

GC-MS-O Coupled with PLS-DA for Classification and Authentication of Eleven Colorful Pomelo Varieties from China

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Abstract To differentiate eleven colorful pomelo varieties, which came from the main producing area of four geographical province in China, physicochemical properties and sensory evaluation was studied. Headspace solid-phase microextraction (HS-SPME) coupled with gas chromatography-mass spectrometry and olfactometry (GC-MS-O) was used to determinate and characterize volatile components. Olefins, aldehydes, alcohols and ketones represented the most abundant volatile compounds in all varieties of colorful pomelo juices. 38 aroma-active compounds were perceived by the trained panel of judges by using detection frequency analysis method (DFA). Partial least squares-discriminant analysis (PLS-DA) combining DFA analysis was applied to distinguish four main origins of the eleven colorful pomelo varieties from China.

Keywords: geographical identification, colorful pomelo, GC-MS, PLS-DA

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1. Introduction

Pomelo (*Citrus grandis* (L.) Osbeck) is one of the most widely cultivated and consumed fruit worldwide due to its nutritional quality and pleasant fresh flavor [1]. It is native to China, southeastern Asia and America (FAO). China is the main producing area of pomelo, with the highest planting area and yield in the world. Pomelo is also classified as common (with white color) or colored (with yellow or pink or red color). There are too many varieties of pomelo in China, of which Fujian, Jiangxi, Zhejiang and Chongqing covering major production areas also covers all colorful pomelo varieties. Compared to white pomelo varieties, colorful pomelo varieties attract more customers and have greater consumption potential. However, the distinctive colorful pomelo physicochemical properties and aroma should be studied. To identify and compare the quality of varieties from different regions and choose suitable varieties for processing, research on origin of colorful pomelo is urgent, in order to break the bottleneck in the industrial development of pomelo.

Many analytical methods, especially based on chromatography and mass spectrometry techniques, such

as IR, UV, FT-IR, TLC, HPLC, ESI-MS, GC-MS, QTOF-MS, LC-QQQ-MS/MS and NMR etc., were carried out on the evaluation of volatile constituents like apples, pears, almonds, peaches, beer, flower, honey, oil, peanuts, etc [2,3,4,5,6,7,8]. One of the most critical steps in flavor analyses is sample preparation [9]. Considering the complexity of natural food matrices, headspace solid-phase microextraction (HS-SPME) is recommended to get a more complete volatile profile, which was widely used for aromas and fragrance preparation in many foods [10,11]. It is simple, fast, solventfree, non-destructive, reproducible and it can obtain a representative aroma extract nearly un-biased under ultra-high vacuum [12]. Therefore, the combined use of HS-SPME and GC-MS could be a good choice for comprehensive capture of odors from the complex colorful pomelo juices matrix.

The headspace of colorful pomelo juice is rather complex; peaks co-elute and some potent odorants without peak signal are observed when using GC-MS [13]. Not all components could contribute to the flavors, and only the odor-active volatiles are regarded as significant. Coupling of GC-MS with olfactometry (GC-MS-O) is an essential tool for location of aroma-active constituents [14]. GC-MS-O has been applied to investigate the complex volatile composition and tracelevel analytes of various foods,

which combines the enhanced efficiency of separation, reliability in qualitative analysis, information on the samples and its components with high sensitivity, selectivity of detection and accurate mass determination [15,16]. Coupling of HS-SPME and GC-MS-O was employed here in to gather information on the analysis of volatile composition and odors in different colorful pomelo varieties.

Most studies related to volatile components in pomelos have been carried out on the qualitative descriptions of volatiles, or merely analysis of volatiles but not odor-active compounds, or characterization of volatiles using single volatile extraction methods, which may not necessarily guarantee the complete capture of all potential volatile components and accurate identification of odor contributors. What is more important, it is difficult to link the phychemical characteristics and odors of different varieties with their geographical location. The objectives of the present study were, firstly to identify aroma attributes based on comparative flavor profile analysis by application of GC-MS; and then to identify and verify the predominant contributors in different colorful pomelo juices by using comparative aroma dilution analysis/gas chromatography-olfactometry (GC-O) techniques, odor activity value (OAV) calculation and addition experiments, and finally to investigate the correlation of between varieties and geographical locations by PLS-DA. The principle of PLS-DA is a classical PLS regression which expresses a class membership and the response variable is binary. Attributing a sample to other groups than the ones first defined is not allowed and all measured variables play the same role with respect to the class assignment in PLS-DA [17]. Therefore, PLS latent variables are built to have a proper compromise between two purposes: describing the set of explanatory variables and predicting the response ones [18]. A PLS-DA classification could well benefit from such a property in the direction of classification and authentication of fruit and vegetable quality characteristics.

2. Materials and Methods

2.1. Colorful Pomelo Juice Samples

Table 1. Origins of different colorful pomelo from China

Samples	Chinese name	Origins (Province)	Locality (City)
FD	Fendu	Chongqing	Fengdu
MJ	Majia you	Jiangxi	Shangrao
NH	Nanhong	Jiangxi	Ganzhou
HR	Hongrou	Jiangxi	Ganzhou
QD	Qiangdele	Jiangxi	Ganzhou
XY	Xiyong	Jiangxi	Ganzhou
PH	Pinghe	Fujian	Guanxi
SH	Sanhong	Fujian	Guanxi
HJ	Huangjin	Fujian	Pinghe
CH	Chuhongyou	Zhejiang	Lishui
HY	Huyong	Zhejiang	Changshan

Colorful pomelo fruit from China were picked and purchased during their harvesting period between November 2023 and January 2024. Eleven varieties of colorful pomelo were selected from four different

geographical regions of China: FD (Fengdu), MJ (Shangrao), NH (Ganzhou), HR (Ganzhou), PH (Guanxi), SH (Guanxi), CH (Lishui), HY (Changshan), QD (Ganzhou), XY (Ganzhou) and HJ (Pinghe). More information about the samples is provided in Table 1. All fresh colorful pomelo fruits were immediately refrigerated, transported to the laboratory, and juiced with a screw extractor (GT6G7, Zhejiang Jixie Co., Zhejiang, China). The colorful pomelo juices were filled into 60-mL EVOH plastic bottles and frozen in liquid nitrogen within 12 h and stored at -80°C to minimize the loss of any volatile constituents [19].

2.2. Physicochemical Properties

The colorful pomelo juices were equilibrated at 25±2°C to measure physicochemical properties, i.e. pH value, TA (titrable acid) and TSS (total soluble solid). The pH was tested by Thermo Orion 868 pH meter (Thermo Fisher Scientific, Inc., MA, U.S.A). TA was quantified by 842 GPD titrino, automatic potentiometric titrator (Metrohm, Switzerland) and the data was expressed as percentages of citric acid content. TSS was evaluated by WAY-2S digital Abbe refractionmeter (Shanghai Precision and Scientific Instrument Co., Shanghai, China), and the results were showed as °Brix [20].

Total phenols were tested by the Folin-Ciocalteu method by a spectrophotometer (UV-726, Shanghai Precision and Scientific Instrument Co., Shanghai, China) described by Singleton et al. with some modifications, and the results were expressed as mg of gallic acid equivalent (GAE) per 100 g colorful pomelo juice [21]. The color value was determined by the color measurement spectrophotometer (Hunter Lab Color Quest XE, Hunter Associates Laboratory, Inc., Virginia, USA) in the reflectance mode. To detect sugar, the liquid chromatograph (LC-20AT) combined with UV/Vis detector (SPD-20AV), as well as the auto sampler (SIL-20A) with column oven (CTO-20A) from Shimadzu Co., Japan, equipped with the Sunfire TM C18, (Waters, 4.6×250 mm i.d, 5 µm particle size), was used as described, the content of fructose, glucose and sucrose in colorful pomelo juices were quantified by the standard curve with R² greater than 0.99 [22].

