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The effect of aged pork fat on the quality and volatile compounds of Chi-aroma Baijiu

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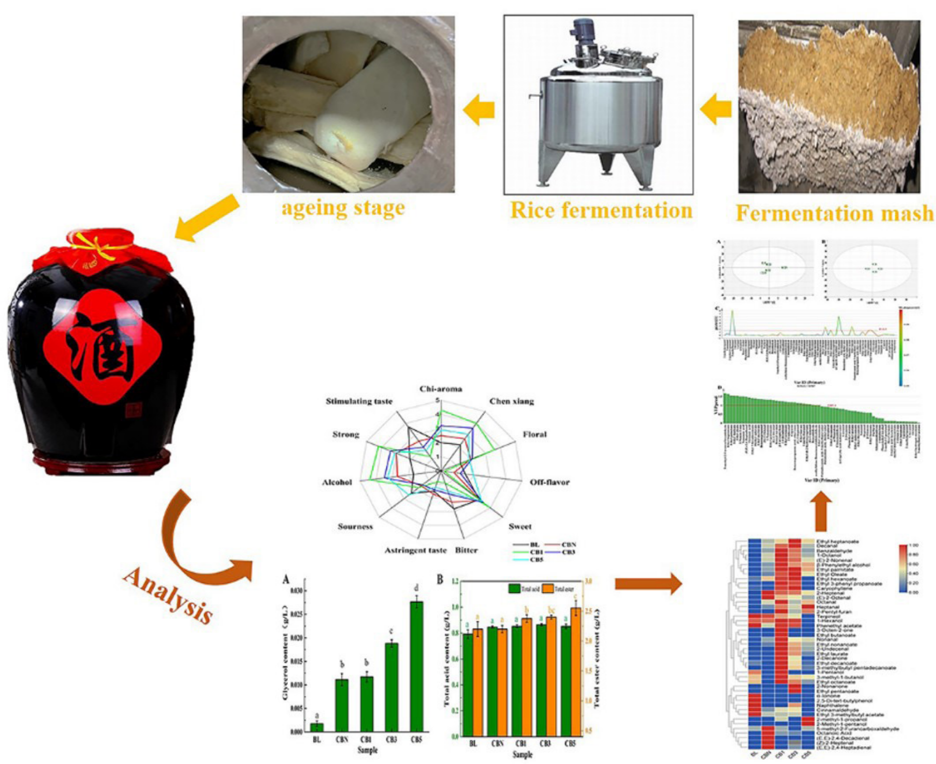
Abstract

This study aimed to explore the effect of aged pork fat on the quality and volatile compounds of Chi-aroma Baijiu during the ageing stage. Result showed that glycerol and total ester contents of CBN, CB1, CB3 and CB5 were significantly higher than that in BL ($P < 0.05$), but total acid contents did not change significantly ($P > 0.05$). Moreover, there was a difference in volatile compounds concentration and OAVs among the Chi-aroma Baijiu samples. With the year of aged pork fat increased, concentrations of esters and aldehydes were increased and then decreased, which reached highest in CB1. CB1 had highest OAVs of medium and long-chain ethyl esters and four aldehydes ((E)-2-nonenal, octanal, (E)-2-decanal, (E)-2-octenal) were the characteristic volatile compounds of Chi-aroma Baijiu during ageing process owing to their OAV > 10. Therefore, CB1 had the highest sensory score, with intense Chi-aroma and pleasant aftertaste. OPLS-DA further effectively distinguish different Chi-aroma Baijiu soaking with different years of aged pork fat, and confirmed 31volatile compounds (especial ethyl esters) were the key volatile compounds responsible for significant differences in Chi-aroma Baijiu. Combined with the results of sensory evaluation, GC–MS and OAVs, one-year aged pork fat was the best to product Chi-aroma Baijiu.

Keywords: aged pork fat; Chi-aroma Baijiu; ageing process; volatile compounds.

Practical Application: The changes of quality and volatile compounds of Chi-aroma baijiu soaking with different aged pork fat during ageing stage were analyzed, and one-year aged pork fat was the best to product Chi-aroma Baijiu, which improved quality and flavour of Chi-aroma baijiu for industry.

Graphical Abstract



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1 Introduction

Baijiu is a traditional distilled spirit produced from grain fermentation and is one of the most popular alcoholic beverage in China (Zheng et al., 2016). It has an annual output exceeded 4.6 million kiloliters in 2021, creating a market profit of > 1560 billion RMB (data from the China Alcoholic Drinks Association). Baijiu is classified into twelve categories, based on their aroma characteristics, including soy sauce, strong, light, rice, herb-like, Feng, complex, Chi-aroma, texiang, fuyu, laobaigan and sesame flavour type (Du et al., 2021; Fan et al., 2021; Li et al., 2019; Wang et al., 2022). Among these types, Chi-aroma Baijiu is regarded as a special liquor from the Pearl River Delta region of China, which is popular owing to its special Chi-aroma (soybean aroma) product by unique ageing technology (soaking with aged pork fat).

Chi-aroma Baijiu uses rice as raw material, and fermentation mash being made of cooked rice, soybean, xiaoqu and water were taken into the fermenter to saccharification and fermentation (He et al., 2019). After rice fermentation, it is directly extracted by kettle distillation to produced low-alcohol liquor with approximately 30% vol, which is commonly known as base liquor (Shen, 1996). Because of the insoluble impurities, basic liquor is generally turbid with a spicy, irritating and unpleasant taste. After soaking with aged pork fat for 30 days, base liquor will become bright and transparent, and possessed sweet and smooth taste, which is known as ageing process of basic liquor. Finally, Chi-aroma Baijiu is produced by blending and filtering the ageing base liquor. Soaking with aged pork fat is an essential factor for the production of Chi-aroma Baijiu, making the flavour of Chi-aroma Baijiu different from other flavour type Baijiu. Aged pork fat plays two key roles in the ageing process (Yue et al., 2022). Aged pork fat absorbs suspended substances to clarify base liquor (Zeng & Zhang, 2002). In addition, oxidative degradation of aged pork fat in base liquor to produce aromatic esters, generating a Chi-aroma flavour. Therefore, aged pork fat is important for improving the quality of Chi-aroma Baijiu.

