Comparison of Aromatic Compounds in Pitaya Wine on Different Columns

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Abstract: The aroma components of pitaya wine was enriched by headspace solid-phase microextraction (HS-SPME). The chromatographic separation was performed on two kinds of polar columns, respectively. GC-MS was used to detect and compare the aroma components. Differences between the separation of aroma components of pitaya wine by polar column HP-5MS and polar column CP-Wax. The results showed that there were significant differences between the two columns in the determination of the aroma components of pitaya wine. The main aroma compounds measured by HP-5MS column had many more types and contents than the CP-Wax column.
Keywords: Pitaya wine; Polar column; Volatile Components

1. Introduction

Pitaya (*Hylocereus* spp.) known as the fairy fruit and auspicious fruit, is a cactus *Scopoma polygamous* fruit cultivars, has the function of fruits, flowers, vegetables[1-2]. At present, countries in the tropical regions of China, Vietnam, Philippines and Israel have achieved commercial planting. Studies have shown that pitaya is a high-fiber, low-fat, low-sugar fruit, in addition to carbohydrates, crude fiber, but also contains unsaturated fatty acids, antioxidants, Vc and anthocyanins even contain rare plants plant protein [3-4]. At present, the processing of dragon fruit has attracted more and more attention. Low-degree fruit wine made from pitaya as the main raw material, with rich fruit aroma, rich nutrition, sweet and sour taste, suitable for women and children drinking requirements, for a broader consumer group. Aroma as an important component of the sensory quality of fruit and wine is an important indicator of wine quality. The determination of aroma is affected by many factors, different varieties of fruit have different aromas[5]. Different fermentation processes or different strain fermentation aromas of the same raw material will produce differences[6-9], different wines produced in the same process have different aromas[10]. But so far there has been less research report on the aroma components of Pitaya fruit wine. Therefore, it is necessary to investigated the aroma components in fruit wine.

At present, in the research of the volatile components of the fruit wine, the pretreatment method of using headspace solid-phase microextraction is mainly carried out and analyzed by gas chromatography-mass spectrometry[11]. After entering the gas chromatogram, the mixture is efficiently separated by a capillary column. Therefore, the key to chromatographic separation is to select an appropriate column, and the appropriate column is selected according to the nature of the target compound[12]. In order to understand the difference in separation effect of different polar column materials on the aroma components of pitaya fruit wine, this paper selected red pitaya as the raw material to ferment fruit wine, using CP-Wax and HP-5MS columns to analyze and compare the aroma components in the fruit wine, In order to provide a theoretical basis for the analysis of aroma components of pitaya wine.

2. Materials and Methods

2.1 Pre-sample treatment

8-15 mL of wine sample (pitaya wine, 12 % vol, dry type) was taken from 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 Gas chromatography-mass spectrometry conditions

CP-Wax column: quartz capillary column (50 m × 0.25 mm, 0.20 μm); inlet temperature was 250°C; carrier gas was high purity helium (purity> 99.999% Column temperature: The initial temperature was raised to 40°C at a rate of 2°C per minute for 5 minutes, then increased to 160°C at a rate of 3°C per minute and finally to 240 °C at a rate of 7°C per minute , Hold for 10 min; splitless injection.

HP-5MS column: quartz capillary column (60 m × 0.32 mm, 0.25 μm); inlet temperature was 250°C; carrier gas was high purity helium (purity> 99.999% Column temperature: the initial temperature of 40°C, for 3 min, 2°C per minute rate rose to 85°C, holding 5 min, and then 3°C per minute rate rose to 160°C, and finally at a rate of 10°C per minute Raise to 240°C for 5 min; splitless injection. Electron ionization (EI) source, ion source temperature 230°C, electron energy 70 eV, quadrupole temperature 150°C, transmission line temperature 280°C, scanning range m/z 30 ~ 500 amu.

2.3 Qualitative and semi-quantitative analysis

Qualitative analysis: The results of the mass spectrometry were analyzed by the Wiley.lib database and national institute of standards and technology NIST14. Library search, Only match values (similarity index, SI) >90 (The maximum match value is 100) identification results to be reported. Semi-quantitative analysis: The relative content of volatile components in the fruit of the pitaya was calculated by the peak area normalization method.

3. Results and discussion

3.1 Analysis of Aroma Components by GC-MS

Figure 1 and Figure 2 is Pitaya wine aroma components GC-MS total ion chromatogram, search by comparison, identify the major volatile components and some of the volatile components of the aroma described in Table 1.

Table 1 shows the results of analysis of flavor compounds obtained from Pitaya wine on HP-5MS columns and CP-Wax columns, respectively. Two different polar columns, because of their different stationary phases, retain the different effects of each compound, resulting in the separation of different results. Compared with pitaya pulp[13-14], which is a volatile compound mainly composed of aldehydes, hydrocarbons and terpenoids, the relative contents of pitaya fruit wines are esters, organic acids and alcohols. As can be seen from Table 1, 42 major volatile substances were detected on the column HP-5MS, including 16 esters, 17 alcohols, 3 acids, and 6 others; the column CP-Wax was isolated 24 times. The main volatile substances include 15 esters, 4 alcohols, 4 acids, and 1 other.

