory analysis technology enables sensory researchers to obtain a comprehensive sensory description of the product and determine which sensory attributes are crucial for acceptance. To determine whether the overall aroma characteristics of the aged sample have improved, the sensory acceptance of the sample is represented by three sensory indicators: "overall aroma intensity", "overall aroma harmony", and "aroma pleasure". Figure 1(a) shows that as the aging time increases, "overall aroma intensity", "overall aroma harmony", and "aroma pleasure" all improve. It can be seen that aging can enhance the harmony and acceptance of the liquor sample, thereby improving the overall aroma quality of the liquor.



**Figure 1.** Sensory Evaluation Form for Wine Samples with Different Aging Times

## 3.2. Analysis of Key Aroma Active Components

### 3.2.1. Characterization of Aromatic Active Compounds

This study employed gas chromatography-mass spectrometry (GC-MS) to conduct a systematic qualitative and quantitative analysis of the key aromatic active compounds in strong-flavored Chinese liquor under different aging conditions. The aim was to reveal the dynamic change patterns of flavor substances during the aging process and their influence on the aroma quality of the liquor. By detecting various characteristic esters such as ethyl caproate, caproic acid, ethyl cinnamate, and octyl acetate, as well as organic acids and trace aromatic components, the differences in the aroma composition of the liquor at different storage stages were compared. The intrinsic relationship between aging time and the content of main aroma, auxiliary aroma, and trace flavor substances was explored. The research results can provide scientific basis and data support for in-depth understanding of the aging mechanism of strong-flavored Chinese liquor, optimization of storage processes, and improvement of the flavor quality of the liquor.

**Table 1.** GC-MS Analysis of Wine Samples with Different Aging Times

Compound	Odor description	Concentration (mg/L)				
		Raw wine	6 months	12 months	18 months	24 months
Ethyl hexanoate	Pineapple, cardamom	1416	1397	1333	1287	1276
Acetaldehyde	Spicy	52	57	59	54	64
Ethyl butyrate	Apple fragrance	11	15	19	23	28
Ethyl valeraldehyde	Fruit fragrance	110	107	103	98	91
1,1-Dioxy-3-methylbutane	Fruit fragrance	71	76	77	74	79
2-Methyl-1-propanol	Stimulating	33	36	41	44	42
1-Hexanol	Resin	34	37	39	41	44
Lauric acid ethyl ester	Grass	532	542	577	593	614
Acetic acid ethyl ester	Fruity aroma,	33	36	39	44	47
Benzaldehyde	Bitter almond	0.9	1.77	2.61	3.42	4.43
Ethyl acetate	Malt	972	951	916	874	841
Heptanoic heptyl ester	Fat	31	33	37	42	48
Propionic acid	Sour and astringent	81	83	88	89	82
Valproic acid	Sweat	2.3	3.1	4.2	5.7	6.2
2-Phenylethanol	Rose	237	261	288	341	377
Palmitoleic acid ethyl ester	Fruit fragrance	5.81	6.23	6.66	7.81	9.17
2,4-Di-tert-butylphenol	Phenols	1.3	2.1	3.8	4	5.9
Ethyl 3-phenylpropionate	Floral fragrance	11	14	17	21	24
Palmitic acid	Soap, fat	55	71	88	94	107
Ethyl valerate	Fruit fragrance	172	163	156	147	133
3-Methylbutanol	Malt	181	188	193	197	211
4-Deoxy-11-keto	Sausage aroma	71	77	79	80	79
Ethyl nonanoate	Fruity aroma, rose aroma	177	182	193	214	242
Hexyl caproate	Fresh fruits	38	41	37	33	29
Ethyl phenylacetate	Fruity aroma, Honey	1.1	1.2	1.1	1.4	1.3
Isobutyl decanoate	Fruit fragrance	17	15	12	11	8.7
3-Methylbutyl Ether	Fruit fragrance	0.3	0.17	0.15	0.11	0.08
Hexanoic acid	Fruit fragrance	534	577	614	635	662

