Comparison of the Volatile Components in Chinese Traditional Xiaoqu Liquor

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Abstract: Volatile components in Sichuan Xiaoqu liquor of Chongqing Jiangjin District, Chongqing Yongchuan District, Sichuan Kaijiang County, Sichuan Zigong were studied by the method of combining headspace solid-phase microextraction (HS-SPME) with gas chromatography coupled with mass spectrometry (GC-MS). Results showed that 29, 31, 44, 45 kinds of aromatic components were identified from the Xiaoqu liquor of Jiangjin, Chongqing Yongchuan, Kaijiang and Zigong respectively. Esters, alcohols as well as aldehydes and ketones are the main substances. According to peak area relative percentage contents, content of ethyl acetate, isoamyl alcohol and ethyl caprate in the 4 kinds of Xiaoqu liquor is relatively high.

Keywords: Sichuan xiaoqu liquor, Headspace solid-phase microextraction, Gas chromatography-mass spectrometer, Volatile components.

INTRODUCTIONS

Sichuan solid–state Xiaoqu liquor has a long history of brewing, typical style, as well as unique flavor, forming three types [1-2] of Fen –flavor liquor together with Daqu Fen –flavor and Fuqu liquor. In recent years, with development of alcoholic beverage, flavor of people turns to be delicate and elegant, Xiaoqu Fen –flavor liquor just conforms to such trend, more and more people drink, and it may stand out from China liquor again [3]. “Sichuan Xiaoqu Liquor” comes from high yield of Xiaoqu liquor in Sichuan region every year, the annual yield of only Sichuan and Chongqing is 200 ~ 300 thousand KL [4-6].

Research of Sichuan Xiaoqu liquor mainly focuses on raw materials of production, koji, technical route [2]. Analysis of aromatic components of products have been seldom researched, therefore, figuring out the flavoring rules of aromatic components of Sichuan Xiaoqu liquor is conducive to promotion of quality and further promotion of Sichuan Xiaoqu liquor as well as further development of Sichuan Xiaoqu liquor enterprises.

Four kinds of Sichuan xiaoqu liquor, which are Chongqing, Dazhou, Yongchuan and Zigong Xiaoqu liquor, are the representative of Fen –flavor liquor in China, and the 4 kinds of liquor selected as experiment materials This research adopts HS-SPME technology [7] which features [8] short operation time, small usage of samples, rapid and easy application, no need of solvent and good reproducibility [9-10], so this method is chosen for pretreatment of samples. On the basis of referring to analysis and relevant research on components of liquor performed by Li Dahe, Xu Yan, Zeng Zuxun, Shen Yifang, *et al*. [5, 11-16], analyze Sichuan Xiaoqu liquor rapidly and easily with HS-SPME and GC-MS combination method, and perform preliminary discussion on aromatic components rules of Fen –flavor liquor according to results for the purpose of figuring out aromatic components rules of Sichuan Xiaoqu liquor and providing theory reference for further research on aromatic components of Sichuan Xiaoqu liquor.

MATERIALS AND METHODS

Materials

Commercially available Xiaoqu liquor of Chongqing Jiangjin District, Chongqing Yongchuan District, Sichuan Kaijiang County, Sichuan Zigong (alcohol content: 50, 63, 52 and 62% vol, respectively) is the test sample.

Angilent 6890 GC and 5975 MS; chromatographic column DB-WAX 60 m x 250 μm x 0.25 μm; SPME manual sampling handle and extraction head, purchased from ANPEL Scientific Instrument Co., Ltd. Wherein, the extraction head is 50/30UM DVB/CAR on PDMS, mansufactured by America Supelco. Sodium chloride (analytical pure, over 99.5%), Chengdu Kelong Chemical Reagent Company.

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Gas Chromatography and Mass Spectrum Conditions

Chromatographic column is DB-WAX 60 m x 250 μm x 0.25 μm, and temperature of sample inlet was 250 °C; temperature raising procedure of column box: keep 35 °C for 5min, rise to 100 °C at a speed of 5 °C/min, and keep 2 min, at last, rise to 230 °C at a speed of 15 °C/min, and keep 10 min, operation time 38.667 min. Carrier gas: high –purity helium, carrier gas flow rate: 1 ml/min.