Aged pork fat is obtained from fresh pork fat with skin that is boiled and soaked in basic liquor with high alcohol content (55% vol) for 4 months, followed by 2 months in basic liquor with low alcohol content (30% vol). Aged pork fat mainly consists of saturated fatty acids (SFA) and unsaturated fatty acid (UFA). Wei et al. (2020) studied the role of different treatment of pork fat significantly affecting the thiobarbituric acid reactive substances (TBARS) values and the contents of SFA and UFA during the soaking process in basic liquor. However, the factory repeated use of aged pork fat in ageing process, resulting in different years of aged pork fat. Therefore, owing to this production method, different years of aged pork fat may have an effect on the taste and flavour of Chi-aroma Baijiu. Recent research involving Chi-aroma Baijiu have only focused on volatile compounds in Chi-aroma baijiu (Fan et al., 2015b; Wei et al., 2020), whereas the effects of different year of aged pork fat on quality in Chi-aroma Baijiu remains unclear.

Accordingly, the aim of this study was to explore the effects of aged pork fat on the quality and volatile compounds of Chi-aroma baijiu by sensory evaluation, main quality indices analysis and chromatography-mass spectrometry (GC-MS). Furthermore, the significant volatile compounds were determined by OAV coupled with multivariate analysis. Results can potentially provide a theoretical support on the quality control on Chi-aroma Baijiu.

2 Experimental

2.1 Chemical and materials

Base liquor, new aged pork fat, one-year aged pork fat, three-year aged pork fat and five-year aged pork fat were provided by Jiujiang Winery of Guangdong Province Co., Ltd (Guangdong, China). Base liquor (BL, 48% ethanol by volume) was stored at 25 °C for 30 days. With a ratio of basic liquor to aged pork fat of 4:1 (L/kg), base liquor soaking with new aged pork fat at 25 °C for 30 days (CBN, 48% ethanol by volume), base liquor soaking with one-year aged pork fat at 25 °C for 30 days (CB1, 48% ethanol by volume), base liquor soaking with three-year aged pork fat at 25 °C for 30 days (CB3, 48% ethanol by volume), and base liquor soaking with five-year aged pork fat at 25 °C for 30 days (CB5, 48% ethanol by volume) were obtained different Chi-aroma baijiu. All Baijiu samples in 50 mL glass bottles were stored at -80 °C until analysis.

Butyl acetate and ethyl alcohol (analytical reagent grade) and Megazyme Glycerol test kit were purchased by Alladdin Co., Ltd (Shanghai, China). N-alkane mixture (C7–C40) were purchased from Sigma-Aldrich Co., Ltd (Shanghai, China). Sodium hydroxide standard titration solution, sulfuric acid standard titration solution and phenolphthalein indicator were purchased by Supelco Co., Ltd (Darmstadt, Germany).

2.2 Determination of glycerol content

Glyceol content was detected according to Chinese national standard GB/T10345 (National Standards of the People's Republic of China, 2007). The sample group was added with distilled water (1.90 mL 25 °C) and 0.10 mL Baijiu sample was added to the sample groups, whereas the blank group used an addition of 2.00 mL 25 °C distilled water. The two groups were added with 0.20 mL bottle 1 solution, 0.20 mL bottle 2 solution and 0.02 mL bottle 3 solution respectively, reacting for 4 min. The absorbance value A_1 was measured by ultraviolet spectrophotometer at 340 nm, and then the bottle 4 solution was added, reacting for 5 min. The absorbance value A_2 was measured. The glycerol content in the sample was calculated according to below formula (Equation 1).

$$C = \frac{2.34 \times MW}{\ni \times d \times 0.1} \times \frac{(A_1 - A_2)_{wine\,sample}}{(A_1 - A_2)_{blank\,sample}} \quad (1)$$

C: glycerol content (g/L); MW: molecular of glycerol (g/mol); ɜ: digestibility coefficient of NADH at 340 nm; d: optical path (cm).

2.3 Determination of total ester and total acid contents

Total ester and total acid contents were detected according to Chinese national standard GB/T10345 (National Standards of the People's Republic of China, 2007). 50.0 mL Baijiu samples were taken in 250.0 mL reflux bottle, and 2 drops of phenolphthalein indicator were added. The sample solution was titrated by sodium hydroxide standard solution, which the end point of titration was pink. Total acid was calculated. Then 25.0 mL sodium hydroxide standard solution was added, and sample solution was refluxed in boiling water bath for 30 min. After cooling, sample solution was titrated with sulfuric acid for disappearance of slight red, and the total ester content was calculated.

2.4 Sensory evaluation

Ten trained panellists (5 males and 5 females) from Jiujiang Winery of Guangdong Province Co., Ltd. participated in the sensory evaluation. Panellists were trained based on the method (He et al., 2021b; Xiao et al., 2016). Each sample was loaded into glass bottles marked with three-digit codes, and then randomly distributed to the panellists. The flavour fingerprints of GB/T16289 (National Standards of the People's Republic of China, 2018) were analyzed for 10 sensory attributes, which were Chi-aroma, chenxiang, floral, off-flavour (rancid odour), sweet, bitter, astringent taste, sourness, alcohol, strong, stimulating taste. The score of each attribute ranged from 0 to 5, where 0 meant not perceivable and 5 meant strongly perceivable. Each sample was evaluated in triplicate by every panellist.

2.5 GC-MS analysis

The volatile extracts were obtained by HS-SPME (Fu et al., 2021). 6 mL sample and 30 μL internal standard solution (butyl acetate, 17650 mg/mL) was added into a screw-capped headspace vial. Then, the sample was equilibrated at 50 °C for 10 min, and extracted with a 50 μm/30 μm DVB/CAR/PDMS fiber (Supelco Inc., Bellefonte, PA) at 50 °C for 40 min. After extraction, the fiber was transferred into the GC injector to desorb at 250 °C for 5 min.

The volatile compounds of Chi-flavour Baijiu were detected by an Agilent 6890-5973N gas chromatograph (Agilent Technologies, Santa Clara, CA) equipped with an Agilent 5975 mass spectrometry detector. A DB-WAX UI column (30 m × 0.25 mm × 0.25 μm; Agilent Technologies, Santa Clara, CA) was used on the GC-MS analysis. Helium was delivered as carrier gas at a flow rate of 1.0 mL/min. The oven temperature was programmed from 40 °C to 120 °C at a rate of 5 °C/min (held for 5 min), then increased to 150 °C at the rate of 5 °C /min (held for 8 min), and finally raised to 250 °C at a rate of 5 °C/min (held for 10 min). The temperature of the ion source was 230 °C, and the temperature of quadrupole was 150 °C. A full scan with the acquisition range was performed from m/z 47 to 550. Identification of volatile compounds was carried out by comparing their MS with the standard mass spectra in NIST14 database, if the matching masses were more than 90%, the compound can be tentatively identified. At the same time, the compound was verified by comparison of retention indices (RIs) with the RI reported in literatures. RIs were calculated using a C7–C40 n-alkane mixture under the same GC-MS conditions (Cates & Meloan, 1963).