The results of GC-MS analysis show that during the 24-month aging process of strong-flavored Chinese liquor, the aromatic active compounds exhibit a significant dynamic change pattern. Among the main flavor substances, core esters such as ethyl caproate, ethyl valeralate, and ethyl caproate show a continuous downward trend as the aging time prolongs. Specifically, ethyl caproate decreases from 1416 mg/L in the original liquor to 1276 mg/L in the 24-month-aged liquor. The trace esters such as ethyl caproate, 3-hexyl butyrate, etc., also decrease simultaneously. This indicates that esters undergo hydrolysis and transformation during the aging process, and the stimulating fruity aroma of the liquor gradually softens. In contrast, the contents of substances such as caproic acid, ethyl laurate, ethyl octanoate, 2-benzyl alcohol, and benzaldehyde continuously increase. Caproic acid rises from 534 mg/L to 662 mg/L, and 2-propanol increases from 237 mg/L to 377 g/L. This indicates that

organic acids, higher alcohols, and aromatic compounds are continuously generated and enriched during the aging process, giving the liquor a more mellow cellar aroma, honey aroma, and aged aroma. Additionally, substances such as ethyl butyrate and 1-hexanol show a steady increase, while acetaldehyde fluctuates slightly before increasing. Overall, it presents the characteristic of "decrease in esters, increase in acids and alcohols, and harmonization of flavors", confirming the optimization effect of aging on the flavor of strong-flavored Chinese liquor, and providing key data support for analyzing the aging mechanism of strong-flavored Chinese liquor.

## 4. RESULTS AND DISCUSSION

This study analyzed the aroma-active compounds of different-aged strong-flavored Chinese liquor using GC-MS, revealing the dynamic evolution patterns of flavor substances during the aging process and the influencing mechanisms on the quality of the liquor. The results showed that aging is the core process by which the flavor of Chinese liquor transforms from "new wine stimulation" to "aged wine richness", and its essence is the balanced reconfiguration of flavor substances such as esters, acids, and alcohols.

The main esters (such as ethyl hexanoate, ethyl caproate, and ethyl hexyl ester) show a continuous downward trend with the extension of aging time. This change mainly results from the hydrolysis reaction of esters, accompanied by the dynamic adjustment of the esterization-hydration equilibrium, which gradually softens the sharp fruity and yeast aromas in the liquor and avoids the harshness of new wine. In contrast, the contents of organic acids such as caproic acid, ethyl hexanoate, and ethyl octanoate, as well as long-chain esters, continue to increase. Among them, hexanoic acid, as the core precursor substance for cellar aroma in strong-flavored Chinese liquor, its content increased from 534 mg/L to 662 mg/L, not only enhancing the body's richness and cellar aroma foundation, but also providing a material basis for the slow generation of esters, confirming the traditional brewing cognition of "acid increases while esters decrease and cellar aroma gradually emerges".

The changes in higher alcohols and aromatic compounds further optimized the flavor layers of the liquor: the contents of substances such as 2-phenylethanol and benzaldehyde significantly increased with aging, endowing the liquor with elegant honey, rose, and almond aromas. The slow accumulation of substances such as 3-methylbutanol, which has a malt aroma, enriched the complex aroma of the liquor. The content of acetaldehyde fluctuated slightly and then increased, reflecting the oxidation and transformation of aldehyde substances during the aging process, which helps to reduce the spiciness of new wine and enhance the smoothness of the liquor.

The results of this study clarified the change patterns of flavor substances during the aging process of strong-flavored Chinese liquor, providing a scientific basis for optimizing the aging process and precisely regulating the flavor of the liquor. In the future, combined with sensory evaluation and aroma activity value (OAV) analysis, the contribution of key flavor substances to the aroma of the liquor can be further clarified, deepening the understanding of the liquor aging mechanism.

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