

Analysis of aroma compounds of pitaya fruit wine

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Abstract: In order to analyze the volatile components in red pitaya fruit wine, the study using headspace solid phase microextraction and gas chromatography-mass spectrometry technology of pitaya fruit juice and wine aroma composition analysis comparison. Results showed that 55 volatile components were detected in red pitaya fruit wine, including 12 kinds of alcohol (18.16%), 18 kinds of esters (66.17%), 7 kinds of acids (5.94%), 11 kinds of alkanes (4.32%), one kind of aldehyde (0.09%), 2 kinds of olefins (0.09%) and 3 kinds of other volatile substances (0.23%). Relative contents among them bigger have 11 species, such as decanoic acid, ethyl ester (22.92%), respectively, diisoamylene (20.75%), octanoic acid, ethyl ester (17.73%), etc. The red pitaya fruit wine contained a lot of aroma components, which offer the products special aroma like brandy, rose and fruit.

1. Introduction

Pitaya (*Hylocereus undulatus* Britt), also known as red dragon fruit, nectar fruit and lover fruit, is native to tropical central America, such as Brazil and Mexico ^[1], and belong to *Hylocereus undatus* and the *Seleniurus* plant in the *Cactaceae*. At present in southern China, such as Hainan, Guangdong and Guangxi, some areas also have a certain scale of cultivation. According to the characteristics of peel and pulp color, they can be divided into three varieties including *Hylocereus undatus*, *Hylocereus megalanthus*, *Hylocereus costaricensis* ^[2]. Pitaya has very high nutrition value, which is rich in albumin and cyanine glycosides with the function of antioxidant, and red pulp is rich in red pigment ^[3, 4].

Study on pitaya is increasing in China and foreign country, however, mostly focus on the processing technology, such as the fermentation conditions optimization with the dragon fruit wine, pitaya pigment extraction, and nutrition composition analysis ^[5, 6]. And the study of dragon fruit aroma components is relatively fewer, especially fruit wine aroma composition analysis. This study using



headspace solid phase micro-extraction and gas chromatography-mass spectrometry technology investigate aroma composition analysis comparison, the research provided the scientific basis for the aroma characters of pitaya wine ^[7-10].

2. Materials and Methods

2.1 Pre-sample treatment

Take 8 to 15 mL of wine sample (red pitaya wine, 12% vol, dry type) from sample bottles for solid phase micro-extraction, and then sealed, balanced 10 min under 60°C. After extraction adsorption 40 min, automatic sampling and GC-MS analysis. Injection port temperature 250°C, and desorption for 3 min.

2.2 GC-MS conditions

GC condition: capillary column VF-Wax (30 m × 0.25 mm, 0.25 μm); Temperature programmed: initial temperature 40°C, keep 5 min, to rise to 120°C, 4°C/min rate to the rate of 5°C/min to 210°C, keep 5 min. Injection port temperature 250°C; High purity helium (He), velocity 1 mL/min; No diversion. MS condition: electronic (electron ionization, EI) source, electronic capacity is 70 Ve ion source temperature 230°C, 4 pole temperature 150°C, full scan mode, the scanning range of 45~600 m/z, solvent delay 5.5 min. The relative content of volatile components in the samples was calculated by using peak area normalization method using NIST 9.0 and 14.0.

