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# Variation of aroma profile in fermentation process of Wuliangye *baobaoqu* starter

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ABSTRACT

Changes of volatile compounds and sensory properties in the fermentation process of Wuliangye *baobaoqu* starter (WBS) were determined by sensory analysis, gas chromatography-olfactometry (GC/O) and GC quantitative analyses. The sensory properties of WBS changed from the sour, fruity, bready notes at the temperature-rise phase (3-day and 7-day) to the nutty and moldy notes at the temperature-fall phase (18-day, 21-day and 27-day). Acetic acid, methional, ethyl acetate, phenylethyl alcohol, ethyl 2-phenylacetate, 4-vinylguaiacol were potential aroma active compounds based on their Osme values. Results of GC quantitative analyses showed that the acids had the highest concentration. The concentration of (*E*)-2-hexenal, ethyl acetate, isoamyl alcohol, phenylethyl alcohol, 2-*n*-pentylfuran, acetic acid and lactic acid increased with the fermentation time, presented the highest level in the temperature-rise phase and decreased after this period. The concentration of (*E,E*)-2,4-hexadienal, 2,6-dimethylpyrazine, trimethylpyrazine, tetramethylpyrazine presented the highest level at the temperature-fall phase. Ethyl 3-methylbutanoate, (*E,E*)-2,4-hexadienal, (*E,E*)-2,4-nonadienal, acetic acid and butanoic acid had high odor active values. Results of principal component analysis showed that acetic acid, hexanal, (*E*)-2-hexenal, lactic acid, isoamyl alcohol, 1-hexanol and 1-octen-3-ol were critical volatile chemicals to distinguish different WBS samples.

## 1. Introduction

Wuliangye is the most popular strong-aroma liquor (*baijiu*) brand in China because of its huge consumption amount ($> 1.4 \times 10^5$ kL/year) as well as its specific aroma and taste features. According to the history of Chinese *baijiu*-making industry, Wuliangye is recongnized as one of most important *baijiu* because a lot of fermentation technologies for Wuliangye are widely applied in Chinese *baijiu*-making industry (Shen, 2007; Xu, Wang, Fan, Mu, & Chen, 2010). Therefore, understanding the fermentation technology of Wuliangye can provide useful information for making other Chinese *baijiu*.

*Daqu*, a mixture of crude enzymes and microbes, is one the most important starter for Chinese *baijiu*. Usually it can be classified into three types including the medium-temperature type (40–50 °C), the medium to high temperature type (MHT, 50–60 °C) and the high-temperature (60–70 °C) type according to their highest incubation temperatures (Zheng, Tabrizi, Nout, & Han, 2011). Wuliangye *baobaoqu* starter (WBS), the necessary starter for Wuliangye, is the representative starter with various unique features. Compared with other MHT starters, the WBS is prepared with pure wheat (100%) and in the fermentation process it experiences a specific temperature changing process. Before incubation, the raw wheat is ground and its moisture content is adjusted to about 38% accordingly. The raw wheat mash is pressed into the brick-shape starter with a big bump in one side (Fig. 1). Then the rough starter is placed into a fermentation room for about 30 days' incubation. During this period, the temperature of rough starter increases from the ambient temperature to the highest temperature (58–60 °C) and the 1st time of manual position exchange (between the edge and center) is implemented at the 7th day (called the temperature-rise phase). After this phase, the incubation temperature of rough starter will be kept at the highest temperature for another one week under the control of operator. Then the 2nd time of manual position exchange is processed at the 14th day (called the high-temperature phase). Vast majority of product-moisture evaporates after this period, and the temperature of starter will slowly decrease (Peng, Zhang, Qiao, Liu, & Chen, 2015). After the 3rd and 4th time of manual position exchanges, the starters are then gathered together to let the moisture evaporated further until the end of incubation process (called the temperature-fall phase). During the period, a lot of aroma compounds are formed along with the changes of temperature and

