Analysis of Volatile Components in Gui Qi Mango Fruit Wine

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Abstract. The volatile components in Gui Qi Mango fruit wine analyzed by headspace solid-phase microextraction-gas chromatography-mass spectrometry. Results showed that 32 kinds of volatile components were detected in Gui Qi Mango fruit wine, including 7 kinds of alcohols (65.69%), 17 kinds of esters (28.92%), 4 kinds of alkanes (2.16%), 3 kinds of aldehydes (0.36%), 2 kinds of olefins (0.32%), 1 kinds of ketones (0.41%), 1 kinds of phenols (0.2%). In the all of the volatile components, the relative content of alcohols and esters were 94.61%. The fermentation process had an effect on the Gui Qi Mango fruit wine, and alcohols and esters played a positive role in the aroma of Gui Qi Mango fruit wine.

1. Introduction
Mango, one of the most famous tropical fruits, belongs to the genus Mangifera of the family Anacardiaceae. It has a smooth pulp, which is unique flavor and rich nutrients. It is known as the “the king of tropical fruits” [1-2]. At present, more than 100 varieties of mangoes have been introduced and cultivated in China, and they are widely planted in large areas in Hainan, Panzhihua, and Guangxi, Guangdong etc. with rapidly expanding planting areas [3]. Mangoes contain more nutrients than ordinary fruits. Further, their sugar content is over 7.0%, and the concentrations of the vitamins A, B, and C are higher than in other fruits. Furthermore, the carotene content in 100 g of pulp is 22816,304 µg. Additionally, it contains abundant essential trace elements (selenium, calcium, phosphorus, potassium, etc.) [4-6]. Owing to its high moisture content, the mango is a delicate fruit with thin skin and juicy pulp. Therefore, physical damage and decomposition may occur during the processes of picking, transportation, and storage, which not only limits the development of the mango industry but also seriously impacts the enthusiasm of mango growers. In recent years, fruit wines produced from fermented mangoes have been proposed as an effective way to solve this problem. There have been many reports regarding the fermentation processes and flavor-component analysis of mango fruit wines [7-12]; however, fewer reports regarding the qualitative and quantitative analysis of volatile substances in these wines.

In this research, the volatile substances in the Guiqi mango fruit wine were qualitatively and quantitatively analyzed through headspace solid phase microextraction (SPME) combined with the GC-MS technique in order to provide a scientific basis for the application of mango fruit wine and promote its standardized production.
2. Experimental

2.1. Materials and methods

2.1.1. Materials. Guiqi mango fruit wine (12% ABV): developed by the South Subtropical Crops Research Institute of the Chinese Academy of Tropical Agricultural Sciences.

2.1.2. Instruments and equipments. GC-MS: 6890-5973 MSD, Agilent Technologies Co. Ltd (Shanghai, China)
   SPME fiber: 50/30 µm DVD/Carboxen/PDMS, Supelco.
   HP-5 capillary GC column: 60 m × 0.25 mm × 0.25 µm, Agilent Technologies Co. Ltd (Shanghai, China)

2.2. Methods

2.2.1. Pretreatment of fruit wine samples. A 5.0-mL sample of the Guiqi mango fruit wine was pipetted into an SPME vial and sealed with a cap. An extraction fiber (50/30 µm DVD/Carboxen/PDMS) was inserted into the headspace of the vial to perform SPME for 60 min at room temperature. After the extraction was performed, the extraction fiber was injected directly into the inlet of the GC-MS. The inlet temperature was set at 250 °C for GC-MS analysis; simultaneously, and data acquisition was performed.

2.2.2. GC-MS analysis [13]
   GC conditions:
   Column: HP-5 capillary GC column (60 m × 0.25 mm × 0.25 µm). The carrier gas was helium, with a purity of 99.999%.
   Temperature program: initial temperature at 35°C, hold for 2 min; ramp to 170°C at 3.0°C /min; ramp to 225°C at 30°C /min, hold for 3 min.
   The flow rate was 1.0 mL/min; inlet temperature was 250°C; the split ratio was 10:1.
   MS conditions
   EI source is 65 eV. Source temperature was 230°C and quad temperature was 150°C. Scan range was m/z 30-600.