3. Results and Discussion

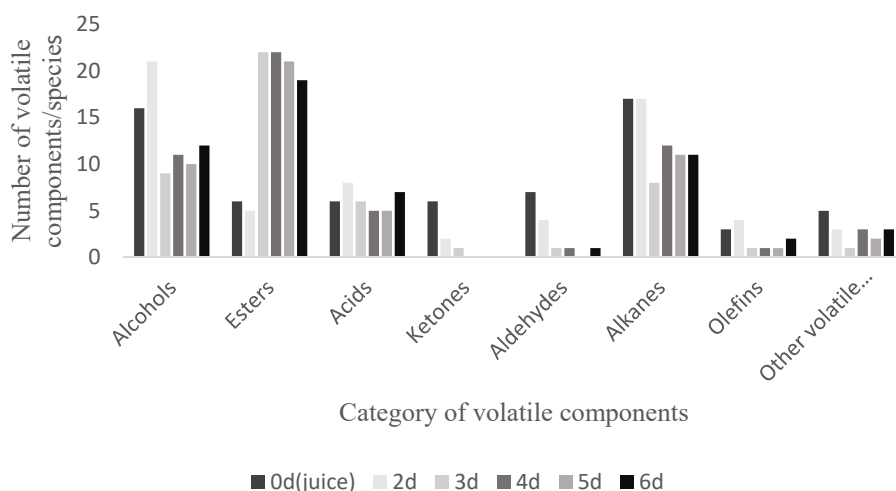


Fig.1. Volatile component changes during fermentation of Pitaya wine

Aroma is one of the important factors that constitute the quality of fruit wine. The aroma of fruit wine central plains constitute a class of wine aroma components. From the fig. 1, the volatile constituents in fruit juice is mainly composed with alkanes, alcohols, acids, etc and esters, alcohols and acids are the main volatile substances in the wine. When the juice is fermented, there produced some complex biochemical reactions. With the increase of the number of fermentation time, the aroma is gradually formed due to the growth and metabolism of yeast. Alcohols have declined, but they are still mainly aromatic substances. Number of esters have obviously rise and acids also has edged up, ketones, aldehydes, alkanes, alkenes and other apparent decline in the number of volatile components. This shows that aroma composition of pitaya wine mainly affected by alcohols and esters.

Table 1 GC-MS analysis of volatile compounds in red pitaya wine

| | No. | Compounds | Retention time /min | Peak area | Relative content/% |
|----------|-----|---|------------------------|-----------|-----------------------|
| Alcohols | 1 | 1-Butanol, 3-methyl- (impure) (CAS) | 3.53 | 16724651 | 3.37 |
| | 2 | 1,2-Propanediol (CAS) | 4.36 | 330675 | 0.07 |
| | 3 | 2,3-Butanediol (CAS) | 5.02 | 27116133 | 5.47 |
| | 4 | Silane, ethoxytriethyl- | 5.08 | 2061545 | 0.42 |
| | 5 | 2,3-Butanediol | 5.18 | 7585965 | 1.53 |
| | 6 | 1-Pentanol, 3-methyl- (CAS) | 5.85 | 83604 | 0.02 |
| | 7 | 1,8-Cineole | 11.42 | 291568 | 0.06 |
| | 8 | 1-Octanol (CAS) | 12.66 | 242951 | 0.05 |
| | 9 | Phenylethyl alcohol | 17.54 | 1217825 | 6.85 |
| | 10 | .beta.-Citronellol | 28.47 | 151175 | 0.25 |
| | 11 | Diisoamylene (CAS) | 29.50 | 449402 | 0.03 |
| | 12 | 1-Tridecanol (CAS) | 3.53 | 16724651 | 0.09 |
| Esters | 13 | Acetic acid, 2-methylpropyl ester (CAS) | 4.20 | 72839 | 0.01 |
| | 14 | Butanoic acid, ethyl ester | 4.76 | 320847 | 0.06 |
| | 15 | 1-Butanol, 3-methyl-, acetate (CAS) | 6.69 | 1798823 | 0.36 |
| | 16 | 1-Butanol, 2-methyl-, acetate | 6.76 | 227600 | 0.05 |
| | 17 | 2(3H)-Furanone, dihydro- (CAS) | 8.05 | 175261 | 0.04 |
| | 18 | Hexanoic acid, ethyl ester (CAS) | 10.39 | 7107855 | 1.43 |
| | 19 | Isoamyl lactate | 12.59 | 382005 | 0.08 |
| | 20 | Octanoic acid, ethyl ester | 16.63 | 87901057 | 17.73 |
| | 21 | Iso ameyl hexanoate | 18.16 | 146187 | 0.03 |
| | 22 | Acetic Acid, 2-Phenylethyl Ester | 18.40 | 2227477 | 0.45 |
| | 23 | Nonanoic acid, ethyl ester (CAS) | 19.52 | 749139 | 0.15 |
| | 24 | Decanoic acid, ethyl ester (CAS) | 22.36 | 113751751 | 22.92 |
| | 25 | Octanoic acid, 3-methylbutyl ester (CAS) | 23.66 | 2394741 | 0.48 |
| | | Octanoic acid, 3-methylbutyl ester (CAS) | 23.75 | 629580 | 0.13 |
| | 26 | Phosphoric acid, tributyl ester (CAS) | 25.57 | 1380870 | 0.28 |
| | 27 | Isobutyl caprate | 26.22 | 475174 | 0.1 |
| | 28 | Heptadecanoic acid, ethyl ester (CAS) | 26.83 | 208118 | 0.04 |
| Acids | 29 | E-11-Hexadecenoic acid, ethyl ester | 27.31 | 424364 | 0.09 |
| | 30 | Dodecanoic acid, ethyl ester (CAS) | 27.47 | 102908851 | 20.75 |
| | 31 | 3-Metjylbutyl decanoate | 28.64 | 4926817 | 0.99 |
| | 32 | Propanoic acid, 2-methyl- | 3.89 | 2417050 | 0.49 |
| | 33 | Hexanoic Acid | 9.82 | 1293073 | 0.26 |
| | 34 | ethyl 4-hydroxybutanoate | 12.21 | 1188676 | 0.24 |
| | 35 | Octanoic acid (CAS) | 15.78 | 14549981 | 2.93 |
| | 36 | Nonanoic acid (CAS) | 18.67 | 335894 | 0.07 |
| | 37 | Decanoic acid (CAS) | 21.46 | 9179631 | 1.85 |
| | 38 | Dodecanamide, N,N-bis(2-hydroxyethyl)- | 26.57 | 511735 | 0.1 |
| | 39 | Silenediol, dimethyl- | 3.72 | 14592804 | 2.94 |
| | 40 | Silane, diethoxydimethyl- | 4.04 | 421535 | 0.09 |
| Alkanes | 41 | Cyclotrisiloxane, hexamethyl- (CAS) | 5.23 | 1798167 | 0.36 |
| | 42 | Benzene, 1-(Dimethoxymethyl)-4-(1-methoxy-1-methylethyl)- Disiloxane, | 9.09 | 551369 | 0.11 |
| | 43 | 1,3-diethoxy-1,1,3,3-tetramethyl- (CAS) | 9.20 | 206144 | 0.04 |
| | 44 | Silicic acid, diethyl bis(trimethylsilyl) ester | 13.40 | 196035 | 0.04 |
| | 45 | Cyclopentasiloxane, decamethyl- (CAS) | 15.35 | 1312446 | 0.26 |
| | 46 | Dodecane (CAS) | 19.62 | 906075 | 0.18 |
| | 47 | Cyclohexa siloxane, dodecamethyl- | 20.44 | 841138 | 0.17 |
| | 48 | Tetradecane (CAS) | 29.81 | 295980 | 0.06 |
| | 49 | Hexadecane, 2,6,10,14-Tetramethyl- | 29.92 | 340155 | 0.07 |
| | 50 | Oxime-, methoxy-phenyl- | 8.21 | 463129 | 0.09 |
| Olefins | 51 | l-Limonene | 11.24 | 253882 | 0.05 |
| | 52 | α -Terpinolene | 13.55 | 177072 | 0.04 |
| Others | 53 | Isocineole | 10.91 | 685610 | 0.14 |
| | 54 | 3,7,7-Trimethyl-1,3,5-cycloheptatrie | 11.19 | 127694 | 0.03 |
| | 55 | Benzene, 1,2,3-trimethoxy-5-(2-propenyl)- (CAS) | 26.52 | 287919 | 0.06 |