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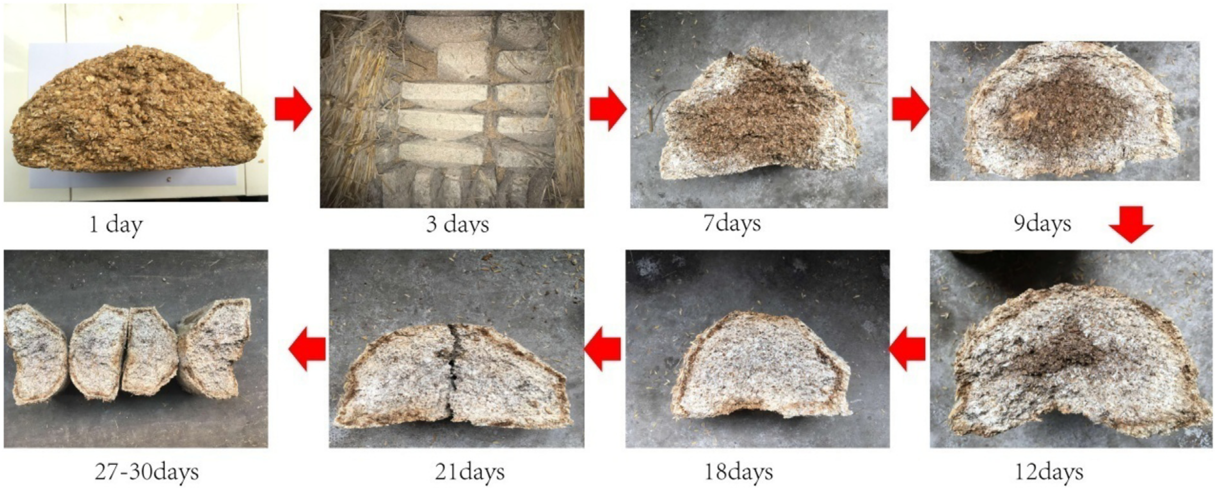


**Fig. 1.** Variation of *Wuliangye baobaoqu* starter (WSB) during fermentation.

moisture.

Recently, a lot of studies have been conducted to understand volatile compounds in strong-aroma *baijiu* and *daqu*. The key aroma compounds in Yanghe, Jiananchun, Wuliangye and Gujinggong *baijiu* were analyzed by gas chromatography/olfactometry (GC/O) (Fan & Qian, 2006a,c; Zhao et al., 2018). Zhang et al. (2012) analyzed the aroma-active compounds in *daqu* by AEDA and hexanal (green grass), unknown (no.u2, nutty, roasted), phenylacetaldehyde (floral, rose), and 4-ethylguaiacol (clove, spicy) were identified to be the aroma active compounds. It has been identified that the volatile compounds of steamed wheat for China *baijiu*-making included aldehydes, alcohols and esters, such as hexanal, hexanol, phenylethyl alcohol and ethyl hexanoate (Ye, Lian, Xie, Li, & Liao, 2014). Due to the thermal and microbial effects, various kinds of volatile compounds were generated during the fermentation of *daqu*. To date, however, no report has been conducted to investigate the variation of aroma profile in the fermentation process of WBS.

Therefore, in order to comprehensively understanding the aroma profile of WBS, the aim of this study was to discuss variations of volatile compounds and sensory properties in the fermentation process of WBS. The results of this study might provide useful information for flavor research of Chinese *baijiu*.