2.3. Headspace Solid-phase Microextraction

The colorful pomelo juices were put in a sealed glass flask and equilibrated for 10 min at 40°C in water bath. A manual headspace solid-phase micro-extraction (HS-SPME, Supelco, Inc., Bellefonte, PA, USA) device was used to extract volatile components of colorful pomelo juices. A volume of 20 mL infusion was transferred to a screw cap headspace vial (50 mL; Beijing Bomex Co., Beijing, China) and equilibrated in the same condition in which 20 µL blended internal standard was added. The fiber coated with 50/30-µm polydimethylsiloxane/divinylbenzene/ carboxen was exposed to the headspace of the vial in 40°C for 30 min with agitation at 90 RPM [23,24]. Internal standards of cyclohexanone were purchased from Sigma-Aldrich Co., Ltd. (Milwaukee, WI, USA) with purity >99%. N-Alkanes (C5-C40) was used to make linear retention index (LRI)

calculation and obtained from J&K Chemical Ltd. (Beijing, China).

2.4. Gas Chromatography-mass Spectrometry Analysis

The SPME extract was injected into the port of the Agilent 7890A-5975C GC/MSD (Agilent Technologies) equipped with a DB-5 capillary column (30 m × 0.25 mm, 0.25 µm film thickness), and desorbed at 250°C for 3 min. The helium (99.999% purity) was used as the carrier gas for vial pressurization and circulated in split mode (1:50). The flow rate was maintained at 1 mL/min. The oven temperature was initially kept at 40°C for 3 min, and increased at 2°C min⁻¹ to 140°C and then increased to 250°C at a speed of 20 °C min⁻¹ where it was held for 6 mins. The mass detection was conducted with electronic impact (EI) mode at 70 eV, and source temperature was operated at 250°C. The mass spectrum was scanned in the range of m/z 39 to 450 amu at 1 s intervals. The volatile components were indentified by matching the mass spectra with their data in the National Institute of Standards and Technology (NIST08), and confirmed with the retention index (RI, error <5%), as well as aroma description (AD). The volatiles chosen in this paper, has gave the similarity of more than 70 (maximum data is 100) [24].

2.5. Gas Chromatography-olfactometry

The sniffing port (Sniffer 9000, Brechbühler, Switzerland) combined with a GC-MS (7890A- 5975C, Agilent Technologies, Inc.) was used to test the odor-active components. Equipped with a capillary column, the effluents were split 1:1 (by volume) into a sniffing port and a MS detector using the Agilent capillary flow technology. The transfer line to the GC-O sniffing port was set at 240 °C. The sample preparation and GC-MS conditions were identical to those described above.

Eight trained assessors, who were mixed with both sexes and age range of 20 to 50 years participated in the detection frequency analysis to sniff the GC effluent and gave their data by GC-O analysis. The panelists were professional trained prior to the sensory evaluation in order to be familiar with odor descriptions of characteristics and intensities for artificial odorants and different colorful pomelo juices. Eight GC-O runs were conducted (one run for each assessor) to perceive aroma-active compounds from the sniffing mask. Any odorant was considered to have potential aroma activity whose detection frequencies were more than twice [24].

2.6. Statistical Data Analysis

The data was analyzed by performing ANOVA (analysis of variance) using SPSS, version 17.0 (SPSS Inc., 2009) and Duncan's test. Significant differences were established at $P < 0.05$. Results have been given as mean ± standard deviation. All experiments were performed in triplicates. Multivariate analysis was conducted using PLS-DA based on the correlation matrix to determine which volatiles contributed most to differentiate the colorful pomelo juices from different origins of China.

3. Results and Discussion

3.1. Physicochemical Properties of Colorful Pomelo Juices

3.1.1. Juice Yield Analysis

Juice yield is an important indicator in juice production, and the higher the juice yield, the higher the economic benefits. As shown in Figure 1, there was a significant difference in juice yield among different colorful pomelo varieties, such as MJ (39.51%), PH (31.56%), SH (31.02%), XY (27.98%), and FD (27.07%) have higher juice yield and are suitable for juice processing. However, QD (20.23%) has the lowest juice yield and higher juice processing costs. This was in accordance with conclusions drawn in other studies that juice yield of Guifei red pomelo (27.7%) from Fujian province was higher than the Honey pomelo (25.03%) from Guanxi province and Shatian pomelo (17.13%) from Guangxi province [25].

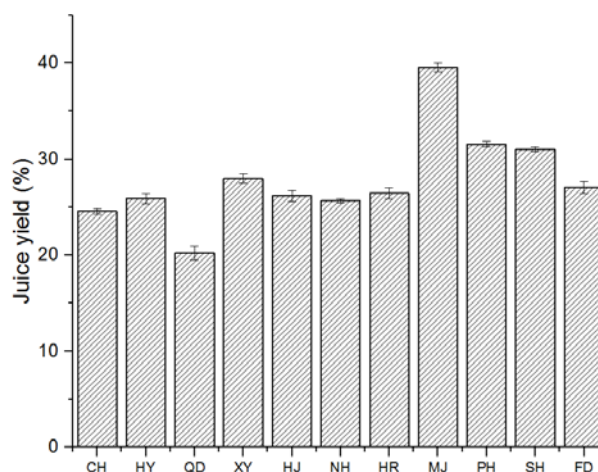


Figure 1. The juice yield of different colorful pomelo varieties

3.1.2. Sugars and TSS Analysis

As shown in Table 2, the fructose and glucose content among different colorful pomelo varieties ranged from 7.81 to 7.84 (g/100 g FW), and the differences were not significant. There were significant differences in sucrose content among different varieties, with the highest content in SH (8.06 g/100 g FW), followed by NH (8.04 g/100 g FW). There was no significant difference in sucrose content among HJ, FD and HR (8.00 g/100 g FW), followed by HY (7.98 g/100 g FW), QD (7.97 g/100 g FW), XY (7.96 g/100 g FW), CH (7.95 g/100 g FW), and MJ pomelo had the lowest content (7.89 g/100 g FW). The content of total sugar ranges from 23.59 to 23.73 (g/100 g FW), in which SH has the highest total sugar content. There is a significant difference in the content of TSS among different pomelo varieties. The TSS content of PH (9.88 ° Brix) and SH (9.64 ° Brix) was relatively close to that of Guifei red pomelo by our previous study (12.96 ° Brix) [17]. All of the three varieties were from Fujian province. In different years, climate conditions, cultivation conditions, and management conditions, the content of sugars and TSS were also different [26].

Table 2. Glucose, fructose, sucrose, total sugar and total soluble solids (TSS) of different colorful pomelo varieties

Samples	Sugar (g/100 g FW)			Total sugar (g/100 g FW)	TSS (°Brix)
	Fructose	Glucose	Sucrose		
CH	7.82±0.01 a	7.83±0.02 ab	7.95±0.01 b	23.59±0.02 b	9.37±0.12 cd
HY	7.81±0.01 a	7.82±0.01 ab	7.98±0.01 de	23.61±0.02 cd	9.34±0.14 c
QD	7.82±0.02 a	7.82±0.01 a	7.97±0.01 cd	23.60±0.12 c	8.84±0.13 b
XY	7.83±0.01 a	7.83±0.01 ab	7.96±0.02 bc	23.62±0.03 de	9.49±0.03 de
HJ	7.81±0.01 a	7.81±0.02 a	8.00±0.02 ef	23.63±0.01 e	9.45±0.14 cd
FD	7.81±0.02 a	7.82±0.01 ab	8.01±0.01 fg	23.63±0.01 e	8.86±0.11 b
MJ	7.82±0.01 a	7.83±0.02 ab	7.89±0.03 a	23.55±0.04 a	8.14±0.18 a
NH	7.81±0.03 a	7.81±0.01 a	8.04±0.01 h	23.66±0.02 f	9.93±0.12 f
HR	7.81±0.01 a	7.82±0.01 ab	8.00±0.02 fg	23.63±0.01 e	9.53±0.11 de
PH	7.82±0.01 a	7.82±0.02 ab	8.01±0.01 g	23.66±0.02 f	9.88±0.09 f
SH	7.83±0.01 a	7.84±0.01 b	8.06±0.01 i	23.73±0.03 g	9.64±0.09 e

All data were the means ± SD, n=3. Values with different letters within one row are significantly different ($P<0.05$).