2.6 Determination of Odour Activity Values (OAVs)

To calculate OAVs, odour thresholds of some aromas were determined in 46% ethanol/water solution by triangle tests based on Czerny et al. (2008).

2.7 Statistical analyses

All analyses were conducted in triplicate. The relevant results were denoted as mean±standard deviation (SD). Significant differences among groups were performed statistically by one-way analysis of variance (ANOVA) combined with Tukey's multiple-range test using SPSS 22.0 (IBM, Armonk, NY). Heat maps were constructed by TBtools (Toolbox for Biologists; version 1.098, China). Orthogonal partial least-square discriminant analysis (OPLS-DA) were performed in SIMCA-P 14.1 (Umetrics, Sweden).

3 Results and discussion

3.1 The effect of aged pork fat on the quality of Chi-aroma Baijiu

The quality of Chi-aroma Baijiu samples was described and evaluated by10 representative sensory descriptors. Based on the sensory evaluation, the BL was perceived to have a stimulating taste with 3.7 points, followed by bitter (2.8) and sourness (2.4). Thereafter, the sensory evaluation of the Chi-aroma Baijiu soaking with different years of aged pork fat were determined (Figure 1). The sensory of CB1 were significantly higher than CBN, CB3 and CB5 (*P*<0.05). CB1 had intense Chi-aroma (4.3) and chenxiang (4.0) flavour, with pleasant aftertaste and prominent alcohol. Moreover, the scores of Chi-aroma (from 4.3 to 2.9), floral (from 3.6 to 1.9) and alcohol (from 4.4 to 3.3) decreased with the increasing years of aged pork fat. Sensory changes indicated that soaking with aged pork fat had an optimization effect on the taste and flavour of Chi-aroma Baijiu, and one-year aged pork fat had the best optimization effect.

Glycerol was one of the most important characteristics of Chi-aroma Baijiu, which could accelerate alcoholization of baijiu, to provide sweet and soft taste (Zeng & Zhang, 2002). The glycerol contents of different baijiu samples were showed in Figure 2A. Glycerol contents of CBN, CB1, CB3 and CB5 were significantly higher than that in BL ($P < 0.05$), and less than 0.6 g/L meeting the requirements of GB/T16289 (National Standards of the People's Republic of China, 2018). Because

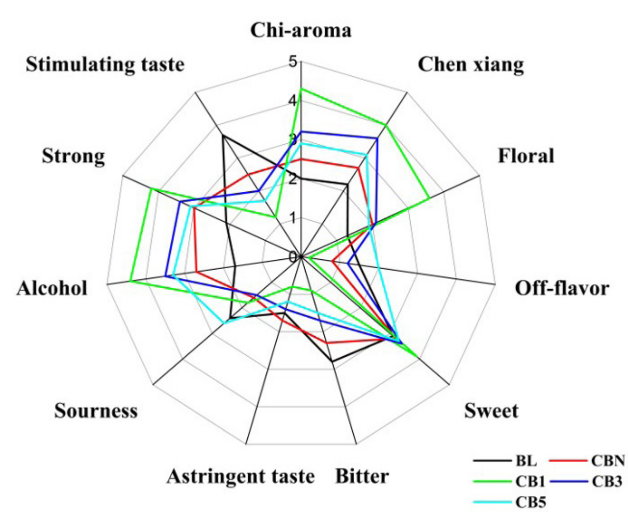

Figure 1. Sensory evaluation of different Baijiu samples.

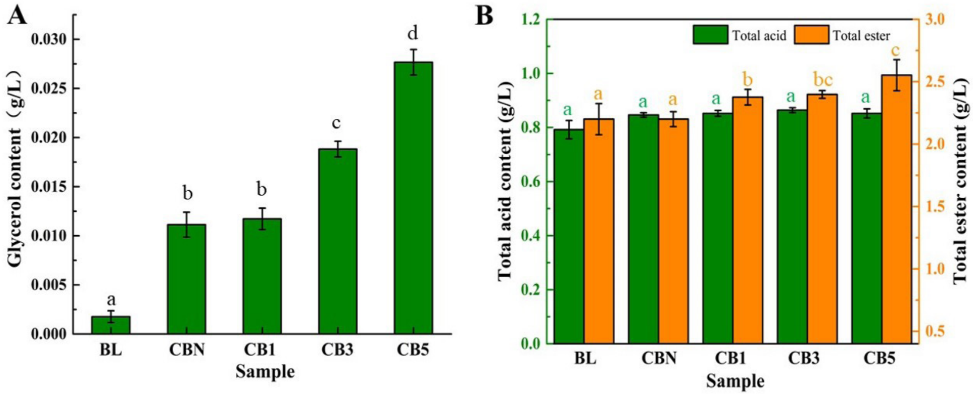

Figure 2. Glycerol (A), total acid and total ester contents (B) of different Baijiu samples. Different letters within same row indicate significant difference (P < 0.05).

fatty in aged pork fat decomposed into glycerol and higher fatty acids (Cabral et al., 2022), which could lead to an increase in glycerol contents during the ageing process of Chi-aroma baijiu. With increasing year of aged pork fat, the pork fat structure was looser and easier to decompose, so the glycerol contents of Baijiu samples increasing.

Total acids in Chi-aroma Baijiu were mainly composed of organic acids, which were one of the main taste substances. Total esters were typically used as an important index to measure the Baijiu quality. The total acid and total ester contents of different Baijiu samples were determined in Figure 2B. Compared with BL, the total acid and total ester contents of CBN, CB1, CB3 and CB5 were higher. However, there was no significant change in total acid contents among Chi-aroma Baijiu samples ($P > 0.05$). Because acids were mainly produced by microorganisms during fermentation stage (Zhang et al., 2021), the changes of total acid contents in each baijiu sample were not obvious during the ageing process (Sun et al., 2022). Nevertheless, total ester content of Chi-aroma Baijiu increased after ageing, which was consistent with the change rule in the aging process of strong-flavour baijiu (Huang et al., 2022) and Fen-flavour baijiu (Jia et al., 2022). With increasing year of aged pork fat, fat structure would be looser, and accelerate unsaturated fatty acids oxidized to form acids (Shahidi & Zhong, 2010), which combined with alcohols to produce esters, slightly increasing the total ester contents. Therefore, the increase year of aged pork fat would improve the total ester content in Chi-aroma baijiu.