Mass spectrum conditions: EI ion source, with temperature of 230 °C, temperature of interface 250 °C, ionization voltage 70 ev, temperature of quadruple rod 150 °C, mass scanning range m/z: 30 ~ 500. Determination of aromatic components: determine aromatic components through comparison with NIST05a.L spectrum base.

Operation Method of HS-SPME

Put 9 mL (20% V/V) liquor sample after ultrapure water dilution as well as 0.9 g of sodium chloride in 20 mL headspace bottle, place the bottle on 45 °C thermostat water bath for 30 min, then put the aging extraction head (aging temperature 230 °C, aging time 30 min) into the headspace bottle for extraction of 50 min, distance from the extraction head and liquid level was 1 cm, and then conduct manual sampling of the aging extraction head on GC-MS for chromatographic analysis.

RESULTS AND DISCUSSIONS

Chromatogram of 4 Kinds of Sichuan Xiaoqu Liquor

Deploy treatment and GC/MS analysis on samples following the above test operation methods and conditions, and get the total ions chromatogram of certain Xiaoqu liquor of Chongqing Jiangjin District, Chongqing Yongchuan District, Sichuan Kaijiang County and Sichuan Zigong, see Figure **1**, Figure **2**, Figure **3** and, Figure **4**, respectively.

The total ions chromatogram of the above 4 samples has relatively high peak separation degree and relatively good separation effect, without tailing, overlap and base line inequality, through comparison with NISTO5a.L database and analysis, 29, 31, 44 and 45 kinds of trace aromatic components are identified in the Xiaoqu liquor of Jiangjin, Yongchuan, Kaijiang and Zigong, see Table **1** for aromatic components.

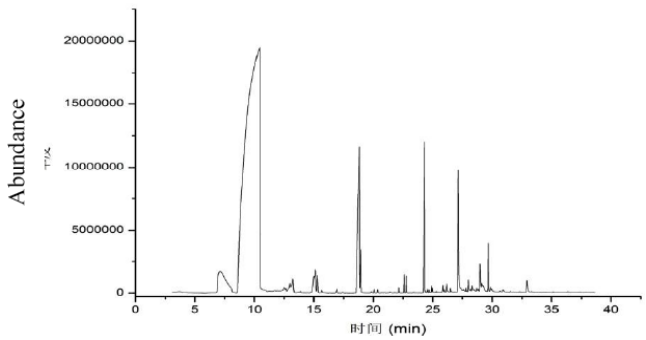

Figure 1: Total ions chromatogram of Jiangjin Xiaoqu Liquor.

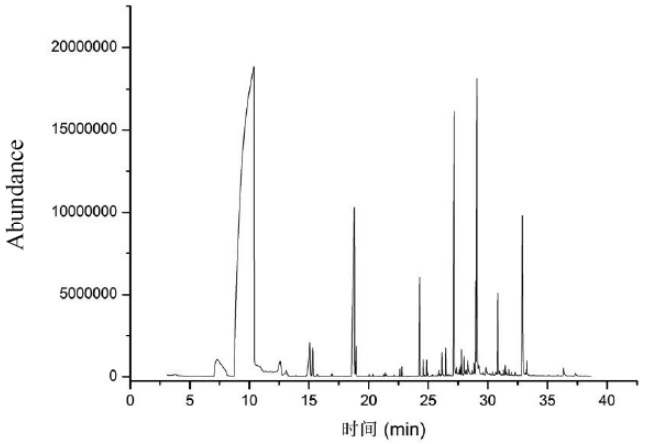

Figure 2: Total ions chromatogram of Yongchuan Xiaoqu Liquor.