3.1.3. pH Analysis

As shown in Figure 2, there was a significant difference in pH values among different colorful pomelo varieties. QD has the highest pH value (4.62), followed by NH (4.22), HJ (4.13), MJ (4.07), SH (3.96), FD (3.94), PH (3.87), HR (3.86), HY (3.8), and XY (3.76), while CH has the lowest pH value (3.73). It was reported that pH values of Guanxi honey pomelo and Guifei red pomelo were 3.52 and 3.48 [23], which were closer to the results of PH (3.87) and SH (3.96), all of which were Guanxi pomelo varieties. The pH values of all eleven colorful pomelo varieties were below 4.6, indicating that they were acidic foods. In general, the higher the acidity, the better the sterilization effect in the same processing conditions, which is beneficial for the subsequent storage, circulation, and preservation after sterilization treatment [27].

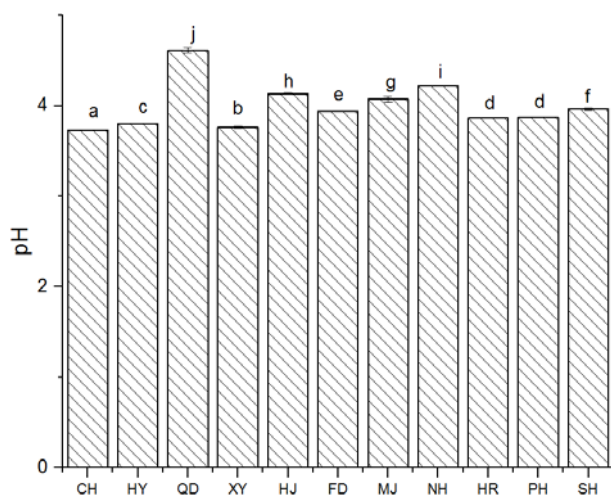


Figure 2. The pH value of different colorful pomelo varieties

3.1.4. TA Analysis

As given in Figure 3, the highest content of TA was HR (0.94%), followed by PH (0.86%), FD (0.85%), and CH (0.82%), which was no significant difference among these three varieties. The content of TA from high to low in turn was XY (0.79%), SH (0.72%), HY (0.71%), NH (0.68%), HJ (0.65%), MJ (0.61%) and QD (0.48%). According to our previous research, the TA content of Guifei red pomelo was 0.89%, which was closest to PH which came from the same origin (0.86%), further confirmed the reliability of the data [19]. QD has the lowest TA content, which corresponded to the highest pH value. The content

of TA is an important indicator for fruit quality, which has significant impact on the flavor, color, quality, and storage characteristics of fruits. In the process of fruit juices (such as lemon, apple, grapefruit, green plum, passion fruit, etc.), it is more inclined to choose varieties with higher acidity [28,29,30,31]. Therefore, when selecting pomelo juice varieties, higher acidity means more suitable processing.

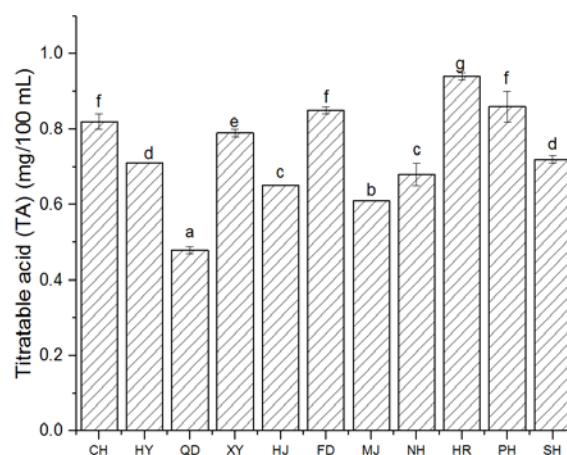


Figure 3. Titratable acid (TA) of different colorful pomelo varieties

3.1.5. Analysis of Sugar To Acid Ratio

The sugar to acid ratio is an indicator to characterise the taste of fruit juice. As shown in Figure 4, QD has the highest sugar to acid ratio (18.18), which has less acidity, more sweetness, and a softer taste, followed by NH (14.54), HJ (14.3), MJ (13.47), SH (13.25), PH (11.52) and FD (10.3). The varieties with a low content of sugar to acid ratio were HR (9.97), HY (9.65), XY (9.33), and CH (7.74). These four varieties of colorful pomelo have a strong acidity, low sweetness, and poor taste after juicing.

3.1.6. Analysis of Total Phenols

As given in Figure 5, the total phenolics have significant difference in different colorful pomelo varieties. The highest total phenolics was found in CH (19.52 mg GAE/100 g FW), followed by QD (18.5 mg GAE/100 g FW), HY (17.18 mg GAE/100 g FW), HJ (15.78 mg GAE/100 g FW), FD (15.74 mg GAE/100 g FW), XY (15.58 mg GAE/100 g FW), MJ (15.33 mg GAE/100 g FW), NH (14.24 mg GAE/100 g FW), SH (13.55 mg GAE/100 g FW), PH (13.24 mg GAE/100 g FW) and HR (12.71 mg GAE/100 g FW).

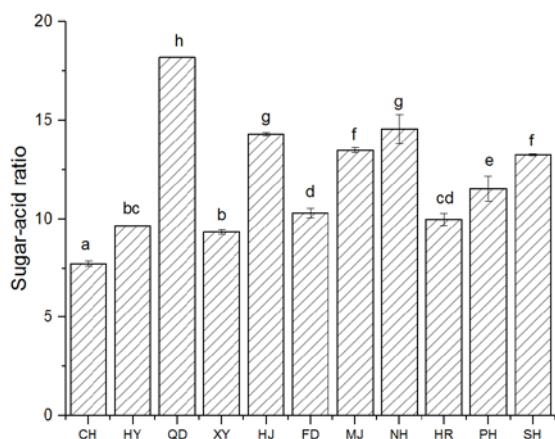


Figure 4. Sugar-acid ratio of different colorful pomelo varieties

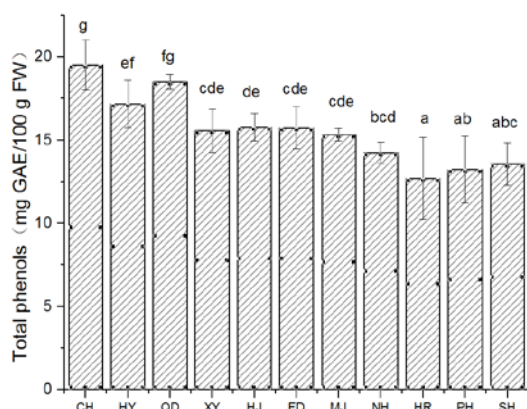


Figure 5. Total phenolics in different colorful pomelo varieties

3.1.7. Color Analysis

Color differences of different pomelo juices were shown in Table 3 by measuring Hunter L^* , a^* , and b^* . A higher L^* value was exhibited in HJ samples compared with other varieties, while the L^* value has no significant difference in other samples. The highest b^* value was also exhibited in HJ samples. This results was similar to what was observed with the naked eye that HJ presented a bright yellow color and other varieties exhibited a relatively weaker brightness and varying degrees of red.