3.2 Analysis of volatile compounds of Chi-aroma Baijiu

HS-SPME-GC-MS analysis was conducted to identify the volatile compounds of Baijiu samples soaking with different years of aged pork fat for 30 days and base liquor storing for 30 days. As shown in Figure 3, a total of 80 volatile compounds in five Baijiu samples were identified, including 12 alcohol, 18 aldehydes, 5 ketones, 28 esters, 2 acids, 10 alkanes, 1 furan and 4 others compounds. After ageing, the composition of volatile compounds in each Baijiu samples was quite different. 36 volatile compounds were detected in the BL, 44 volatile compounds in CBN, 58 volatile compounds in the CB1, 55 volatile compounds in the CB3, and 43 volatile compounds in the CB5. The result

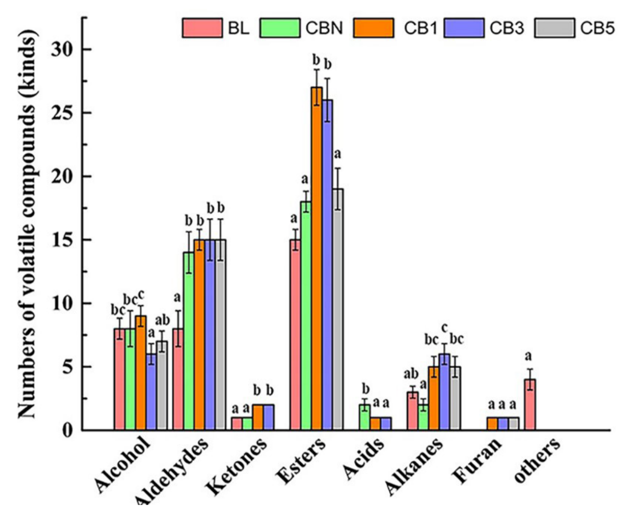

Figure 3. The number of volatile compounds in different Baijiu samples. Different letters within same row indicated significant difference at different Baijiu samples (P < 0.05).

showed that volatile compounds in CB1 were significantly enriched, and the majority of volatile compounds determined were esters, aldehydes and alcohols (Zhang et al., 2021).

The concentrations of total volatile compounds were different among five Baijiu sample (Table 1). Esters were mainly produced during the fermentation or ageing stage, contributing to fruity, sweet flavours and more complex aromas (González-Rompinelli et al., 2013; Zhu et al., 2020). Esters were the largest proportion of total volatile compounds, which is consistent with a previous report (Fan & Qian, 2006). The esters contents of CB1 (473.89 mg/kg) were the highest, followed by CB3 (370.51 mg/kg), CB5 (186.14 mg/kg), CB1 (157.16 mg/kg), and BL (154.03 mg/kg) were the lowest. Ethyl esters had already been reported as the major contributors to the characteristic flavour of Chi-aroma baijiu, which were mainly formed through esterification of alcohols with fatty acids during the ageing process (Rahman et al., 2022). As the years of aged pork fat increased, the contents of medium and long-chain ethyl esters in CBN, CB1, CB3 and CB5 increased

Table 1. Volatile compounds of Chi-aroma Baijiu soaking with different years of aged pork fat.