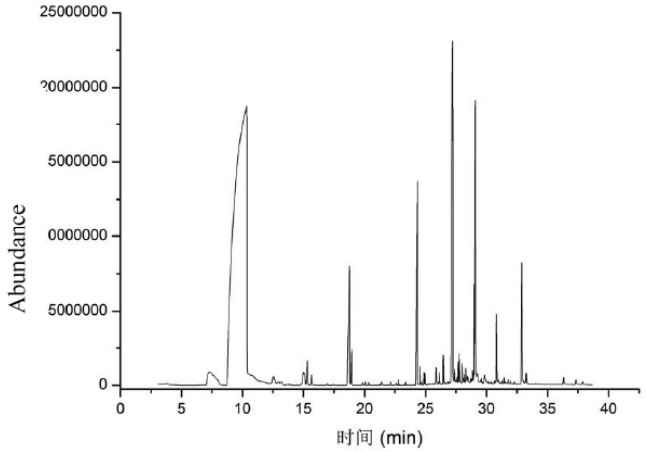

Figure 3: Total ions chromatogram of Kaijiang Xiaoqu Liquor.

Contrastive Analysis of Aromatic Components of 4 Kinds of Sichuan Xiaoqu Liquor

Analysis results show that 29 aromatic components are identified for Jiangjin Xiaoqu liquor (except ethyl alcohol and other components with low similarity), including 15 kinds of esters, 9 kinds of alcohols, 2 kinds of aldehydes and ketones as well as 3 kinds for others. Among aromatic components, esters and alcohols are

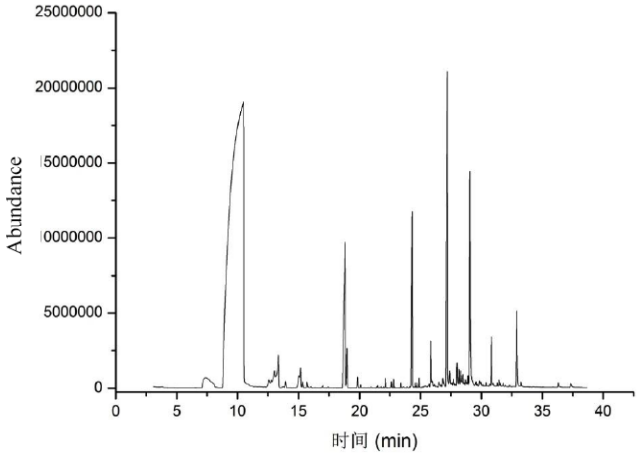

Figure 4: Total ions chromatogram of Zigong Xiaoqu Liquor.

the main substances, with isoamyl alcohol accounting for 28.45%, 19.61% for ethyl acetate accounting, 10.54% for ethyl caprylate, 10.04% for ethyl caprate, 5.04% for isobutanol, 3.37% for ethyl caproate, and 2.58% for n-propyl alcohol.

31 aromatic components are identified for Yongchuan Xiaoqu liquor, including 19 kinds of esters, 7 kinds of alcohols, 4 kinds of aldehydes and ketones as well as 1 kind for others. Esters are the main aromatic components, accounting for up to 65.47%, and alcohols take the second place, accounting for 25.53%. Among aromatic components, relative percentage is from high to low: isoamyl alcohol accounting for 19.43%, 17.71% for ethyl caprate, 13.80% for ethyl laurate, 9.93% for ethyl acetate, 8.83% for ethyl palmitate, etc.

41 aromatic components are identified for Kaijiang Xiaoqu liquor, including 28 kinds of esters, 5 kinds of alcohols, 2 kinds of aldehydes and ketones as well as 6 kinds for others. In this sample liquor, relative content percentage of esters is up to 81.22%, far more than that of alcohols which account for 15.42%. Among aromatic components, relative percentage is from high to low: 27.17% for ethyl caprate, 14.29% for ethyl laurate, 12.42% for ethyl caprylate, 11.55% for isoamyl alcohol, 6.97% for ethyl palmitate, 5.63% for phenethyl acetate, 3.13% for phenethyl acetate, etc. 45 aromatic components are identified for Zigong Xiaoqu liquor, including 22 kinds of esters, 7 kinds of alcohols, 5 kinds of aldehydes and ketones as well as 11 kinds for others. Among aromatic components, esters account for 63.66%, and alcohols account for 29.42%. Relative percentage is from high to low: 24.34% for ethyl caprate, 17.61% for isoamyl alcohol, 12.18% for ethyl laurate, 9.42% for ethyl caprylate, 7.32% for n-propyl alcohol, 4.89% for ethyl palmitate, etc.