3.2. Volatile Compounds of Colorful Pomelo Juices

3.2.1. Characterization of Volatile Compounds Analyzed by HS-SPME-GC-MS

Table 4 showed the volatile compounds and their relative content extracted from eleven colorful pomelo juices by HS-SPME. 70 volatiles of colorful pomelo juice samples were identified and categorized as olefins, aldehydes, alcohols, ketones, esters, etc. There were significant differences in the types and contents of volatile components in colorful pomelo from different regions and varieties. A total of 34 compounds were detected from FD, followed by XY (32), CH (31), HJ (24), SH (23), QD (22), MJ and NH (20), HY and PH (18) and HR (16).

Obviously, olefins were the predominant compounds in colorful pomelo juices which was also reported by other studies [1]. According to the relative content of olefin classes, the XY (62.77%) contained the highest amount, followed by XY (62.77%), FD (53.35%), MJ (50.23%), QD (50.1%), CH (38.88%), etc. The alcohols, as the second most important family of volatile compounds in colorful pomelo juices, has shown the higher content in HJ (33.08%), HR (31.78%), HY (31.69%) and PH (30.31%), and the lowest content in QD (0.05%). In addition, there were small amounts of aldehydes, ketones, esters and other substances in each variety.

Limonene, nonanal and decanal were common components among eleven colorful pomelo varieties in China, while their contents vary among different varieties. This was in accordance with conclusions drawn in other studies that limonene and nonanal were important volatiles in pomelo peel and citrus fruits [32]. As the predominant volatile, limonene has showed the highest relative content of volatiles identified by SPME/GC-MS in QD (33.47%), followed by FD (31.79%), XY (21.64%), SH (12.07%), HY (9.7%) and CH (7.9%). The main volatile components of HJ were hexanol and hexanal, with the relative content of 28.48% and 5.82%, respectively.

Each colorful pomelo variety has some unique volatiles. Junipene, perillene, α -guaiene and (E)-linalool were only detected in CH samples; (E)-2-heptenal and 3-octanone were only detected in HY samples; β -elemene and 4-hydroxy-3-methoxystyrene were only detected in QD samples; cuminal were only detected in XY samples; α -ionone, (E)-geranylacetone and β -ionone were only detected in HJ samples; 3-carene and γ -terpinene were only detected in FD samples.

Table 3. Color parameters in different colorful pomelo varieties

Samples	Color	Color index		
		L^*	a^*	b^*
CH	slight red	33.69±0.17 abc	-0.11±0.28 d	1.08±0.51 c
HY	slight yellow	33.63±0.66 ab	-1.98±0.27 a	2.85±0.21 e
QD	red	34.36±0.42 bcd	2.54±0.27 f	2.3±0.25 d
XY	slight yellow	33.7±0.13 ab	-0.97±0.2 bc	0.53±0.24 b
HJ	yellow	36.01±0.27 e	-0.21±0.11 d	2.38±0.21 d
FD	pink	34.45±0.36 cd	0.52±0.35 e	1.22±0.23 c
MJ	pink	35.39±0.79 d	-0.86±0.25 c	0.63±0.15 b
NH	slight red	33.27±0.25 a	-1.29±0.18 b	-0.79±0.23 a
HR	slight red	33.88±0.26 abc	-0.44±0.18 cd	-0.33±0.16 a
PH	slight yellow	34.69±0.25 abcd	-0.72±0.15 c	-0.78±0.18 a
SH	red	33.83±0.31 abc	0.9±0.22 e	0.16±0.34 b

All data were the means $\pm$ SD, n=3. Values with different letters within one row are significantly different ($P<0.05$)

Table 4. Composition and relative content (%) of volatiles identified by SPME/GC-MS in different colorful pomelo juices

Compounds	RI ^a	MI	Relative content (mean±SD)/% ^b										
			CH (31)	HY (18)	QD (22)	XY (32)	HJ (24)	FD (34)	MJ (20)	NH (20)	HR (16)	PH (18)	SH (23)
Olefins (42)													
<i>α</i> -pinene	926	RI, MS, AD	-	-	-	-	-	0.07± 0.01	-	-	-	-	-
<i>β</i> -pinene	968	RI, MS, AD	-	-	0.36± 0.02	0.25±0.01	-	0.17± 0.05	-	-	-	-	-
myrcene	969	RI, MS, AD	0.05±0.02	-	1.68± 0.8	-	-	-	-	-	-	-	-
<i>β</i> -myrcene	969	RI, MS, AD						1.36± 0.13	-	5.41±0.11	-	-	0.1±0.06
junipene	969	RI, MS, AD	0.05±0.01	-	-	-	-	-	-	-	-	-	-
limonene	1025	RI, MS, AD	7.9±1.09	9.7±0.88	33.47 ±2.18	21.64±1.65	3.24±0.68	31.79 ±3.14	3.4±0 .33	7.23±0.07	6.61± 0.51	5.21±0.72	12.07±2.07
terpinolene	1056	RI, MS, AD	0.04±0.01	0.06±0.02	-	0.18±0.01	-	-	-	-	-	-	-
perillene	1101	RI, MS, AD	0.09±0.01	-	-	-	-	-	-	-	-	0.15±0.04	0.3±0.03
<i>δ</i> -elemene	1340	RI, MS, AD	5.62±0.78	-	0.75± 0.23	2.31±0.38	-	6.43± 0.38	3.38± 0.04	0.65±0.05	-	-	0.04±0.04
<i>α</i> -copaene	1375	RI, MS, AD	0.31±0.02	-	0.13± 0.07	0.08±0.01	-						
copaene	1375	RI, MS, AD	-	-	-	-	-	0.31± 0.03	1.22± 0.3	-	-	-	-
germacrene	1389	RI, MS, AD	5.05±0.78	0.05±0.02	1.32± 0.07	0.74±0.12	0.04±0.05	0.11± 0.01	8.33± 0.25	0.60±0.01	0.11± 0.01	-	0.02±0
elemene	1391	RI, MS, AD	-	-	-	-	-	2.24± 0.02	-	-	-	-	-
<i>β</i> -elemene	1391	RI, MS, AD	-	-	0.34± 0.05	-	-	-	-	-	-	-	-
<i>α</i> -gurjunene	1408	RI, MS, AD	0.04±0.01	-	-	0.92±0.02	-	0.13± 0.03	0.08± 0.01	-	-	-	-
aromadendrene	1410	RI, MS, AD	0.36±0.02	-	0.06± 0.02	2.19±0.19	-	0.22± 0.02	0.77± 0.01	-	-	-	-
isocaryophylle ne	1415	RI, MS, AD	-	-	-	18.47±0.03	0.07±0.02	-	-	-	-	-	-
<i>β</i> -cubebene	1424	RI, MS, AD	6.02±0.19	0.05±0.02	0.71± 0.09	0.38±0.04	-	1±0.0 9	8.3±1 .21	0.2±0.01	0.07± 0.04	-	0.02±0.01
alloaeromaden drene	1435	RI, MS, AD	0.27±0.01	-	-	0.1±0.04	-	0.09± 0	-	-	-	-	-
<i>α</i> -guaiene	1435	RI, MS, AD	0.18±0.1	-	-	-	-	-	-	-	-	-	-
cadinene	1440	RI, MS, AD	3.97±0.11	-	1.4±0 .04	1.07±0.12	-	-	-	-	-	-	-
<i>α</i> - caryophyllene	1449	RI, MS, AD	3.42±0.24	-	1.69± 0.66	10.47±0.03	-	1.2±0 .04	-	2.27±0.05	-	-	-
<i>β</i> - caryophyllene	1420	RI, MS, AD	-	-	-	-	-	-	-	0.08±0.01	0.11± 0.03	1.70±0.66	16.47±0.03
<i>γ</i> -muurolene	1464	RI, MS, AD	2.11±0.14	0.02±0.01	0.47± 0.01	0.50±0.05	0.05±0.11	1.55± 0.17	6.30± 1.85	0.34±0.02	0.12± 0.05	-	0.04±0.01
<i>β</i> -ocimene	1045	RI, MS, AD	-	-	-	-	-	0.05± 0.01	-	0.49±0.01	-	-	-
<i>α</i> -ocimene	1045	RI, MS, AD	-	-	-	-	-	0.06± 0.02	-	-	-	-	-
<i>γ</i> -selinene	1466	RI, MS, AD	-	-	-	0.62±0.08	-	-	-	0.33±0.05	0.09± 0.03	0.05±0.02	0.04±0
<i>β</i> -selinene	1468	RI, MS,	0.32±0.03	-	-	0.13±0.02	-	0.23± 0.02	0.49± 0.06	-	-	0.17±0.06	-