2.3. Qualitative and semi-quantitative analyses
   Qualitative analysis: The MS identification results were searched in the MS database library, and the compounds with matching values >90 (maximum matching value was 100) were identified as volatile components in the fruit wine.
   Semi-quantitative analysis: The relative contents of the volatile components in the mango fruit wine were calculated through peak area normalization method.

3. Results and discussions
The volatile components in the mango fruit wine were detected through gas chromatography-mass spectrometry combined with computer-assisted data analysis. The mass spectra of the individual components were analyzed through a spectral library search and literature review and analysis to determine the structures of the compounds. The peak area normalization method was used to obtain the individual compounds' relative contents (shown in Table 1 and 2).
Table 1. Analysis of volatile flavor components in Guiqi mango wine

| Types       | Serial Number | Volatile Flavor Components            | Relative content/% |
|-------------|---------------|---------------------------------------|--------------------|
| Alcohols    | 1             | Ethanol                               | 53.06              |
|             | 2             | 1-Propanol                            | 0.92               |
|             | 3             | 2-methyl-1-Propanol                   | 1.07               |
|             | 4             | 3-methyl-1-Butanol                    | 4.54               |
|             | 5             | 2-methyl-1-Butanol                    | 0.97               |
|             | 6             | 2,3-Butanediol                        | 1.35               |
|             | 7             | Phenylethyl Alcohol                   | 3.78               |
|             | 8             | Ethyl Acetate                         | 9.06               |
|             | 9             | Butanolic acid, ethyl ester           | 0.87               |
|             | 10            | 1-Butanol, 3-methyl-, acetate         | 0.80               |
|             | 11            | Hexanoic acid, ethyl ester            | 0.45               |
|             | 12            | Octanoic acid, ethyl ester            | 0.45               |
|             | 13            | Acetic acid, 2-phenoxyethyl ester     | 0.18               |
| Esters      | 14            | Nonanoic acid, ethyl ester            | 0.03               |
|             | 15            | Decanoic acid, ethyl ester            | 0.39               |
|             | 16            | Dodecanoic acid, ethyl ester          | 2.07               |
|             | 17            | Tetradecanoic acid, ethyl ester       | 5.98               |
|             | 18            | Ethyl 9-hexadecenoate                 | 2.76               |
|             | 19            | Hexadecanoic acid, ethyl ester        | 5.68               |
|             | 20            | Isoamyl isobutyrate                   | 0.03               |
|             | 21            | Phenethyl acetate                     | 0.17               |
|             | 22            | Acetaldehyde                          | 0.29               |
| Aldehydes   | 23            | Benzaldehyde                          | 0.04               |
|             | 24            | Nonanal                               | 0.03               |
| Ketones     | 25            | 3-hydroxy-2-Butanone                  | 0.41               |
| Phenols     | 26            | 2,6-Di-tert-butyl -p-cresol           | 0.20               |
| Olefins     | 27            | 1-methyl-4-(1-methylthylidene)-Cyclohexene | 0.29         |
|             | 28            | D-Limonene                            | 0.03               |
|             | 29            | 2,4-dimethyl-1,3-Dioxane              | 0.98               |
| Alkyl hydrocarbons | 30      | 1,1-dietioxy-Ethane                  | 0.84               |
|             | 31            | 2,4,5-trimethyl-1,3-Dioxolane         | 0.28               |
|             | 32            | 2-heptyl-1,3-Dioxepane                | 0.06               |