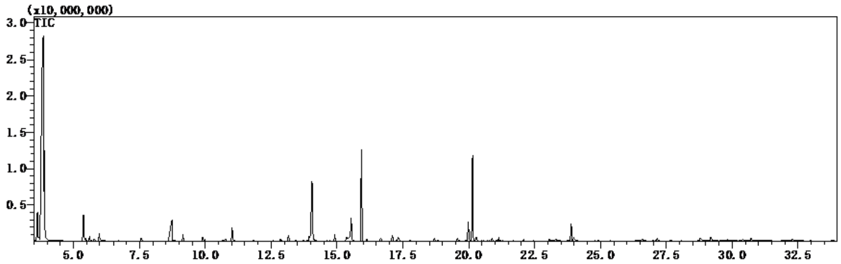

Fig.1 Total ion chromatogram of volatile components in pitaya wine on HP-5MS

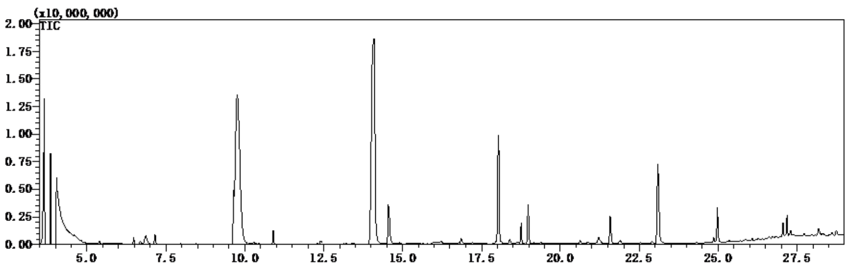

Fig.2 Total ion chromatogram of pitaya wine on CP-Wax

Table 1. The relative content of aroma volatile compounds in pitaya wine by HP-5MS and CP-Wax

No.	Compounds	Formula	Molecular Mass	Relative Abundances/% HP-5MS	CP-Wax
Esters					
1	Octanoic acid, ethyl ester	$C_{10}H_{20}O_2$	172.26	12.69	12.1
2	Butyl glycolate	$C_6H_{12}O_3$	132.15	9.79	/
4	Dodecanoic acid, ethyl ester	$C_{14}H_{28}O_2$	228.37	7.93	1.34
5	Decanoic acid, ethyl ester	$C_{12}H_{24}O_2$	200.31	/	5.02
6	Butanedioic acid, diethyl ester	$C_8H_{14}O_4$	174.19	2.92	0.8
7	Hexanoic acid, ethyl ester	$C_8H_{16}O_2$	144.21	2.49	0.3
8	Ethyl dec-9-enoate	$C_{12}H_{22}O_2$	198.30	0.96	0.76
9	Acetic acid, 2-phenylethyl ester	$C_{10}H_{12}O_2$	164.20	0.87	0.8
10	1-Butanol, 3-methyl-, acetate	$C_7H_{14}O_2$	130.18	0.76	0.98
11	Octanoicacid,3-methylbutyl ester	$C_{13}H_{26}O_2$	214.34	0.5	0.33
12	2(3H)-Furanone, dihydro-	$C_4H_6O_2$	86.08	0.4	/
13	Pentadecanoic acid, ethyl ester	$C_{17}H_{34}O_2$	270.45	0.33	0.16
14	Isoamyl lactate	$C_8H_{16}O_3$	160.21	0.32	0.89
15	Nonanoic acid, ethyl ester	$C_{11}H_{22}O_2$	186.29	/	0.24
16	Propanoic acid, 2-hydroxy-, ethyl ester	$C_5H_{10}O_3$	118.13	0.26	0.18
17	Hexadecanoic acid, ethyl ester	$C_{18}H_{36}O_2$	284.47	0.24	/
18	ethyl 3-methylbutyl butanedioate	$C_{14}H_{26}O_4$	269.36	/	0.19
19	Geranyl acetate	$C_{12}H_{20}O_2$	196.28	/	0.18
20	Butyl octanoate	$C_{12}H_{24}O_2$	200.31	0.11	/
21	2-Propenoic acid, octyl ester	$C_{11}H_{20}O_2$	184.27	0.11	/
Alcohols					
1	1-Butanol, 3-methyl-	$C_7H_{14}O_2$	130.18	32.27	32.76
2	Phenylethyl Alcohol	$C_8H_{10}O$	122.16	6.81	5.35
3	1-Butanol, 2-methyl-	$C_5H_{12}O$	88.14	5.13	/
4	(2R,3R)-(-)-2,3-Butanediol	$C_4H_{10}O_2$	90.12	2.25	/
5	1-Butanol, 2-methyl-, (S)-	$C_5H_{12}O$	88.14	2.06	1.08
6	1-Propanol, 2,2-dimethyl-	$C_5H_{12}O$	88.14	0.66	/
7	1-Nonanol	$C_9H_{20}O$	144.25	0.64	/
8	1-Hexadecanol	$C_{16}H_{34}O$	242.44	0.48	/
9	2,3-Butanediol	$C_4H_{10}O_2$	90.12	0.42	/
10	2-Propanol, 1-propoxy-	$C_6H_{14}O_2$	118.18	0.23	/
11	beta.-Citronellol	$C_{10}H_{20}O$	156.27	0.29	0.19
12	2-Tridecanol	$C_{13}H_{28}O$	200.36	0.22	/
13	1-Undecanol	$C_{11}H_{24}O$	172.30	0.19	/
14	1,8-Cineole	$C_{10}H18O$	154.24	0.18	/
15	1-Octanol	$C_8H_{18}O$	130.23	0.17	/
16	1-Tridecanol	$C_{13}H_{28}O$	200.36	0.17	/
17	1-Decanol	$C_{10}H_{22}O$	158.28	0.12	/
Acids					/
1	Acetic acid	$C_2H_4O_2$	60.05	/	2.58
2	Hexanoic acid	$C_6H_{12}O_2$	117.15	0.54	/
3	Decanoic acid	$C_{10}H_{20}O_2$	172.26	0.36	0.33
4	Octanoic acid	$C_8H_{16}O_2$	144.21	0.31	1.54
5	Benzoic acid	$C_7H_6O_2$	123.11	/	0.28
Others					
1	Hexadecane	$C_{16}H_{34}$	226.44	16.48	/
2	Benzeneacetaldehyde	C_8H8O	120.14	0.11	0.13
3	Dodecane	$C_{12}H_{26}$	170.33	0.63	/
4	Pentadecane	$C_{15}H_{32}$	212.41	0.23	/
5	3-Phenylphthalide	$C_{14}H_{10}O_2$	210.22	0.27	/
6	Ethanone, 1-phenyl-	C_8H_8O	120.14	0.11	/