Compounds	RI ^a	MI	Relative content (mean±SD)/% ^b										
			CH (31)	HY (18)	QD (22)	XY (32)	HJ (24)	FD (34)	MJ (20)	NH (20)	HR (16)	PH (18)	SH (23)
		AD											
α -selinene	1481	RI, MS, AD						0.17±0	-	0.32±0.02	0.09±0.01	-	0.05±0
α -cubebene	1472	RI, MS, AD	0.65±0.03	-	0.21±0.04	0.22±0.03	-	1.15±0.62	2.63±1.1	-	-	-	-
δ -cadinene	1440	RI, MS, AD	-	-	-	-	-	3.19±0.29	9.58±0.1	0.98±0.07	-	-	-
γ -cadinene	1480	RI, MS, AD	-	-	-	-	-	1.46±0.11	2.37±0.09	0.38±0.03	-	-	-
3-carene	1055	RI, MS, AD	-	-	-	-	-	0.05±0.02	-	-	-	-	-
γ -terpinene	1055	RI, MS, AD	-	-	-	-	-	0.06±0.01	-	-	-	-	-
terpinene	1056	RI, MS, AD	-	-	-	-	-	0.28±0	-	-	0.07±0.01	0.07±0.02	0.04±0.01
α -terpinene	1340	RI, MS, AD	-	-	-	-	-	-	3.34±0.2	0.67±0.02	-	0.07±0.02	-
γ -cadinene	1480	RI, MS, AD	1.95±0.14	-	0.32±0.05	0.23±0.11	0.08±0.03	-	-	-	-	-	-
calamenene	1483	RI, MS, AD	-	0.1±0.01	0.16±0.05	1.07±0.12	0.13±0.06	-	-	-	-	-	-
β -caryophyllene oxide	1500	RI, MS, AD	0.25±0.08	-	-	0.4±0.02	-	0.03±0.01	-	-	-	-	-
valencene	1510	RI, MS, AD	0.23±0.04	-	1.08±0.02	0.08±0.02	-	-	0.02±0.01	1.35±0.05	0.3±0.02	0.27±0.09	0.23±0.02
guaiene	1529	RI, MS, AD	-	-	-	-	-	-	0.02±0.02	-	-	-	-
styrene	890	RI, MS, AD	-	-	5.95±1.92	0.72±0.02	0.65±0.12	-	-	-	-	-	-
Total			38.88±2.21	9.98±1.21	50.1±3.54	62.77±3.31	4.26±0.06	53.35±2.35	50.23±4.25	21.3±2.14	7.57±0.32	7.69±0.54	29.42±2.32
Alcohols (9)													
hexanol	871	RI, MS, AD	3.33±0.71	15.25±2.12	-	3.36±0.15	28.48±3.97	5.02±1.44	1.81±0.01	12.47±1.0	27.05±2.61	27.21±5.53	15.69±2
1-heptanol	965	RI, MS, AD	-	14.79±0.85	-	0.08±0.02	2.50±0.04	-	-	-	2.44±0.15	1.77±0.2	2.11±0.19
octanol	1069	RI, MS, AD	-	1.27±0.66	-	-	1.46±0.07	-	-	-	1.74±0.32	0.99±0.12	1.67±0.1
(E)-linalool	1071	RI, MS, AD	0.23±0.01	-	-	-	-	-	-	-	-	-	-
cuminol	1101	RI, MS, AD	-	-	-	0.06±0.01	-	-	-	-	-	-	-
nonanol	1175	RI, MS, AD	-	0.38±0.03	-	-	0.72±0.08	-	-	-	0.55±0.12	0.34±0.01	0.72±0.02
α -terpineol	1193	RI, MS, AD	-	-	-	-	-	0.01±0	-	-	-	-	-
α -cadinol	1515	RI, MS, AD	-	-	-	-	-	0.07±0.01	0.38±0.1	-	-	-	-
4-hydroxy-3-methoxystyrene	1317	RI, MS, AD	-	-	0.05±0.01	-	-	-	-	-	-	-	-
Total			3.56±0.15	31.69±2.22	0.05±0.03	3.5±0.09	33.08±2.65	5.1±0.15	2.19±0.94	12.47±2.32	31.78±1.23	30.31±1.98	20.19±0.54
Aldehydes (9)													
hexanal	818	RI, MS, AD	-	12.13±0.97	-	-	5.82±0.25	0.75±0.18	-	4.71±0.19	-	2.14±0.43	1.57±0.25
trans-2-octenal	1056	RI, MS, AD	-	-	-	-	-	-	-	-	-	-	0.61±0.1
heptanal	898	RI, MS, AD	-	1.09±0.04	-	0.67±0.08	1.08±0.31	-	-	-	-	-	-

Compounds	RI ^a	MI	Relative content (mean±SD)/% ^b										
			CH (31)	HY (18)	QD (22)	XY (32)	HJ (24)	FD (34)	MJ (20)	NH (20)	HR (16)	PH (18)	SH (23)
(E)-2-heptenal	951	RI, MS, AD	-	0.35±0.03	-	-	-	-	-	-	-	-	-
nonanal	1105	RI, MS, AD	0.19±0.01	1.94±0.11	0.41±0.11	0.92±0.07	2.4±0.35	0.59±0.18	0.12±0.03	1.2±0.3	0.85±0.2	0.34±0.01	0.59±0.03
decanal	1210	RI, MS, AD	0.16±0.03	0.66±0.05	0.21±0.05	0.52±0.04	1.33±0.52	0.21±0.09	0.09±0.02	0.51±0.11	0.56±0.42	0.22±0.09	0.6±0.04
β-cyclocitral	1224	RI, MS, AD	0.04±0	-	0.06±0.02	0.04±0.01	0.9±0.11	0.03±0.01	-	-	0.08±0.01	0.06±0.02	0.14±0.04
5-hydroxymethylfurfural	1236	RI, MS, AD	-	-	-	-	0.54±0.09	-	-	-	-	-	-
citral	1276	RI, MS, AD	0.05±0.01	-	-	-	-	-	-	-	-	-	0.29±0.02
Total			0.44±0.02	16.17±1.22	0.68±0.03	2.15±0.06	6.25±0.13	0.83±0.09	0.21±0.04	1.71±0.15	1.49±0.02	0.62±0.46	2.23±0.08
Ketones (6)													
3-octanone	981	RI, MS, AD	-	1.55±0.08	-	-	-	-	-	-	-	-	-
6-methylhept-5-en-2-one	983	RI, MS, AD	3.51±0.51	-	1.77±0.54	7.58±0.01	-	2.76±0.66	0.85±0.02	2.98±0.21	-	8.49±0.86	13.06±1.35
α-ionone	1423	RI, MS, AD	-	-	-	-	0.1±0.01	-	-	-	-	-	-
(E)-geranylacetone	1446	RI, MS, AD	-	-	-	-	0.32±0.01	-	-	-	-	-	-
β-ionone	1468	RI, MS, AD	-	-	-	-	0.71±0.02	-	-	-	-	-	-
nootkatone	1543	RI, MS, AD	0.31±0.08	-	-	1.29±0.04	0.12±0.06	0.11±0.01	-	-	-	-	-
Total			3.82±0.23	1.55±0.06	1.77±0.01	8.87±0.15	1.25±0.05	2.87±0.33	0.85±0.03	2.98±0.51	-	8.49±0.19	13.06±2.13
Esters (2)													
ethyl hexanoate	997	RI, MS, AD	0.02±0	-	-	-	0.29±0.07	-	-	-	-	-	-
ethyl caprylate	1203	RI, MS, AD	0.44±0.09	-	-	-	0.34±0.04	-	-	-	-	-	-
Total			0.46±0.31	-	-	-	0.63±0.06	-	-	-	-	-	-
Others (2)													
p-cymene	1019	RI, MS, AD	-	0.89±0.04	-	0.12±0.03	-	-	-	-	-	0.08±0.01	-
2-amylfuran	986	RI, MS, AD	-	2.09±0.1	-	-	2.78±0.52	-	-	-	-	-	-
Total			-	2.98±0.07	-	0.12±0.01	2.78±0.12	-	-	-	-	0.08±0.02	-

