

葡萄汁中糖酸组成分析及在掺假鉴别中的应用

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摘 要:【目的】研究葡萄汁的糖酸组成特征及品种和采收成熟度的影响,探索其在葡萄汁掺假鉴别中的应用效果。【方法】采用高效液相色谱法对17个品种共51个葡萄汁样品中可溶性糖和有机酸组成进行分析,结合化学计量学方法探索其在葡萄汁掺假鉴别中的应用。【结果】葡萄汁中可溶性糖主要由葡萄糖和果糖组成,二者之和占总糖含量的97.88%~99.86%,而且二者的比值在0.81~1.05。有机酸主要是酒石酸、柠檬酸和苹果酸,分别占总酸含量的57.95%~92.37%、5.26%~36.11%和2.08%~6.67%。不同品种葡萄汁中各种可溶性糖和有机酸的含量存在较大差异,但其基本组成特征具有较好一致性。通过分析葡萄原汁及其掺假样品中可溶性糖和有机酸组成,结合主成分分析和线性判别分析,可以实现对葡萄汁与掺加梨汁、苹果汁、杏汁和桃汁等掺假样品的区分。【结论】葡萄汁糖酸组成可用于掺假鉴定和质量控制,综合应用可溶性糖和有机酸组成的鉴别效果优于单一利用可溶性糖或有机酸组成。

关键词:葡萄;果汁;可溶性糖;有机酸;掺假鉴定

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Profiles of soluble sugars and organic acids in grape juice and their application for authentication

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Abstract: 【Objective】Soluble sugars and organic acids are the main nutrients and taste components in fruit juices, which show a lower susceptibility to changes as compared with other components such as pigments, antioxidants, and flavour compounds. Therefore, characterization of the composition of sugars and/or organic acids in fruit juices is considered as a desirable approach for fruit juice authenticity as well as quality control. The composition and content of sugars and organic acids of grape juice may be influenced by variety, harvest date and cultivation conditions. This work aims to find the common pattern of grape juices and their implication for authentication by comparing the sugar and organic acid profiles of grape juices prepared from different varieties and harvest dates. 【Methods】Grape berries of 17 varieties, including ‘Ruby seedless’ ‘Red globe’ ‘Gold finger’ ‘Jingxiu’ ‘Kyoho’ ‘Jumeigui’ ‘Moldova’ ‘Niagara’ ‘Centennial seedless’ ‘Victoria’ ‘Summer black’ ‘Xiazhihong’ ‘Xiuyu’ ‘Shine-muscat’ ‘Flame’ ‘Zexiang’ ‘Zuijinxiang’ were harvested at mature stage for three times and washed, and then pureed using a lab-scale food processor. After pasteurization in boiling water for 5 min, the puree was cooled and treated with pectinase at 50 °C for 40 min, and then centrifuged at 4 000 r · min⁻¹ for 10 min. The resulted supernatant was recovered and stored at -80 °C before high performance liquid chromatography (HPLC) analysis for sugars and organic acids. For assessment of the possibility of application of sugar and organic acid profiles for grape juice authentication and quality control, adulterate samples were prepared by adding 20%, 40%, 60%, 80% and 100% of apple juice, pear