Table 2. Analysis of volatile flavor components in Guiqi mango wine

| Serial Number | Types of Volatile Flavor Component | Relative content/% | Content of total volatile flavor component/% |
|---------------|------------------------------------|--------------------|---------------------------------------------|
| 1             | Alcohols                           | 65.69              | 66.99                                        |
| 2             | Esters                             | 28.92              | 29.49                                        |
| 3             | Aldehydes                          | 0.36               | 0.37                                         |
| 4             | Ketones                            | 0.41               | 0.42                                         |
| 5             | Phenols                            | 0.20               | 0.204                                        |
| 6             | Olefins                            | 0.32               | 0.33                                         |
| 7             | Alkyl hydrocarbons                 | 2.16               | 2.20                                         |

During the process of headspace solid phase microextraction, the volatile compounds from the samples were extracted and enriched using the polymer layer on the surface of the quartz fiber. Then,
they underwent thermal desorption at the GC inlet; subsequently, were analyzed through mass spectrometry [14-15]. This technique can effectively analyze the volatile flavor components in the mango fruit wine. The volatile flavor substances of mango fruit wine are shown in Tables 1 and 2.

Tables 1 and 2 show that 32 volatile components were detected in the Guiqi Mango fruit wine, including 7 alcohols (65.69%), 17 esters (28.92%), 4 alkanes (2.16%), 3 aldehydes (0.36%), 2 olefins (0.32%), 1 ketone (0.41%), and 1 phenol (0.2%). The top 10 components with the highest relative contents are ethanol (53.06%), 2-methyl-1-propanol (1.07%), 3-methyl-1-butanol (4.54%), 2,3-Butanediol (1.35%), phenylethyl alcohol (3.78%), ethyl acetate (9.06%), ethyl laurate (2.07%), ethyl myristate (5.98%), ethyl 9-hexadecenoate (2.76%) and ethyl palmitate (5.68%), all of which account for 89.35% of the total volatile compounds in the wine.

Compared with the mango pulp, in which terpenes are the main volatile components [16-18], the components with relatively high contents in the Guiqi mango fruit wine are alcohols and esters, which account for 94.61% of the total volatile components. The fermentation process affected the formation of the wine components, and alcohols and esters boosted the aroma of the mango fruit wine. Higher alcohols are an important type of volatile aroma substance in alcoholic beverages [14]. The tested mango fruit wine contains higher alcohols such as phenylethyl alcohol and 3-methyl-1-butanol. The content of phenylethyl alcohol is up to 3.78%; it releases the scent of roses. Further, the wine has esters such as ethyl acetate, ethyl laurate, ethyl myristate, ethyl 9-hexadecenoate, and ethyl palmitate, all of which endow a sweet fruity aroma to the fruit wine. Moreover, a small amount of olefinic compounds, such as limonene, were detected in the Guiqi mango fruit wine, which was consistent with the detection results of volatile components in different varieties of mangoes by Pino [19-20] and Liu [21].

4. Conclusion

In this study, the volatile components in Guiqi mango fruit wine were analyzed through headspace solid phase microextraction combined with GC-MS. There were 32 volatile compounds were detected in the fruit wine, including 7 alcohols (65.69%), 17 esters (28.92%), 4 alkanes (2.16%), 3 aldehydes (0.36%), 2 olefins (0.32%), 1 ketone (0.41%), and 1 phenol (0.2%).

Compared with the mango pulp, the main volatile components of which are terpenes [16-18], the Guiqi mango fruit wine has relatively high contents of alcohols and esters, which account for 94.61% of the total volatile components. The fermentation process played a role in the formation of the wine components, and alcohols and esters boosted the flavor of the mango fruit wine. These research results will provide a scientific basis for the production and application of the mango fruit wine.

GC-MS is a semi-quantitative, analytical method for the determination of the structure and content of volatile components in samples; however, it does not identify the odor and aroma characteristics of the aroma essence [22]. In the future, the gas chromatography-olfactometry (GC-O) analysis will be applied to further identify the characteristic flavor and its components in the mango fruit wine.

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