NO.	Aroma compound	Concentration[2] (mg/kg)					Odorous Description[3]
		BL	CBN	CB1	CB3	CB5	
	Alcohols						
1	2-methyl-1-propanol	-[1]	0.98 ± 0.08 ^{a}	7.25 ± 0.45^{b}	-	17.61 ± 0.82^{c}	frusel
2	2-methyl-1-pentanol	-	-	-	-	3.98 ± 0.46	-
3	1-pentanol	89.81 ± 9.06 ^{b}	0.11 ± 0.05 ^{a}	115.61 ± 13.96 ^{c}	-	-	fruity
4	3-methylbutanol	90.14 ± 13.56^{a}	-	147.69 ± 15.84^{b}	85.43 ± 11.46^{a}	74.36 ± 13.16^{a}	whiskey, malt
5	1-hexanol	0.56 ± 0.06^{ab}	0.54 ± 0.04^{a}	0.65 ± 0.02^{c}	0.61 ± 0.05^{bc}	-	green
6	1-octanol	0.91 ± 0.31^{a}	1.05 ± 0.46^{a}	1.25 ± 0.38^{a}	1.17 ± 0.32^{a}	1.02 ± 0.42^{a}	fruity
7	α-terpineol	2.40 ± 0.34^{b}	2.22 ± 1.09^{b}	1.78 ± 0.51^{b}	1.29 ± 0.96^{ab}	0.33 ± 0.15^{a}	oil
8	Benzenemethanol	1.28 ± 0.07^{a}	2.77 ± 0.92^{a}	2.14 ± 1.38^{a}	2.00 ± 0.56^{a}	1.63 ± 1.26^{a}	sweet, ester
9	2-phenylethanol	25.58 ± 0.84^{b}	27.60 ± 3.62^{b}	34.38 ± 2.51^{c}	32.05 ± 1.38^{c}	13.42 ± 1.06^{a}	rose
10	3-Phenylpropanol	-	0.18 ± 0.05^{a}	0.15 ± 0.07^{a}	-	-	fruity
11	(E)-spathulenol	1.05 ± 0.08	-	-	-	-	-
	Aldehydes						
12	Heptanal	-	0.29 ± 0.06^{a}	1.08 ± 0.03^{c}	0.59 ± 0.06^{b}	1.39 ± 0.08^{d}	fatty
13	Octanal	-	0.91 ± 0.46^{ab}	2.14 ± 1.24^{b}	0.74 ± 0.58^{ab}	1.34 ± 0.56^{b}	green
14	(E)-2-heptenal	-	1.65 ± 0.75^{a}	1.67 ± 0.56^{a}	1.31 ± 0.79^{a}	1.26 ± 0.82^{a}	fatty
15	Nonanal		1.09 ± 0.58^{ab}	4.18 ± 0.94^{c}	2.09 ± 0.85^{b}	1.8 ± 0.92^{b}	Fatty, soapy
16	(E)-2-octenal	0.38 ± 0.06^{a}	8.34 ± 2.61^{c}	5.27 ± 1.70^{b}	4.08 ± 1.89^{b}	2.57 ± 1.25^{ab}	sweet
17	(E, E)-2,4-heptadienal	-	0.69 ± 0.28^{c}	0.34 ± 0.15^{b}	0.15 ± 0.09^{ab}	0.08 ± 0.04^{ab}	green
18	1-ethenyl-3-ethylbenzene	0.09 ± 0.02	-	-	-	-	-
19	(E)-2-decanal	-	0.24 ± 0.09^{a}	0.52 ± 0.17^{ab}	0.61 ± 0.36^{b}	-	fatty
20	Benzaldehyde	7.55 ± 1.48^{a}	9.05 ± 2.10^{ab}	13.07 ± 2.42^{c}	12.07 ± 2.65^{bc}	8.97 ± 2.25^{ab}	almond, burnt sugar
21	(E)-2-nonenal	-	0.83 ± 0.16^{a}	2.11 ± 0.37^{c}	1.75 ± 0.48^{b}	-	fatty
22	5-methyl-2-furancarboxaldehyde	0.71 ± 0.24^{a}	1.59 ± 0.34^{b}	0.70 ± 0.28^{a}	0.33 ± 0.16^{a}	0.59 ± 0.14^{a}	almond, sweet
23	2-undecenal	-	-	12.37 ± 1.56^{c}	8.67 ± 1.38^{b}	3.65 ± 1.34^{a}	orange
24	3-phenylpropionaldehyde	0.52 ± 0.08	-	-	-	-	honey
25	2,4-decadienal	-	-	1.80 ± 0.11^{b}	2.52 ± 0.16^{c}	0.49 ± 0.12^{a}	oily
26	(E, E)-2,4-decadienal	-	0.82 ± 0.27	-	-	-	green
27	2-phenyl-2-butenal	-	0.64 ± 0.25^{a}	0.72 ± 0.36^{a}	0.91 ± 0.24^{a}	0.50 ± 0.17^{a}	floral
28	Cinnamaldehyde	1.73 ± 0.68^{b}	0.69 ± 0.21^{a}	1.01 ± 0.55^{ab}	1.09 ± 0.64^{ab}	0.41 ± 0.15^{a}	cinnamon
	Ketones						
29	2-nonanone	-	-	-	0.16 ± 0.05	-	sweet, fruity
30	3-octen-2-one	-	-	0.37 ± 0.09	-	-	fruity
31	2-decanone	-	-	1.21 ± 0.72^{b}	0.58 ± 0.15^{a}	-	fruity
32	3-nonen-2-one	-	0.07 ± 0.04	-	-	-	-
33	α-lonone	6.78 ± 2.74	-	-	-	-	sweet, rose-like
	Esters						
34	Ethyl acetate	18.68 ± 1.38^{b}	17.27 ± 1.25^{b}	17.07 ± 1.92^{b}	21.93 ± 1.85^{c}	2.49 ± 0.85^{a}	pineapple
35	Ethyl butanoate	-	-	0.16 ± 0.08	-	-	pineapple
36	3-methylbutyl acetate	7.95 ± 1.12^{b}	2.94 ± 0.96^{a}	2.89 ± 0.922^{a}	3.18 ± 0.86^{a}	-	banana
37	Ethyl pentanoate	-	-	-	1.35 ± 0.73	-	floral
38	Hexyl butyrate	0.17 ± 0.06	-	-	-	-	fruity
39	Ethyl hexanoate	1.91 ± 0.37^{a}	5.95 ± 1.09^{bc}	7.27 ± 1.46^{c}	7.08 ± 1.88^{c}	4.47 ± 1.52^{b}	fruity
40	Ethyl hydrogen succinate	-	0.44 ± 0.18^{a}	0.63 ± 0.24^{b}	0.49 ± 0.14^{a}	-	floral
41	Ethyl heptanoate	0.40 ± 0.14^{a}	2.33 ± 0.53^{b}	3.50 ± 0.98^{b}	5.93 ± 0.92^{c}	3.50 ± 0.94^{b}	floral
42	Methyl 2,2-dimethoxyacetate	6.36 ± 2.15^{a}	6.22 ± 2.06^{a}	9.13 ± 3.52^{b}	6.07 ± 2.04^{a}	9.56 ± 3.49^{b}	fruity
43	Hexyl formate	-	-	-	-	0.87 ± 0.15	rose-like
44	Ethyl octanoate	25.55 ± 3.84^{b}	18.58 ± 4.49^{a}	50.92 ± 4.28^{d}	41.26 ± 4.31^{c}	23.50 ± 5.17^{b}	pineapple
45	3-Octenoic acid, ethyl ester	-	-	-	-	0.18 ± 0.05	-
46	Ethyl sorbate	0.25 ± 0.09	-	-	-	-	-
47	Ethyl nonanoate	-	5.92 ± 2.38^{a}	37.98 ± 4.59^{d}	24.72 ± 4.68^{c}	12.35 ± 3.27^{b}	rose-like
48	Ethyl dl-2-hydroxycaproate	7.10 ± 2.68^{a}	7.61 ± 2.35^{a}	8.26 ± 2.57^{a}	6.08 ± 2.74^{a}	6.77 ± 2.62^{a}	floral
49	Isobutyl n-octanoate	-	-	1.31 ± 0.32^{b}	0.69 ± 0.13^{a}	-	-
50	Ethyl decanoate	22.23 ± 7.13^{a}	45.45 ± 7.36^{b}	147.61 ± 9.72^{c}	90.02 ± 9.44^{c}	58.15 ± 7.50^{b}	ester
51	Ethyl benzoate	61.27 ± 6.24^{c}	26.0 ± 5.82^{a}	49.18 ± 5.33^{b}	47.02 ± 6.92^{b}	-	honey
52	Diethyl succinate	1.29 ± 0.45^{a}	3.06 ± 0.86^{ab}	4.65 ± 1.24^{b}	3.66 ± 1.08^{b}	3.09 ± 1.26^{ab}	fruity
53	Ethyl undecanoate	-	-	0.71 ± 0.26^{b}	0.30 ± 0.12^{a}	-	fatty
54	Phenethyl acetate	2.55 ± 0.75^{a}	1.76 ± 0.81^{a}	2.51 ± 0.72^{a}	2.42 ± 0.87^{a}	1.20 ± 0.79^{a}	rose/honey
55	Ethyl laurate	1.11 ± 0.26^{b}	0.17 ± 0.06^{a}	44.46 ± 2.32^{e}	31.79 ± 2.46^{d}	7.90 ± 2.34^{c}	leaf
56	3-methylbutyl pentadecanoate	-	-	3.68 ± 1.62^{c}	2.11 ± 1.34^{bc}	0.55 ± 0.21^{a}	-
57	Ethyl 3-phenylpropanoate	-	-	1.41 ± 0.58^{a}	1.30 ± 0.42^{a}	-	fruity, sweet
58	γ-nonalactone	-	0.23 ± 0.06^{ab}	0.32 ± 0.09^{b}	0.19 ± 0.05^{a}	-	coconut
59	Ethyl myristate	-	4.81 ± 2.08^{a}	18.36 ± 2.54^{d}	15.79 ± 3.82^{c}	9.35 ± 2.68^{b}	sweet, waxy
60	Diethyl suberate	-	-	0.55 ± 0.16^{b}	0.34 ± 0.13^{a}	-	-
61	Ethyl palmitate	0.21 ± 0.94 ^{a}	5.52 ± 1.27 ^{b}	19.81 ± 1.94^{d}	20.13 ± 2.35^{d}	15.44 ± 1.98^{c}	-
62	Ethyl 9-hexadecenoate	-	-	19.65 ± 3.24^{b}	18.05 ± 2.72^{b}	6.97 ± 3.17^{a}	honey
63	Ethyl oleate	-	2.87 ± 0.93^{a}	13.86 ± 2.51^{c}	15.55 ± 2.86^{c}	7.30 ± 2.18^{b}	-
64	Ethyl linoleate	-	-	1.67 ± 0.52^{b}	2.66 ± 0.63^{c}	0.50 ± 0.12^{a}	-