In liquor of the 4 regions, substances with comparatively large relative content percentage include ethyl caprate, isoamyl alcohol, ethyl laurate, ethyl palmitate, phenethyl acetate, isobutyl alcohol and ethyl caprylate. Such substances have large proportion in terms of content of Sichuan Xiaoqu liquor, especially ethyl caprate and isoamyl alcohol. Besides, common aromatic components for the 4 kinds of Xiaoqu liquor are: phenethyl acetate, isoamyl alcohol, ethyl caproate, ethyl lactate, ethyl caprylate, ethyl caprate, ethyl laurate, ethyl myristate, ethyl palmitate, n-propyl alcohol, isobutyl alcohol, isoamyl alcohol, n-amyl alcohol, n-caprylic alcohol and furfural, 15 kinds in total, including 9 kinds of esters, 5 kinds of alcohols, and 1 kind of aldehydes. All 4 kinds of Xiaoqu liquor contain furfural which is one of the harmful products in food manufacturing process, Maillard reaction of food, caramelization reaction; thermal processing course and high temperature sterilization process provide good generation conditions for furfural [17]. However, content of furfural in Maotai–flavor liquor is highest among all China liquor [1]. Therefore, determination of mass concentration and research on function of furfural in Sichuan Xiaoqu liquor make more sense.

According to comparative analysis, percentage of content of phenethyl acetate, isoamyl alcohol, isobutyl alcohol and ethyl caproate in Jiangjin Xiaoqu liquor is higher than that in other three regions, and category and total percentage of alcohols are also higher than other 3 regions. According to current market share and world of mouth of ordinary people, the delicate and elegant quality of Jiangjin Xiaoqu liquor may be reflected in one of such substances, interaction of several substances or other substances. On the whole, compared with Daqu Fen –flavor liquor, Fen –flavor Sichuan Xiaoqu liquor has fewer kinds of aromatic components [18]. In Fenjiu liquor, representative of Daqu Fen –flavor liquor, 10 kinds of aromatic substances which contribute much to aromatic components system of Fenjiu liquor are identified, including ethyl caprylate, isoamyl alcohol, n-caprylic alcohol, phenethyl alcohol and more, and they are initially identified as characteristic aromatic substances of Daqu Fen –flavor liquor [19]. Test data show that Sichuan Xiaoqu liquor also contains ethyl caprylate, isoamyl alcohol, n-caprylic alcohol and phenethyl alcohol, and content of ethyl caprylate and isoamyl alcohol is not small. Therefore, contribution of such substances to aroma system of Sichuan Xiaoqu liquor may also be relatively great.

Table 1: Aromatic Components of four Kinds of Commercially Available Fen-Flavor Sichuan Xiaoqu Liquor and Relative Contents