^a Identification method: MS, mass spectrum comparison using NIST and Wiley libraries; RI: retention index in agreement with literature value; AD: aroma description (odor). ^b SD: standard deviation. -: not detected.

3.2.2. Analysis of Aroma-active Substances by GC-MS-O

Seven aroma notes, namely “balsamic”, “pine”, “woody”, “citrus”, “grass”, “floral” and “mint”, were chosen by the sensory panel as the most representative characteristic odor attributes in eleven colorful pomelo samples in China after discussion and consensus (Table 5).

Significant odor of balsamic were found in all colorful pomelo juices, representative substances were myrcene in CH and QD, (E)-2-heptenal and 3-octanone in HY, 5-hydroxymethylfurfural in HJ, respectively. Pine aroma was also an important aroma of pomelo juices, induced by β-pinene in QD, XY and FD, and terpinolene in CH, HY, XY and FD.

As the most important odor, citrus flavor was attributed

by limonene, nonanal, decanal and octanol in most of colorful pomelo varieties. Nootkatone and β-cubebene also formed this flavor in some colorful pomelo samples such as CH, XY and HJ. Germacrene played an important role on the flavor of woody in CH, HY, QD, XY, HJ, FD and HR. In addition, this flavor was formed by δ-elemene, aromadendrene and γ-cadinene in CH, QD, XY, FD, etc.

While, the aroma-active substances that contributed to the odor of grassy were γ-murolene, β-caryophyllene oxide, hexanal, hexanol, heptanol, nonanol and (E)-geranylacetone. The mint flavor was smelled in varieties of CH, QD, XY, HJ, HR and PH because of β-cyclocitral. It is worth mentioning that special floral odor was smelled in most varieties except HY, XY and SH.

Table 5. Principal aroma-active compounds of different colorful pomelo juices

Compounds	Ordor ^a	DF ^b										
		CH	HY	QD	XY	HJ	FD	MJ	NH	HR	PH	SH
β-pinene	pine			7	8		5					
myrcene	balsamic, spice	6		7								
limonene	citrus, mint	8	8	8	8	8	8	8	8	8	8	8
terpinolene	pine	7	8		8		6					
δ-elemene	wood	7		8	8		8					
germacrene	wood	6	8	7	7	6	7			6		
α-gurjunene	wood, balsamic	2			5			8	7		8	
aromadendrene	wood	4		6	6		6	5	2			
isocaryophyllene	wood				8	4	3	5				
β-cubebene	citrus, fruity	8	6	8	7		2	6	3	3		
α-caryophyllene	wood, grassy, pine	5		3	5		6					
γ-murolene	grassy, spice	5	2	2	4	3	4	2				
γ-cadinene	wood	5		3	4	2	3	5				
β-caryophyllene oxide	grassy, sweet, spice	7			8				6	7	7	
hexanal	grassy, balsamic		8			7	7	8	6	7		
heptanal	balsamic, citrus		6		5	6		6				
(E)-2-heptenal	balsamic, almod		8									
nonanal	balsamic, citrus, grassy	6	8	5	6	7	3		4			
decanal	citrus, balsamic	6	7	8	8	8	3	7	2	2		
β-cyclocitral	mint	4		4	5	6			3	2		
5-hydroxymethylfurfural	balsamic, spice, pine					6	5	6				
citral	lemon	7										8
hexanol	pine, floral, grassy	8	8		8	8			3	2	2	
heptanol	grassy		6		2	3	7					
octanol	pine, citrus, rose		7		7	8	2		4			
(E)-linalool	floral	5										
nonanol	balsamic, grassy		7			8	5		7		6	
4-hydroxy-3-methoxystyrene	clove, spice			7								
3-octanone	grassy, balsamic, pine		7									
6-methylhept-5-en-2-one	spice, mushroom, pine	4		2	5		5			5	7	
α-ionone	wood, violet					6						
(E)-geranylacetone	magnolia, grassy					8						
β-ionone	seaweed, violet					5						
nootkatone	grapefruity	8			8	8	7					
ethyl hexanoate	apple, balsamic	4				6	3	2	3		5	
styrene	balsamic, pine			7	4	3	7					
p-cymene	pine, citrus		8		7							
2-amylfuran	green bean, balsamic		5			5						

^a Odor description obtained from <http://www.flavournet.org>. ^b Sum of times determined by the panel of assessors.

3.2.3. PLS-DA of Aroma-Active Components

As a versatile algorithm that can be used for predictive and descriptive modelling as well as for discriminative variable selection, PLS-DA was applied to build linear discriminant models according to their volatile compositions attributes. As given in Figure 6, each point represented a variety and different colors represented different origins. According to the score plot, different varieties and origins of colorful pomelo were clearly distinguished. The location of eleven different colorful pomelo varieties from four main origins as explanatory variables and 37 aroma-active components (Table 5) as dependent variables has given a good linear correlated model.

It is interesting that the distribution of these four main production areas is similar to the geographical distribution

of the four provinces in China. Chongqing province is located in the southwest China and is not adjacent to the other three provinces which is situated in the southeast China, pictured here. Jiangxi province, like Chongqing province, belongs to the inland region, while both province of Zhejiang and Fujian province are adjacent and belongs to coastal areas. As given in Figure 6, the colorful pomelo varieties produced in Zhejiang and Fujian have a relatively closer location perhaps because their closer geographical situation. The confidence ellipses can display the degree of clustering of varieties within the same production area, helping to determine the degree of overlap between categories and thus understanding the model's discriminative ability. It can be seen that all the five varieties in Jiangxi province have a good clustering in Figure 6.