[1]"-": not detected. [2]Concentrations of these compounds in BL, CBN, CB1, CB3 and CB5, respectively. [3]Odorous description was obtained according to references Fan et al. (2015b); Fan & Qian (2006); Zhao et al. (2018). [a-d]Different superscripts within the same row indicated the significant differences ($P < 0.05$). BL (base liquor), BCN (base liquor soaking with new aged pork fat), BC1 (base liquor soaking with one-year aged pork fat), BC3 (base liquor soaking with three-year aged pork fat), BC5 (base liquor soaking with five-year aged pork fat).

Table 1. Continued...

NO.	Aroma compound	Concentration[2] (mg/kg)					Odorous Description[3]
		BL	CBN	CB1	CB3	CB5	
	Acids						
65	Octanoic Acid	-	5.34 ± 0.52 [b]	1.26 ± 0.58 [a]	0.88 ± 0.24 [a]	-	cheesy
66	Nonanoic acid	-	0.09 ± 0.07	-	-	-	-
	Alkanes						
67	Heneicosane	-	-	-	-	0.05 ± 0.02	-
68	Decane	-	0.12 ± 0.08	-	-	-	-
69	Copaene	-	-	5.14 ± 0.92 [c]	4.04 ± 0.95 [b]	0.79 ± 0.36 [a]	-
70	β-elemene	-	-	9.38 ± 1.32 [c]	3.01 ± 1.25 [b]	1.18 ± 0.96 [a]	spicy, fennel
71	β-caryophyllene	0.19 ± 0.05 [a]	-	2.73 ± 0.83 [b]	2.88 ± 0.95 [b]	-	woody, spice
72	Alloaromadendrene	-	-	9.77 ± 0.32[c]	1.43 ± 0.54 [b]	0.66 ± 0.27 [a]	-
73	Hexadecane	-	0.38 ± 0.06	-	-	-	-
74	1-ethenyl-4-methoxybenzene	2.04 ± 0.53	-	-	-	-	-
75	α-muurolene	-	-	2.65 ± 0.31 [b]	1.52 ± 0.36 [a]	-	-
	Furan						
76	2-pentylfuran	-	-	2.16 ± 0.23[c]	0.73 ± 0.29[a]	1.70 ± 0.18[b]	sweet, fruit

[1]"-": not detected. [2]Concentrations of these compounds in BL, CBN, CB1, CB3 and CB5, respectively. [3]Odorous description was obtained according to references Fan et al. (2015b); Fan & Qian (2006); Zhao et al. (2018). [a-d]Different superscripts within the same row indicated the significant differences ($P < 0.05$). BL (base liquor), BCN (base liquor soaking with new aged pork fat), BC1 (base liquor soaking with one-year aged pork fat), BC3 (base liquor soaking with three-year aged pork fat), BC5 (base liquor soaking with five-year aged pork fat).

and then decreased. CB1 had the highest contents, such as ethyl hexanoate (fruity), ethyl nonanoate (rose-like), ethyl octanoate (fruity), ethyl decanoate (ester), ethyl laurate (leaf) and ethyl myrisrate (sweet). It might be that more unsaturated fatty acids (UFA) in one-year aged pork fat were slowly decomposed and oxidized to corresponding acids, reacting with the ethanol in basic liquor to product more ethyl esters (Fan et al., 2019). These medium and long-chain ethyl esters played a positive role in characteristic volatile compounds of Chi-aroma Baijiu, and most of which were responsible for the typical fruity and floral aroma in Baijiu (He et al., 2021a). Accordingly, CB1 had better flavour than other samples, and this result was consistent with the results of the 3.1 sensory evaluation.

Alcohols played a flavouring role in Baijiu, which were produced by wine yeast through catabolic and synthetic pathways. Higher alcohols made the wine body mellow and harmonious, and had a positive effect on the aroma of the Chi-aroma Baijiu. Compared with 211.73 mg/kg of higher alcohols in BL, the amount in CB1 was the highest (310.90 mg/kg), while that in other Baijiu samples were lower. 3-methybutanol and 2-phenylethanol reached the highest concentrations in CB1, CB3 and CB5. These two alcohols were previously found in strong flavour Baijiu (He et al., 2022). β-phenylethanol was responsible of a strong rose flavour, which was the most important and symbolic flavour substance in Chi-aroma baijiu. Hence, it was a crucial component in generating the complex Chi-aroma along with other flavour substances. It was detected in all five wine samples, whereas β-phenylethanol contents in CB1 was the highest (34.38 mg/kg).

Aldehydes, especially unsaturated aldehydes, were oxidized from fat and this could contribute fruit and fat flavour to wine body (González-Rompinelli et al., 2013; Li et al., 2021; Li & Liu, 2022). Compared to BL, the aldehydes contents of other four Baijiu samples increased significantly. For instance, the aldehydes contents of CB1 (53.96 mg/kg) were the highest, followed by CB3 (43.19 mg/kg) and CBN (32.49 mg/kg), and BL (17.53 mg/kg) was the lowest. In addition, (E)-2-heptanal, nonanal, (E,E)-2,4-heptadienal, (E)-2-decanal, (E)-2-nonenal, and Heptanal were detected in CBN, CB1, CB3 and CB5, exhibiting intense fatty aroma (Fan et al., 2015a; Poisson & Schieberle, 2008), these have been regarded previously as one of the characteristic aromas of Chi-aroma Baijiu (Zhang et al., 2010). The data in Table 1 demonstrated that the highest concentrations of above six aldehydes in CB1 were the highest, up to 43.57 mg/kg. Ketones were detected in BL, CBN, CB1 and CB2. The total content of ketones in other wine samples was less than 2 mg/kg, except BL.

Acids could give baijiu an aftertaste (Lu et al., 2022). 2 fatty acids were detected in the CBN, CB1, CB3 and CB5, except for BL. The detected fatty acids included octanoic acid and nonanoic acid, which might have been caused by decomposition of unsaturated fatty acids in aged pork fat during the ageing process (Wei et al., 2020). In small amounts, fatty acid can contribute to a balanced aroma in liquors by hindering hydrolysis of their esters.

