



Characterization of the key aroma compounds in different light aroma type Chinese liquors by GC-olfactometry, GC-FPD, quantitative measurements, and aroma recombination



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ABSTRACT

The aroma profiles of five Chinese light aroma-type liquors were investigated in this study. Gas chromatography-olfactometry (GC-O) and GC-flame photometric detection (FPD) were employed to investigate aroma-active compounds. A total of 80 odorants were identified, including ten sulfur compounds. In addition, aroma-active compounds ($FD \geq 16$) were determined, according to quantitative study and odor activity values (OAVs). Finally 27 key aroma compounds, mainly possessing fruity and floral notes, were successfully used to simulate the aromas of Chinese light aroma-type liquors by dissolving them in 53% (v/v) hydroalcoholic solution in their natural concentrations. Moreover, the correlation between the key aroma compounds, seven sensory attributes and liquor samples was evaluated through partial least-squares regression analysis.

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1. Introduction

Chinese liquor is a distilled alcoholic beverage of great reputation, consumed worldwide especially in China (Du, Fan, & Xu, 2011). Due to the great variety of production processes, Chinese liquors from different manufacturers have significant differences in aroma profile. On the basis of these aroma characteristics, they are generally classified into the following categories: light aroma type, strong aroma type, soy sauce aroma type, sweet and honey aroma type, and miscellaneous aroma type liquors (Xu, Wang, Fan, Mu, & Chen, 2010). Among them, light aroma type liquor is widely welcomed by consumers in southern China. In general, the main raw materials used for light aroma type liquor production are sorghum, barley, and pea: the former is used for making liquor, and the latter two are used for making qu. Qu, which is made of pulverized barley and pea, is usually used as a fermentation starter to initiate the fermentation process (Fan & Xu, 2000). Light aroma type liquors should be colorless, clear and transparent, with no suspension and no precipitation, pure in fragrance, with the elegant and harmonious aroma of ethyl acetate.

The contribution of key aroma compounds in Chinese liquors has been investigated previously. Nie Qingqing et al. (Qingqing,

Wenlai, & Yan, 2011) found that ethyl hexanoate was the most important aroma compound in mellow aroma-type liquors (TianZhiLan liquor, MengZhiLan liquor and HaiZhiLan liquor), followed by hexanoic acid, ethyl butanoate, dimethyl trisulfide, trimethylpyrazine and γ -nonalactone. These key aroma compounds were identified by using gas chromatography-olfactometry (GC-O) and gas chromatography-mass spectrometry (GC-MS) after liquid-liquid extraction (LLE). Haiyan, Wenlai, and Yan (2015) identified 34 key odorants according to quantitative study and odor activity values (OAVs), including ethyl octanoate, hexanal and ethyl 3-methylbutanoate. Furthermore, omission experiments confirmed (*E*)-2-nonenal as the key odorant and revealed the significance of (*E*)-2-octenal for the overall aroma of Chixiang aroma-type liquor. Peipei, Zhao, and Tingting (2015) found that 13 key aroma compounds, including ethyl acetate, ethyl hexanoate, ethyl octanoate, ethyl butanoate and ethyl pentanoate, which have $OAV \geq 1$, were important aroma compounds in Daohuaxiang liquors by GC-MS, GC-O and GC-FID. However, the characterization of sulfur-containing compounds was ignored by these researchers. The possible causes were that the concentration of sulfur-containing compounds was extremely low or the aroma of sulfur-containing compounds was not pleasant.

Sulfur-containing compounds may have a significant impact on the overall aroma of liquors due to their extremely low odor thresholds. Yuan-yuan, Jin-yuan, and Guo-feng (2012) detected

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four sulfur-containing compounds, namely 3-methylthio-1-propanol, ethyl 3-methylthiopropionate, methyl trisulfide and ethyl (methylthio)acetate, by using direct immersion solid-phase microextraction (DI-SPME) and LLE coupled with GC-MS in Bandongjing Zhima aroma-type liquors. The same authors (Yuan-yuan, Jin-yuan, & Guo-feng, 2014) indicated that in addition to the previously reported four sulfur compounds, another seven were found, namely dimethyl disulfide, dimethyl tetrasulfide, 3-(methylthio)propanal, furfuryl mercaptan, difurfuryl disulfide, isopropyl disulfide and *S*-methyl thiomethanesulfinate. In 2016, Zhao Dongrui et al. (Dong-rui, Li-mo, & Feng-guo, 2016) identified 14 sulfur compounds in 36 Zhima aroma type liquors by SPME and LLE coupled with GC-MS. Benzothiazole and benzothiophene were detected for the first time. However, the characterization of sulfur-containing compounds in light aroma-type liquors has not been reported so far. Therefore, in order to further investigate the effect of sulfur-containing compounds on the overall aroma of light aroma-type liquors, the identification and quantitative analysis of sulfur-containing compounds was performed in this study.

Recently, multivariate statistical methods have been used to explore the relationship between sensory profiles and wine volatiles, such as principal component analysis (Vilanova, Genisheva, Masa, & Oliveira, 2010) and partial least-squares regression (Niu et al., 2011). These techniques can be used to examine the relationship between light aroma type liquor samples, sensory attributes and aroma-active compounds.

The primary aims of the present study were (a) to identify the key aroma compounds; (b) to apply descriptive sensory analysis to describe the aroma attributes of different light aroma type liquors; and (c) to elucidate the correlation between aroma compounds, sensory attributes and liquor samples through partial least-squares regression (PLSR) analysis. A better understanding of this knowledge will be helpful for the improvement of characteristic aroma of light aroma type liquors. Further, this study is of significant importance for improving brewing technology and providing a new research method of light aroma type liquors. More importantly, different light aroma type liquors can be more easily differentiated by the difference on numbers and concentration of sulfur compounds.