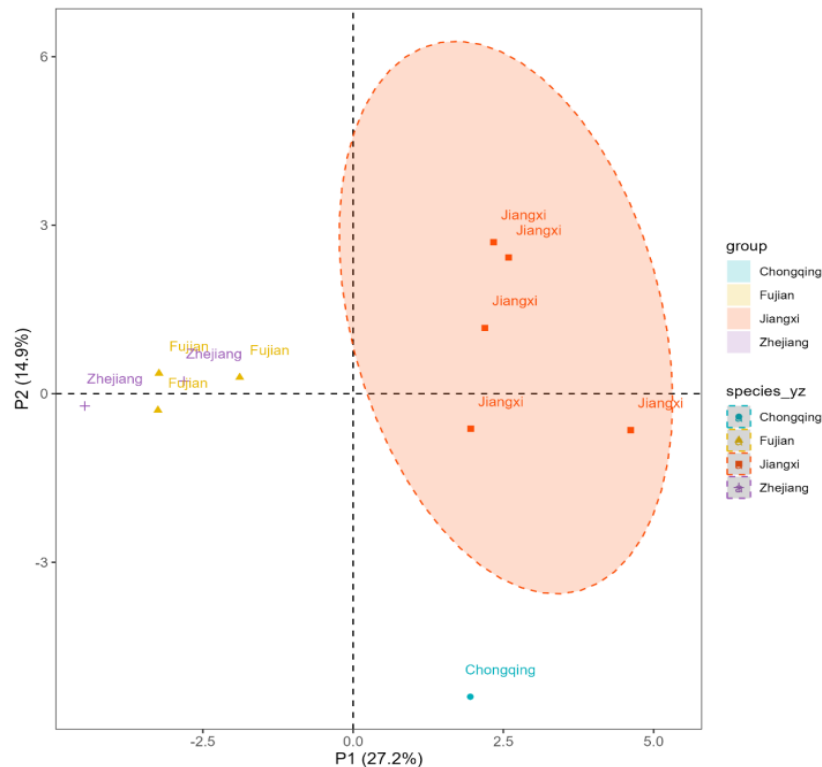


Figure 6. PLS-DA biplots of aroma-active compounds of colorful pomelo juices correlating with origins given as a two-dimensional representation of the scores

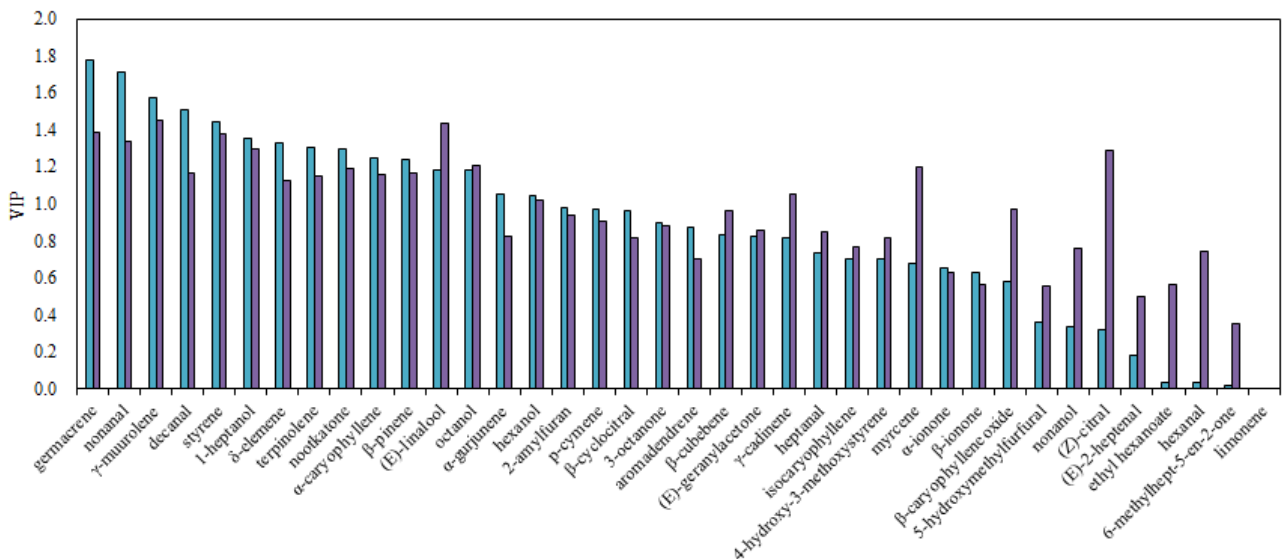


Figure 7. Aroma-active components of different colorful pomelo juices ordered by variable importance criterion VIP

Note that the score plot of VIP given in the Figure 7 corresponded to the model with selected variables. As a rule of thumb, it is customary to retain variables with $VIP > 1$, all the distinctive aroma-active component is considered as different varieties [33]. Based on this rule, variables with both VIP1 and VIP2 were greater than 1 were selected as markers. The distinctive aroma-active components that can distinguish the four main origins of pomelo were germacrene, nonanal, γ -muurolene, decanal, styrene, heptanol, δ -elemene, terpinolene, nootkatone, α -caryophyllene, β -pinene, (E)-linalool, octanol, α -gurjunene and hexanol. Actually, the differences in the odor of wood, citrus, grassy, balsamic, pine and floral among different varieties can achieve the differentiation

and geographical identification. Hence, it is feasible to achieve the discrimination of different colorful pomelo varieties by multivariate statistical analysis.

4. Conclusion

There were differences in the indicators of total sugar, TA, total phenols, color and volatile substances among the eleven colorful pomelo varieties in China, no significant difference existed in the level of glucose and fructose among all the varieties.

Olefins, aldehydes, alcohols, ketones and esters were the main volatiles in the eleven colorful pomelo varieties,

of which olefins were the predominant volatile components. 38-active aroma components and seven aroma notes were detected, such as “balsamic”, “pine”, “woody”, “citrus”, “grass”, etc. The PLS-DA results showed that the four main origins of eleven colorful pomelo varieties from China could be well distinguished.