The pitaya wine can be detected with retention time and peak area of the volatile components, there are 55 kinds of compounds including 12 kinds of alcohol (18.16%), 18 kinds of esters (66.17%), 7 kinds of acids (5.94%), 11 kinds of alkanes (4.32%), one kind of aldehyde (0.09%), 2 kinds of

olefins (0.09%) and 3 kinds of other compounds (0.23%). Relative contents among them bigger have 11 species, respectively, decanoic acid, ethyl ester (22.92%), respectively, diisoamylene (20.75%), octanoic acid, ethyl ester (17.73%), phenylethyl alcohol (6.85%), 2,3-butanediol (5.47%), isoamyl alcohol (3.37%), trimethylfluorosilane (2.94%), octanoic acid (2.93%), decanoic acid (1.85%), (R,R)-2,3-butanediol (1.53%), ethyl caproate (1.43%), accounted for 87.77%. The relative content of alcohols and esters was relatively high, indicating the influence of the aroma of fruit wine, and alkane, aldehyde, ketone, alkene, and other three components of the relative content is less, indicating that it has less effect on the aroma of fruit wine.

4. Conclusion

It is a quick and simple method to analyze the volatile components in fruit wine with headspace solid phase micro extraction and gas chromatography-mass spectrometry technology. In this study, using headspace solid phase micro extraction and gas chromatography-mass spectrometry technology to analyze the qualitative and semi-quantitative, and discussed and analyzed the result. There were 55 volatile substances detected in the fruit wine. Among them, 18 kinds of ester substances, 12 kinds of alcohols, 7 kinds of acids, 11 kinds of alkanes, one aldehyde, 2 kinds of olefins, 3 other kinds of substances. More relative content of esters, acids and alcohols compound, accounting for over 90% of the total volatile compound detection. It shows that these volatile components play an important role in the quality of the fruit wine. The aroma of fruit wine is mostly made up of esters and alcohols, and the variety and content of them determines the aroma of wine. Like diisoamylene, phenylethyl alcohol, benzene ethanol and ethyl decanoate, the these volatile ingredients can give the fruit wine similar to the aroma of brandy, rose and fruit.

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