2. Material and methods

2.1. Samples

Five Chinese light aroma type liquors were under investigation: Guoseqingxiang liquor (GSQXJ, 53% ethanol by volume, from Henan province in central China), Hongxing liquor (HXJ, 53% ethanol by volume, from Beijing in northeastern China), Xinhuaacun liquor and Fenjiu liquor (XHCJ, FJ, 53% ethanol by volume, from Shanxi province in north-central China), Jinmen liquor, (JMJ, 58% ethanol by volume, from Taiwan in southeastern China). All liquors (500 mL of each) were supplied by corresponding distilleries and stored at 4 °C until analysis.

2.2. Chemicals

Authentic standards were obtained from commercial sources: 1-propanol, 1-butanol, 1-hexanol, 2-methyl-1-propanol, 3-methyl-1-butanol, 1-octen-3-ol, diisobutylcarbinol, ethyl acetate, ethyl propanoate, ethyl 2-methylpropanoate, ethyl butanoate, 3-methylbutyl acetate, ethyl pentanoate, ethyl hexanoate, ethyl lactate, ethyl heptanoate, ethyl octanoate, ethyl nonanoate, ethyl decanoate, diethylbutanedioate, ethyl dodecanoate, ethyl hexadecanoate, ethyl linoleate, acetic acid, isobutyric acid, 2-methylbutanoic acid, 3-methylbutanoic acid, butanoic acid, pen-

tanoic acid, hexanoic acid, octanoic acid, nonanoic acid, decanoic acid, phenylacetaldehyde, ethyl 2-phenylacetate, 2-phenylethyl acetate, ethyl 3-phenylpropanoate, benzenemethanol, 2-phenylethanol, phenol, 4-ethylphenol, guaiacol, 4-ethylguaiacol, furfuryl alcohol, furfural, 2-furaldehyde diethyl acetal, trimethylpyrazine, tetramethylpyrazine, β -damascenone, and 3-hydroxy-2-butanone were purchased from Sigma-Aldrich (Shanghai, China). 2-Octanol and dipropyl disulfide (Sigma-Aldrich, Shanghai, China) were used as internal standards (IS). A C₇–C₃₀ *n*-alkane mixture (Sigma-Aldrich,) was employed for determination of linear retention indices (RIs). All of the substances used were of AR quality, with at least 97% purity. Pure water was obtained from a Milli-Q purification system (Millipore, Waltham, MA). Sodium chloride (analytical grade), anhydrous sodium sulfate (analytical grade), and dichloromethane (analytical grade) were purchased from Sino-pharm Chemical Reagent Co., Ltd. (Shanghai, China).

2.3. Liquid-liquid extraction

Volatile compounds were extracted by the following method. Five liquor samples (50 mL of each) were diluted to 10% ethanol with 200 mL boiled, deionized water. The diluted liquor samples were saturated with sodium chloride, and then the saturated solution was poured into a separatory funnel. 2-Octanol (400 mg/L, 1 mL) was added to the solution. Next, the solution with 2-octanol was extracted three times with dichloromethane (20 mL, 20 mL, 10 mL, respectively) in the separatory funnel. Afterward, all extracts were dried by adding 40 g anhydrous Na₂SO₄ overnight. Finally, all extracts were slowly concentrated to 200 μ L under a gentle stream of nitrogen. These final concentrations were stored at –20 °C prior to analysis.

2.4. Identification by GC-O, GC-FPD and GC-MS

GC-MS analysis was performed on a 7890 GC with a 5975 mass selective detector (MSD) (Agilent Technologies, Santa Clara, CA). GC-O analysis was performed using an Agilent 7890A gas chromatograph equipped with an olfactometer (ODP 2; Gerstel, Mülheim an der Ruhr, Germany). GC-FPD analysis was performed on an Agilent 7890A gas chromatograph equipped with flame photometric detection (FPD).

2.4.1. GC-MS analysis

The concentrated fraction (1 μ L) was injected in splitless mode. Samples were analyzed on both an HP-Innowax column (60 m \times 0.25 mm i.d. \times 0.25 μ m; Agilent) and a DB-5 column (60 m \times 0.25 mm i.d. \times 0.25 μ m; Agilent). Helium (purity = 99.999%) was used as a carrier with a constant flow of 1 mL/min. The quadrupole mass filter was at 150 °C. The transfer line temperature was held at 250 °C (Jiancai Zhu et al., 2015). The oven temperature was held at 40 °C for 6 min, then ramped to 100 °C at the rate of 3 °C/min, and ramped at the rate of 5 °C/min to 230 °C for the last 20 min. The MSD operated in electron ionization (EI) mode (70 eV, ion source temperature 230 °C) with the quadrupole in scan mode (scan range was *m/z* 30–450 at a scan rate of 1 scan/s).

2.4.2. GC-O analysis

GC effluent was split 1:1 between the flame ionization detector (FID) and sniffing port. Samples were separated on both a HP-Innowax analytical fused silica capillary column (60 m \times 0.25 mm \times 0.25 μ m; Agilent) and a DB-5 analytical fused silica capillary column (60 m \times 0.25 mm \times 0.25 μ m, Agilent). The flow rate of carrier gas (hydrogen) was 2 mL/min. The oven temperature was first increased from 40 °C (6 min), at 3 °C/min to 100 °C, and then ramped at 5 °C min⁻¹ to 230 °C (20 min); the

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