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References

- [1] Mun Wai Cheong, Shao Quan Liu, Weibiao Zhou, Philip Curran, Bin Yu, Chemical composition and sensory profile of pomelo (*Citrus grandis* (L.) Osbeck) juice, *Food Chemistry*, 2012, 135 (4), 2505-2513.
- [2] Azevedo, M. S.; Tischer Seraglio, S. K.; Rocha, G.; Balderas, C. B.; Piovezan, M.; Gonzaga, L. V.; Falkenberg, D. d. B.; Fett, R.; Leal de Oliveira, M. A.; Oliveira Costa, A. C., Free amino acid determination by GC-MS combined with a chemometric approach for geographical classification of bracinga honeydew honey (*Mimosa scabrella* Benth). *Food Control*, 2017, 78, 383-392.
- [3] Raymond, C. A.; Davies, N. W.; Larkman, T., GC-MS method validation and levels of methyl eugenol in a diverse range of tea tree (*Melaleuca alternifolia*) oils. *Analytical and Bioanalytical Chemistry*, 2017, 409, 1779-1787.
- [4] Hemalatha, R.; Nivetha, P.; Mohanapriya, C.; Sharmila, G.; Muthukumar, C.; Gopinath, M., Phytochemical composition, GC-MS analysis, in vitro antioxidant and antibacterial potential of clove flower bud (*Eugenia caryophyllus*) methanolic extract. *Journal of Food Science and Technology-mysore*, 2016, 53, 1189-1198.
- [5] Qin, G.; Tao, S.; Cao, Y.; Wu, J.; Zhang, H.; Huang, W.; Zhang, S., Evaluation of the volatile profile of 33 *Pyrus ussuriensis* cultivars by HS-SPME with GC-MS. *Food Chemistry*, 2012, 134, 2367-2382.
- [6] Xiao, L.; Lee, J.; Zhang, G.; Ebeler, S. E.; Wickramasinghe, N.; Seiber, J. N.; Mitchell, A. E., HS-SPME GC/MS characterization of volatiles in raw and dry-roasted almonds (*Prunus dulcis*). *Food Chemistry*, 2014, 151, 31-39.
- [7] Riuaumatell, M.; Miro, P.; Serracayuela, A.; Buxaderas, S.; Lopezmames, E., Assessment of the aroma profiles of low-alcohol beers using HS-SPME-GC-MS. *Food Research International*, 2014, 57, 196-202.
- [8] Shin, E.; Craft, B. D.; Pegg, R. B.; Phillips, R. D.; Eitenmiller, R. R., Chemometric approach to fatty acid profiles in Runner-type peanut cultivars by principal component analysis (PCA). *Food Chemistry*, 2010, 119, 1262-1270.
- [9] Jon G Wilkes, Eric D Conte, Yongkyoung Kim, Manuel Holcomb, John B Sutherland, Dwight W Miller, Sample preparation for the analysis of flavors and off-flavors in foods, *Journal of Chromatography A*, 2000, 880(1-2), 3-33.
- [10] Simona Gherghel, Ruth M. Morgan, Javier Arrebola-Liébanas, Roberto Romero-González, Chris S. Blackman, Antonia Garrido-Frenich, Ivan P. Parkin, Development of a HS-SPME/GC-MS method for the analysis of volatile organic compounds from fabrics for forensic reconstruction applications, *Forensic Science International*, 2018, 290, 207-218.
- [11] Minwei Xu, Zhao Jin, Yang Lan, Jiajia Rao, Bingcan Chen, HS-SPME-GC-MS/olfactometry combined with chemometrics to assess the impact of germination on flavor attributes of chickpea, lentil, and yellow pea flours, *Food Chemistry*, 2019, 280, 83-95.
- [12] Mónica Bueno, Virginia C. Resconi, M. Mar Campo, Vicente Ferreira, Ana Escudero, Development of a robust HS-SPME-GC-MS method for the analysis of solid food samples. Analysis of volatile compounds in fresh raw beef of differing lipid oxidation degrees, *Food Chemistry*, 2019, 281, 49-56.
- [13] S. Yogeswari, S. Ramalakshmi, R. Neelavathy and J. Muthumary, Identification and Comparative Studies of Different Volatile Fractions from *Monochaetia kansensis* by GCMS, *Global Journal of Pharmacology*, 2012, 6(2): 65-71.
- [14] Fengling Tan, Peng Wang, Ping Zhan, Honglei Tian, Characterization of key aroma compounds in flat peach juice based on gas chromatography-mass spectrometry-olfactometry (GC-MS-O), odor activity value (OAV), aroma recombination, and omission experiments, *Food Chemistry*, 2022, 366.
- [15] Xueli Pang, Xingfeng Guo, Zihan Qin, Yubo Yao, Xiaosong Hu, and Jihong Wu. Identification of Aroma-Active Compounds in Jiashi Muskmelon Juice by GC-O-MS and OAV Calculation, *J. Agric. Food Chem.* 2012, 60(17), 4179-4185.
- [16] Serkan Selli, Gamze Guclu, Onur Sevidik, Hasim Kelebek, Variations in the key aroma and phenolic compounds of champignon (*Agaricus bisporus*) and oyster (*Pleurotus ostreatus*) mushrooms after two cooking treatments as elucidated by GC-MS-O and LC-DAD-ESI-MS/MS, *Food Chemistry*, 2021, 354.
- [17] Sylvie Chevallier, Dominique Bertrand, Achim Kohler, Philippe Courcoux, Application of PLS-DA in multivariate image analysis, *Journal of Chemometrics*, 2006, 20(5): 221-229.
- [18] O. Galtier, O. Abbas, Y. Le Dréau, C. Rebufa, J. Kister, J. Artaud, N. Dupuy, Comparison of PLS1-DA, PLS2-DA and SIMCA for classification by origin of crude petroleum oils by MIR and virgin olive oils by NIR for different spectral regions, *Vibrational Spectroscopy*, 2011, 55(1): 132-140.
- [19] Ge Gao, Liang Zhao, Yan Ma, Yongtao Wang, Zhijian Sun, Xiaojun Liao. Microorganisms and Some Quality of Red Grapefruit Juice Affected by High Pressure Processing and High Temperature Short Time. *Food Bioprocess Technol.*, 2015, 8, 2096-2108.
- [20] Gao Ge, Pang Xueli, Liu Haihua, Zou Hui, Liao Xiaojun. Volatiles Identification of Pomelo Based on GC-MS-O and Multivariate Statistical Analysis[J]. *Journal of Chinese Institute of Food Science and Technology*, 2020, 20(5), 283-292.
- [21] Singleton, V. L.; Orthofer, R.; Lamuelaraventos, R. M., Analysis of Total Phenols and Other Oxidation Substrates and Antioxidants by Means of Folin-Ciocalteu Reagent. *Methods in Enzymology*, 1999, 299, 152-178.
- [22] Cao, X.; Bi, X.; Huang, W.; Wu, J.; Hu, X.; Liao, X., Changes of quality of high hydrostatic pressure processed cloudy and clear strawberry juices during storage. *Innovative Food Science and Emerging Technologies*, 2012, 16, 181-190.
- [23] Luo, D.; Chen, J.; Gao, L.; Liu, Y.; Wu, J., Geographical origin identification and quality control of Chinese chrysanthemum flower teas using gas chromatography-mass spectrometry and olfactometry and electronic nose combined with principal component analysis. *International Journal of Food Science and Technology*, 2017, 52, 714-723.
- [24] Pang, X.; Chen, D.; Hu, X.; Zhang, Y.; Wu, J., Verification of Aroma Profiles of Jiashi Muskmelon Juice Characterized by Odor Activity Value and Gas Chromatography-Olfactometry/Detection Frequency Analysis: Aroma Reconstitution Experiments and Omission Tests. *Journal of Agricultural and Food Chemistry* 2012, 60, 10426-10432.
- [25] Xiao Zhang. The effect of ultra-high pressure on the processing quality of grapefruit juice. China Agricultural University. 2014.
- [26] R. M.; Yanes, O., Mass spectral databases for LC/MS- and GC/MS-based metabolomics: State of the field and future prospects. *Trends in Analytical Chemistry*, 2016, 78, 23-35.
- [27] Ulf Örtengren, Fredrik Andersson, Ulrika Elgh, Björn Terselius, Stig Karlsson, Influence of pH and storage time on the sorption and solubility behaviour of three composite resin materials, *Journal of Dentistry*, 2001, 29(1), 35-41.
- [28] Kristina L. Penniston, Stephen Y. Nakada, Ross P. Holmes, Dean G, Quantitative Assessment of Citric Acid in Lemon Juice, Lime Juice, and Commercially-Available Fruit Juice Products Assimos, *Journal of Endourology*, 2008, 22(3).
- [29] Zhenyu Huang, Huiyao Hu, Fei Shen, Bei Wu, Xuanxuan Wang, Baoguo Zhang, Wuqian Wang, Li Liu, Jing Liu, Chijie Chen, Rui Zhang, Ruiting Chen, Yi Wang, Ting Wu, Xuefeng Xu, Zhenhai Han, Xinzhang Zhang, Relatively high acidity is an important breeding objective for fresh juice-specific apple cultivars, *Scientia Horticulturae*, 2018, 233, 29-37.
- [30] J.A. Hernández-Herrero, M.J. Frutos, Influence of rutin and

ascorbic acid in colour, plum anthocyanins and antioxidant capacity stability in model juices, *Food Chemistry*, 2015, 173, 495-500.

- [31] Edwin Vera Calle, Jenny Ruales, Manuel Dornier, Jacqueline Sandeaux, Roger Sandeaux, Gérald Pourcelly, Deacidification of the clarified passion fruit juice (*P. edulis f. flavicarpa*), *Desalination*, 2002, 149(1-3), 357-361.
- [32] Shahnawaz Ahmed, H S Rattanpal, Khalid Gul, Rouf Ahmad Dar, Akash Sharma, Chemical composition, antioxidant activity and GC-MS analysis of juice and peel oil of grapefruit varieties cultivated in India, *Journal of Integrative Agriculture*, 2019, 18(7), 1634-1642.
- [33] Hervé, Partial Least Square Regression PLS-Regression. 2007.



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