Aroma Components			Jiangjing Liquor				Yongchuan Liquor				Kaijiang Liquor				Zigong Liquor			
			29 kinds				31 kinds				44 kinds				45 kinds			
	No	Components	Similarity %	Relative Percentage (%)	%	kinds	Similarity %	Relative percentage	%	kinds	Similarity %	Relative percentage	%	kinds	Similarity %	Relative percentage	%	kinds
Esters	1	Ethyl acetate	95	19.61	50.52	15	90	9.93	65.47	19	97	5.63	81.22	28	93	0.77	63.66%	22
	2	Isoamyl acetate	90	1.70			90	1.94			90	1.71			87	0.47		
	3	Isovaleric acid ethyl ester	-	-			-	-			-	-			88	0.40		
	4	Ethyl caproate	90	3.37			93	1.42			93	1.83			83	2.21		
	5	Heptanoic acid ethyl ester	87	0.24			-	-			82	0.14			87	0.36		
	6	L (-) - ethyl lactate	87	0.89			87	0.26			87	0.10			87	0.27		
	7	Octanoic acid ethyl ester	90	10.54			88	3.72			87	12.42			80	9.42		
	8	Caproic acid isoamyl acetate	97	0.15			-	-			87	0.13			85	0.17		
	9	Pelargonic acid ethyl ester	-	-			-	-			93	0.77			93	2.29		
	10	Decanoic acid ethyl ester	97	10.04			96	17.71			96	27.17			96	24.34		
	11	Trans - 4 - decanoic acid ethyl ester	-	-			-	-			94	0.16			96	1.03		
	12	Ethyl butyrate	-	-			-	-			90	1.10			-	-		
	13	Succinic acid diethyl ester	-	-			-	-			81	0.09			62	0.14		
	14	Bitterness 3 - methyl butyl ester	-	-			-	-			90	0.92			-	-		
	15	octylic acid butyl ester	-	-			-	-			86	0.11			-	-		
	16	phenyl ethyl propionate	-	-			-	-			-	-			87	0.35		
	17	Twelve acid ethyl ester	98	1.79			97	13.80			97	12.29			96	12.18		
	18	3 - styrene acrylic acid ethyl ester	-	-			95	0.11			80	0.23			94	0.20		
	19	Tridecane acid ethyl ester	-	-			-	-			-	-			84	0.26		
	20	Myristic acid ethyl ester	96	0.07			91	2.68			95	2.38			96	2.31		
	21	Fifteen acid ethyl ester	-	-			95	0.69			93	0.48			94	0.48		
	22	Palmitic acid ethyl ester	97	1.25			96	8.83			97	6.97			96	4.89		
	23	9-16 carbon olefine acid ethyl ester	-	-			99	0.10			97	0.50			94	0.27		
	24	Ethyl oleate	-	-			99	0.67			99	0.56			99	0.40		
	25	Octadecane diene acid ethyl ester	-	-			99	0.26			99	0.42			99	0.43		
	26	hexyl acetate	87	0.24			87	0.11			86	0.14			-	-		
	27	octyl acetate	91	0.28			87	0.14			-	-			-	-		
	28	DL - 2 - hydroxy - 4 - methyl - pentanoic acid ethyl ester	87	0.12			-	-			-	-			-	-		
	29	decyl acetate	-	-			91	0.23			86	0.65			-	-		
	30	ethyl benzoate	87	0.23			-	-			-	-			-	-		

	31	phenethyl acetate	-	-			88	2.47			87	3.13			-	-		
	32	2, 8 dimethyl decanoic acid methyl ester	-	-			-	-			90	0.08			-	-		
	33	diisobutyl phthalate	-	-			-	-			80	0.23			-	-		
	34	9 - decyl dilute acid ethyl ester	-	-			87	0.40			84	0.91			-	-		
	1	sec-butyl alcohol	90	0.37			-	-			82	0.13			86	0.85		
	2	normal propyl alcohol	91	2.58			91	0.76			90	0.20			91	7.32		
	3	isobutanol	96	5.04			96	4.09			95	2.31			95	3.13		
	4	n-butyl alcohol	87	0.35			86	0.16			-	-			93	0.20		
alcohols	5	isoamyl alcohol	99	28.45	38.99	9	90	19.43	25.53	7	99	11.55	15.42	5	99	17.61	29.42	7
	6	1-Pentanol	97	0.23			92	0.09			92	0.17			90	0.18		
	7	n-caprylic alcohol	91	0.59			91	0.71			91	0.39			86	0.12		
	8	phenethyl alcohol	92	0.35			-	-			95	0.51			-	-		
	9	hexyl alcohol	90	0.72			86	0.29			90	0.16			-	-		
	1	furfural	95	0.31			95	0.48			95	0.39			95	0.31		
	2	Nonanal diethyl acetal	-	-			-	-			-	-			89	0.09		
	3	Methylnonylketone	-	-			-	-			-	-			80	0.13		
Carbonyl	4	2-tridecanone	-	-	0.73	2	-	-	1.03	4	-	-	0.49	2	93	0.19	0.91	5
	5	2,6,10-trimethyl-14-pentadecanone	-	-			92	0.15			95	0.10			91	0.19		
	6	2-oxopentadecane	-	-			89	0.17%			-	-			-	-		
	7	benzaldehyde	89	0.43			91	0.24%			-	-			-	-		
	1	riethylene glycol monoethyl ether	-	-			-	-			-	-			87	1.06%		
	2	1,1-diethoxy-3-methyl-Butane	83	0.14			87	0.09%			-	-			84	0.46%		
	3	styrol	-	-			-	-			96	0.15%			96	0.68%		
	4	1,1,3 - triethoxy propane	-	-			-	-			-	-			91	0.12%		
	5	Dimethyl trisulfide	-	-			-	-			97	0.11%			95	0.19%		
	6	1,1-diethoxy-Heptane	-	-			-	-			-	-			83	0.13%		
others	7	1 - tetradecene	-	-	4.68	3	-	-	0.09%	1	-	-	1.09%	6	81	0.30%	4.75%	11
	8	1 - caryophyllene	-	-			-	-			99	0.14%			99	0.72%		
	9	cadinene	-	-			-	-			93	0.22%			94	0.48%		
	10	2-methylnaphthalene	-	-			-	-			-	-			94	0.19%		
	11	1-Methylnaphthalene	-	-			-	-			91	0.15%			94	0.43%		
	12	N - (2 - phenyl ethyl) acetamide	87	2.09			-	-			-	-			-	-		
	13	Butylated hydroxytoluene	96	2.02			-	-			-	-			-	-		
	14	nye	-	-			-	-			93	0.32%			-	-		
	-	-	< 80	-	5.07		< 80	-	7.88%	-	< 80	-	1.78%	-	< 80	-	1.26%	-