A comparison chart of the main substances detected by HP-5MS and CP-Wax is shown in Figure 3, the relative contents of pitaya wine are esters, alcohols and acids, among which the main aromas are Octanoic acid, ethyl ester, Dodecanoic acid, ethyl ester, Butanedioic acid, diethyl ester, 1-Butanol, 3-methyl-, acetate 1-Butanol, 3-methyl-, Phenylethyl Alcohol, and Octanoic acid can be separated using HP-5MS and CP-Wax. The main aroma components of pitaya wine can be well separated into two kinds of chromatographic columns. The two columns have similar effects on the separation of esters, but the effect on separation of alcohols is quite different, such as 1-Butanol, 2-methyl-, (2R,3R)-(-)-2,3-Butanediol. 1-Propanol, 2,2-dimethyl-, 1-Nonanol can only be separated in HP-5MS, which is related to the polarity of the column. In addition, hydrocarbons and ketones are mainly separated by column HP-5MS, such as Ethanone, 1-phenyl-, Hexadecane, Dodecane, and they are only separated by HP-5MS. Therefore, if the hydrocarbons and ketones in pitaya wine are studied, HP-5MS columns should be used for separation.

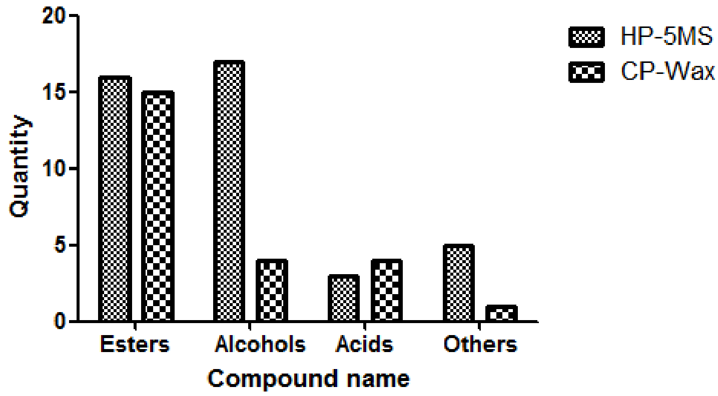

Fig. 3 The main aroma components in pitaya wine by HP-5MS and CP-Wax

Except for the main aroma component of pitaya, most of the other aroma components detected by two different polarity columns were detected after separation by column HP-5MS. Therefore, it is better to choose the HP-5MS column for the determination of non-essential aroma substances in pitaya wine. Therefore, in the analysis of other aroma components in pitaya, different columns should be selected according to the different target substances studied.

4. Conclusion

In this paper, HS-SPME was used to enrich the aroma components of pitaya wine, which were separated by two different polar chromatographic columns. GC-MS analysis was used to compare the aroma components of Pitaya wine with different polarity columns. The results showed that there were significant differences between the two columns in the determination of the aroma components of pitaya wine. The main aroma compounds measured using the HP-5MS column had more types and contents than the CP-Wax column.

5. Acknowledgment

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