Alkanes were detected in the CB1, CB3 and CB5. Other volatile compounds were only detected in BL. The result showed other volatile compounds did not exist in the Chi-aroma Baijiu after soaking with aged pork fat, which might be related to adsorption of aged pork fat, making the wine clearer.

3.3 OAV of volatile compounds in Chi-aroma Baijiu

To further evaluate the contributions of odorants to the overall volatile compounds of Baijiu samples, the OAVs of all volatile compounds were calculated. Heat map illustrated the OAVs distribution of volatile compounds in Baijiu sample by a standard colour intensity ranging from maximum (dark red) to minimum (dark blue). Volatile compounds with OAV > 1 were confirmed to be the important flavour in Baijiu samples. As shown in Figure 4, the OAVs of volatile compounds in each baijiu samples were quite different ($P < 0.05$). Overall, 20 volatiles were found to have OAV > 1 in BL, while 23 volatiles in CBN, 34 volatiles in CB1, 34 volatiles in CB3 and 22 volatiles in the CB5. Thus, these volatile compounds were considered to contributors to the flavour of base liquor and Chi-aroma baijiu. And CB1 and CB3 had richer flavour profiles.

Among the volatile compounds with OAV > 1 of CBN, CB1, CB3 and CB5, esters and aldehydes accounted for the largest proportion. The highest OAV of aldehydes were (E)-2-nonenal,

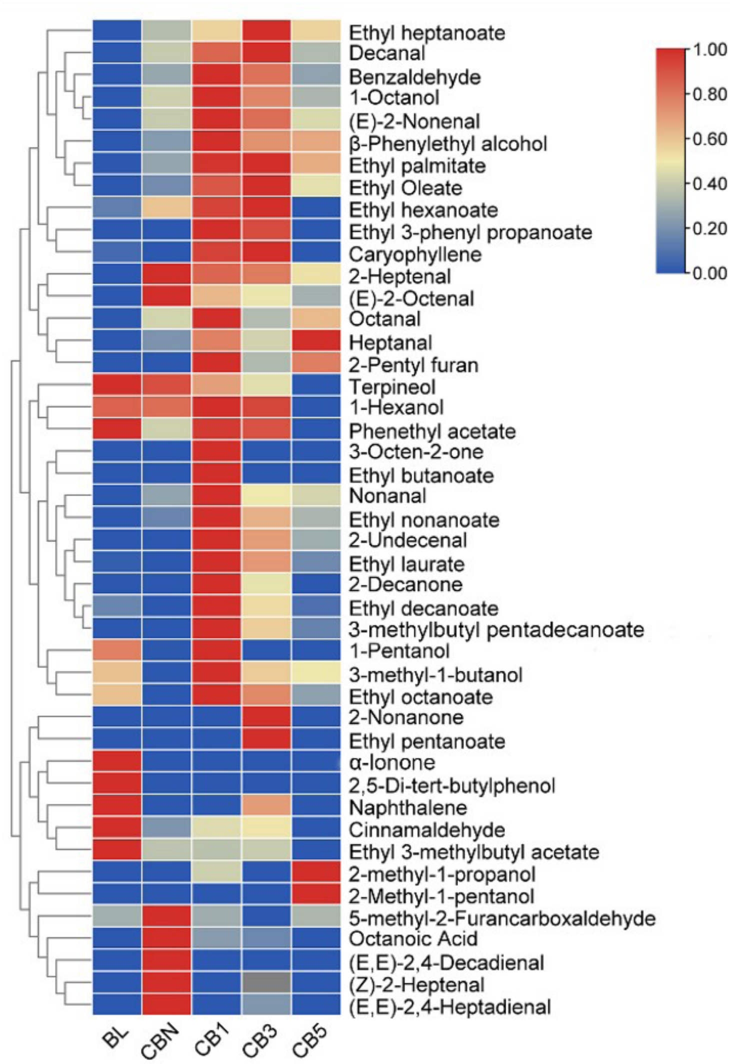

Figure 4. Heat maps of OAVs of different Baijiu samples.

followed by octanal, (E)-2-decanal, (E)-2-octenal, heptanal and (E)-2-heptenal. CB1 was much higher than other Baijiu samples in the OAVs of (E)-2-nonenal (32462), octanal (3057) and (E)-2-decanal (124), which contributed to fatty flavour (Willner et al., 2013). Researchers had indicated that (E)-2-nonenal, (E)-2-decenal, (E)-2-octenal were detected with OAVs > 1 in chi-aroma Baijiu (Fan et al., 2015a). (E)-2-nonenal, octanal, (E)-2-decanal, (E)-2-octenal and octanal were formed by lipid oxidation (Vanderhaegen et al., 2006). Aged pork fat contained abundant lipids, which could lead to generation of these aldehydes during ageing process. In addition, as the year of aged pork fat increased, the OAVs of medium and long-chain ethyl esters increased and then decreased. CB1 had the highest OAVs of ethyl esters, such as ethyl hexanoate (OAV = 14160), ethyl octanoate (OAV = 51), ethyl decanoate (OAV = 1315), ethyl heptanoate (OAV = 20.59), ethyl laurate (OAV = 13), ethyl nonanoate (OAV = 32) and ethyl palmitate (OAV = 13). Interestingly, ethyl hexanoate and ethyl octanoate were also identified as important odour-active compounds in Baofengjiu, Fenjiu and Qingkejiu liquors (Gao et al., 2014). Therefore, it might be believed that medium and long-chain ethyl esters and four aldehydes ((E)-2-nonenal, octanal, (E)-2-decanal, (E)-2-octenal) were the characteristic volatile compounds of Chi-aroma Baijiu during ageing process owing to their OAV > 10. Besides, CB1 have the intense Chi-aroma due to their higher OAV of medium and long-chain ethyl esters and four aldehydes.

3.4 Multivariate statistical analysis

OPLS-DA was performed on the volatile compounds of five Baijiu samples, and quantified the differences among the Baijiu samples resulting from characteristic volatile compounds (Galtier et al., 2011). The values of R^2 (Y) and Q^2 were used to evaluate the applicability and predictability of the model, respectively (He et al., 2021a). The model obtained a goodness-of- fit of 99.3% (R^2Y (cum) = 0.993) and a goodness-of-prediction of 88.5% (Q^2 (cum) = 0.885). The high values of R^2 (Y) and Q^2 indicated that the model was reliable and had no overfitting phenomenon. The score plot (Figure 5A) revealed the five Baijiu samples were divided into three groups. The results indicated

CBN, CB1 and CB3 had significant differences in the volatile compounds, comparing with BL.