In the Sichuan Xiaoqu liquor of these 4 regions, because of regional difference, different water for brewing, as well as different water quality, microbial metabolism variation may arise during fermentation, thus leading to difference of aromatic components in Xiaoqu liquor. Jiangjin region is located in upper and middle Yangtze River, the unique natural environment creates the microorganism community here, and water quality as well as climate also becomes important factors for brewing of quality liquor. Such factors may be the reason for hither contents of phenethyl acetate, isoamyl alcohol, and isobutyl alcohol and ethyl

caproate among aromatic components of Xiaoqu liquor in this region. Second, sorghum is the raw material for the 4 kinds of liquor, but taste and flavor of Xiaoqu liquor produced with sorghum of different producing areas and different varieties may also vary. The liquor produced with glutinous sorghum of Sichuan is fragrant, but the local glutinous sorghum is of low yield, and most glutinous sorghum is directly purchased by large liquor enterprises, and it is hard to buy for small breweries, most of which produce Xiaoqu liquor with northern red sorghum. As for production technology, all adopt traditional production technology, there may be slight difference for different liquor enterprises, but all produce liquor strictly following production process of Xiaoqu liquor, so influence in this aspect is relatively small. However, the process for production is easily affected by natural environment, especially the temperature, which is likely to be a reason influencing quality of Xiaoqu liquor.

CONCLUSION

Analysis of aromatic components of Sichuan Xiaoqu liquor in 4 regions was made through HS-SPME, combining with GC-MS, and totally 64 kinds of aromatic components were finally identified, including 29 kinds for Jiangjin Xiaoqu liquor, 31 kinds for Yongchuan Xiaoqu liquor, 41 kinds for Kaijiang Xiaoqu liquor and 45 kinds for Zigong Xiaoqu liquor, there are up to 15 kinds of common components for the 4 kinds of Sichuan Xiaoqu liquor, with esters and alcohols are the main aromatic components. In the 4 kinds of Xiaoqu liquor, substances with comparative large relative content include ethyl caprate, isoamyl alcohol, ethyl laurate, ethyl palmitate, ethyl acetate, isobutyl alcohol and ethyl caprylate, especially isoamyl alcohol, ethyl acetate and ethyl caprate.

Fen –flavor Xiaoqu liquor is of short production cycle, high yield, light taste and certain fermented grains flavor, which is the unique feature of Xiaoqu liquor. However, due to short originals storage period, deficient blending technology, poor flavor, low price as well as market restriction, the additional value is low, so it is different to sell in high –end market, so it is only considered to be low –end product. In order to break through the bottleneck, researchers may extend the range of raw materials, for example, mixed grains fermentation of corn, wheat and rice, or certain aroma producing yeast may be added, and thus the flavor and taste of Xiaoqu liquor may be improved. Aroma extraction of this research focuses on esters, alcohols as well as aldehydes and ketones, acids, alkanes, aromatic, furans and acetals are not identified, and further study is needed.

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