## 2. Materials and methods

### 2.1. Chemicals

Ethyl acetate, ethyl 3-methylbutanoate, butyl 2-methylbutanoate, ethyl hexanoate, ethyl octanoate, ethyl heptanoate, hexyl hexanoate, ethyl benzoate, methyl phenylacetate, ethyl 2-phenylacetate, ethyl 3-phenylpropanoate, phenylethyl acetate, hexanal, heptanal, (*E*)-2-hexenal, nonanal, (*E,E*)-2,4-hexadienal, (*E,E*)-2,4-heptadienal, octanal, decanal, (*E*)-2-nonenal, (*E*)-2-octadienal, (*Z*)-2-pentenal, (*E*)-decenal, (*E,E*)-2,4-nonadienal, (*E,Z*)-2,4-nonadienal, 1-octen-3-ol, furfural, isoamyl alcohol, 1-hexanol, octanol, (*E*)-2-octen-1-ol, 4-octanone, 3-octen-2-one, 2-*n*-pentylfuran, 3-octanone, methylpyrazine, 2,5-dimethylpyrazine, 2,6-dimethylpyrazine, 2,3-dimethylpyrazine, trimethylpyrazine, tetramethylpyrazine, benzaldehyde, benzeneacetaldehyde, phenylethyl alcohol, naphthalene, guaiacol, 4-methylguaiacol, *p*-cresol, 4-ethylguaiacol, β-caryophyllene, γ-nonalactone and 4-octanol were purchased from TCI (Shanghai, China). Acetic acid, propanoic acid, butanoic acid, lactic acid, acetone (AR grade), dichloromethane ($CH_2Cl_2$, ACS grade) and absolute ethanol (ACS grade) were purchase from Aladdin (Shanghai, China). A mixture of $C_7$-$C_{30}$*n*-alkanes was purchased from Sigma-Aldrich (Shanghai, China).

### 2.2. Sampling and pretreatment

WBS samples with different fermentation days (Fig. 1) including 1-day (initial day), 3-day, 7-day, 9-day, 12-day, 18-day, 21-day, and 27-day were collected from the *baobaoqu*-making department of Wuliangye Yibin Co. Ltd. (Yibin, Sichuan province, China). As shown in Fig. 2, the whole fermentation process of WBS can be divided into three main periods including the temperature-rise phase (1-day to 7-day, from ambient temperature to 60 °C), the high-temperature phase (8-day to 14-day, hold at 60 °C), and the temperature-fall phase (15-day to 27-day, from 60 °C to ambient temperature). Three parallel samples were randomly selected in each fermentation time point and chilled to −20 °C during transportation. The whole time from sample collection to laboratory was controlled to < 30 min. Each sample was ground under frozen condition by blender (Taisite, Tianjin, China), and the WBS powder samples were separately sealed in storage bag and stored in −20 °C until further analysis.

### 2.3. GC/O analysis

Fifty grams of WBS powder were weighed into the Erlenmeyer flask, and 100 mL of $CH_2Cl_2$ was then added into the flask. The flask was shaken with the rate of 150 r/min. After 1 h extraction, the mixture in

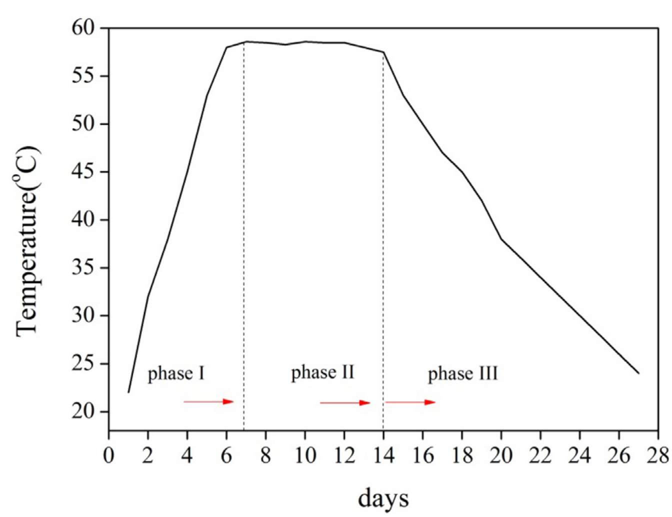


**Fig. 2.** Variation of temperature of WBS in the fermentation process. Phase I is the temperature-rise phase, Phase II is the high-temperature phase, and Phase III is the temperature-fall phase.