In order to further analyse the characteristic volatile compounds causing difference among Chi-aroma baijiu soaking with different years of aged pork fat, CBN, CB1, CB3 and CB5 were used as Y variables. OPLS-DA model (Figure 5B) revealed the four Chi-aroma Baijiu were divided into four groups, indicating this model could be used to distinguish the different Chi-aroma Baijiu soaking with different years of aged pork fat. An S-line diagram could be used to analyse the marker volatile compounds causing difference among samples. As shown in Figure 5B, 1-pentanol, 3-methylbutanol, 2-undecenal, ethyl octanoate, ethyl nonanoate, ethyl decanoate, ethyl benzoate, ethyl laurate, ethyl myristate, ethyl palmitate, ethyl oleate and ethyl 9-hexadecenoate were the most significant indexes (p(corr) ≥ 0.5).

Additionally, VIP was taken to distinguish the most outstanding volatile compounds (Kosek et al., 2019), and volatile compounds with VIP ≥ 1 were thought to have a remarkable effect on the identification of Chi-aroma baijiu (Ye et al., 2022). As shown in Figure 5C, a total of 31 volatile compounds could function as indicators of Chi-aroma soaking with different years of aged pork fat, such as ethyl heptanoate, ethyl palmitate, ethyl linoleate, ethyl oleate, 3-octen-2-one, (E, E)-2,4-heptadienal, nonanoic acid, octanoic acid, et.

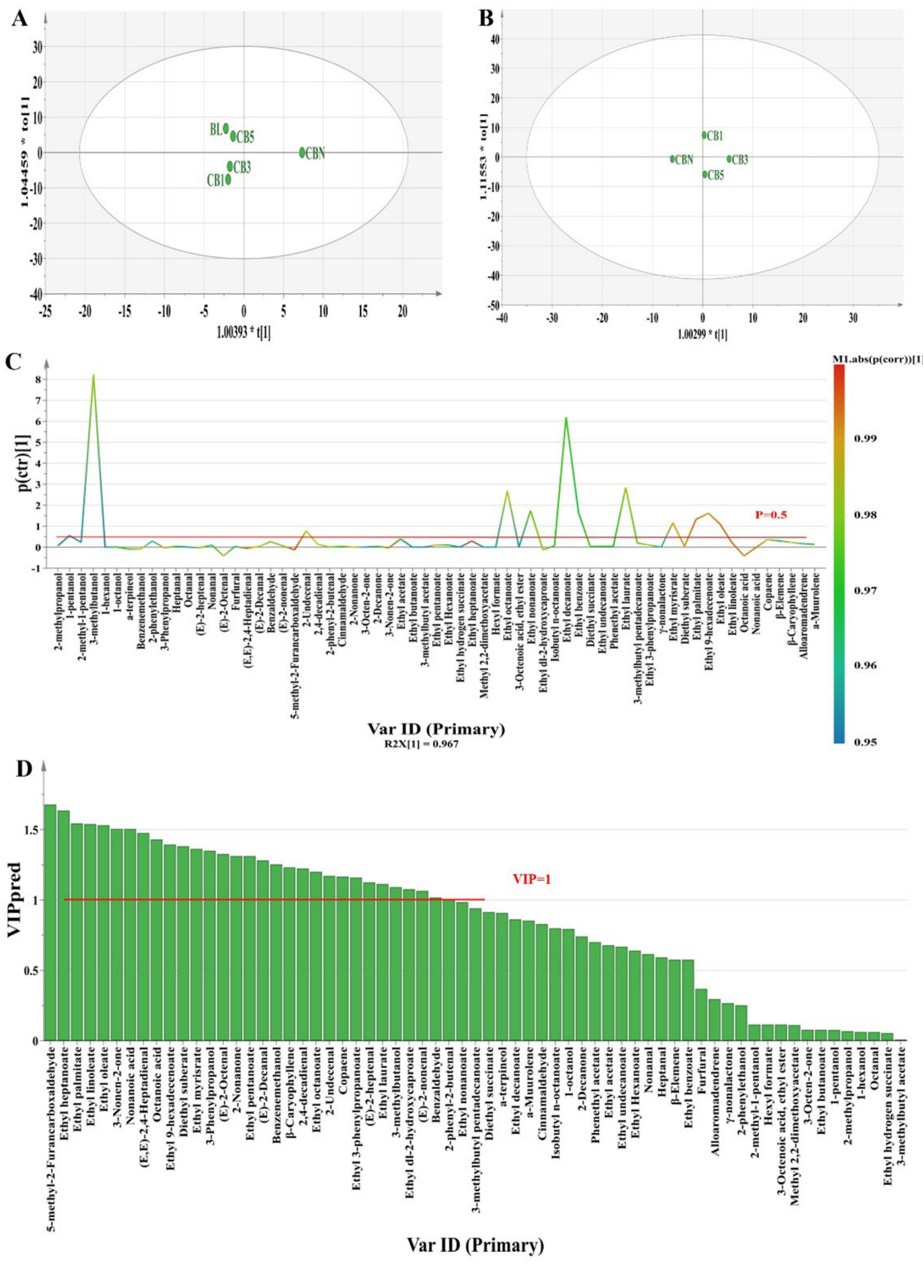

Figure 5. OPLS-DA of the detected volatile compounds, score plot of OPLS-DA of five Baijiu samples (A) and four Baijiu samples (B), the S-line (C) and the VIP (Variable Importance in Projection) plot (D).

4 Conclusion

In this study, the changes of the quality and volatile compounds in Chi-aroma Baijiu soaking with different aged pork fats were evaluated. The result showed that the flavour and taste of CB1 were the best. With increasing years of aged pork fat, the glycerol and total ester contents increased gradually, but the total acid content did not change significantly ($P > 0.05$). The composition of volatile compounds in each Baijiu samples was quite different after ageing. 36 volatile compounds were detected in the BL, 44 volatile compounds in CBN, 58 volatile compounds in the CB1, 55 volatile compounds in the CB3, and 42 volatile compounds in the CB5, indicating volatile compounds in CB1 were significantly enriched. Medium and long-chain ethyl esters were the most important volatile compounds of Chi-aroma Baijiu during ageing process owing to their higher OAV. Moreover, OPLS-DA could effectively distinguish different Chi-aroma Baijiu soaking with different years of aged pork fat, and the model was both adaptable and predictable.1-pentanol, 3-methylbutanol, 2-undecenal, ethyl octanoate, ethyl nonanoate, ethyl decanoate, ethyl benzoate, ethyl laurate, ethyl myristate, ethyl palmitate, ethyl oleate and ethyl 9-hexadecenoate were the most significant indexes. Combined with the results, one-year aged pork fat was the best year to product Chi-aroma Baijiu.

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