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juice, apricot juice or peach juice and the composition of sugars and organic acids was determined by HPLC. Principal component analysis (PCA) and linear discriminant analysis (LDA) were used to discriminate grape juices from their adulterate samples with apple, pear, apricot or peach juice. **【Results】** The soluble sugars in grape juices mainly consisted of glucose, fructose and sucrose, while tartaric acid, citric acid and malic acid were the main organic acids in grape juices. There were great differences in contents of individual soluble sugars and organic acids among grape juices of different varieties and harvest dates, with a coefficient of variation of 135.86%, 9.89%, 11.46%, 16.93%, 41.13%, 34.89%, 76.65% and 64.82% for sucrose, glucose, fructose, tartaric acid, malic acid, citric acid, shikimic acid, and fumaric acid, respectively. Glucose and fructose contributed 97.88% to 99.86% of the total content of identified soluble sugars, with a ratio in a range of 0.81-1.05. Tartaric acid was the predominant organic acid in grape juices of all varieties and harvest dates, with a proportion of 57.95% to 92.37% in total content of identified organic acids. Malic acid and citric acid accounted for 5.26% to 36.11% and 2.08% to 6.67%, respectively. By determining the compositions of soluble sugars and/or organic acids coupled with principal component analysis (PCA) and linear discriminant analysis (LDA), grape juices could be discriminated from their adulterates with apple juice, pear juice, peach juice, and apricot juice. The discriminant effects depended on the ratios of adulterations and dataset used for PCA and LDA. When the soluble sugar composition was used as analytical data for PCA, the adulterate samples could be distinguished from grape juices except juice sample adulterated with 20% of apple juice. The juice sample adulterated with 20% of apricot juice was also not discriminated from grape juices by using analytical data of organic acid content for PCA. However, when combining the soluble sugar and organic acid composition as analytical dataset for PCA, all the adulterate samples could be distinguished from grape juices. As for LDA, the combination of soluble sugar and organic acid composition also showed a better discriminant effect than soluble sugar or organic acid composition alone, with a correct classification rate of 100% for the original and 95.8% for the cross validation method, while they were 95.8% and 93.0%, respectively, when only soluble sugar composition was used as analytical dataset for LDA. As for organic acid composition, the correct classification rates were only 90.1% for the original and 83.1% for the cross validation method, suggesting an even worse discriminant effect than soluble sugar. **【Conclusion】** The profiles of soluble sugars and organic acids in grape juice may be used for the authentication and quality control of grape juice. By using soluble sugar and organic acid composition coupled with principal component analysis and linear discriminant analysis, the samples adulterated with apple juice, pear juice, peach juice and apricot juice could be discriminated from grape juices. The combination of sugar and organic acid composition for principal component analysis and linear discriminant analysis was superior to sugar or organic acid composition alone.

Key words: Grape; Juice; Soluble sugar; Organic acid; Authentication

葡萄汁是葡萄最重要的加工产品之一,富含糖、有机酸、维生素、氨基酸、矿质元素等营养物质和膳食纤维、花色苷、酚酸、黄酮类、白藜芦醇等功能性成分,长期饮用具有抗氧化、抗衰老、抗糖尿病、抗肥胖、调节血脂、降血压、改善记忆力、保护生殖系统、预防心脑血管疾病和神经退行性疾病等功效^[1-8],因此常被作为保健饮品。然而,受经济利益驱动,一些不法商人可能会通过掺水、添加廉价果汁甚至采用

各种添加剂勾兑等方式来降低生产成本以获取高额利润,严重扰乱葡萄汁饮料正常生产秩序,损害消费者的利益和身体健康。为了识别葡萄汁的掺假行为,谭梦茹等^[9]通过分析葡萄汁产品的碳同位素比值来检测碳-4来源糖浆和有机酸的掺假,Miaw等^[10]利用衰减全反射傅里叶变换红外光谱(attenuated total reflectance Fourier-transform mid-infrared spectroscopy, ATR-FTIR)结合多元分类法判别葡萄汁中

是否掺假苹果汁。通过测定葡萄汁产品中的根皮苷和山梨醇含量也可鉴别掺假苹果汁的行为^[11]。这些研究主要针对特定的掺假行为,应用范围有限,对于愈来愈复杂、多样的掺假行为,迫切需要建立识别范围广、结果可靠的鉴别方法。

可溶性糖和有机酸在果汁中含量高,分析手段简单,而且在果汁加工、贮藏过程中相对比较稳定^[12-13],因此利用果汁的可溶性糖和有机酸组成特征进行掺假鉴别具有独特的优势。Navarro-Pascual-Ahuir等^[14-15]根据不同种类果汁中的可溶性糖或有机酸组成,结合线性判别分析(linear discriminant analysis, LDA),实现了对苹果、葡萄、柑橘、橙、菠萝等5种果汁以及掺入50%葡萄汁的橙汁、菠萝汁的区分。苏光明等^[16]利用高效液相色谱分析苹果原汁和添加水、苹果酸、蔗糖的模拟掺假苹果汁中有机酸的

组成与含量,结合二元逻辑回归分析方法建立鉴别模型,成功实现掺假苹果汁的鉴别。本研究采用高效液相色谱法对不同品种及采收批次葡萄果汁中的可溶性糖和有机酸组成与含量进行分析,以了解葡萄汁的糖酸组成特征及品种和采收成熟度的影响,在此基础上通过对模拟掺假葡萄汁的糖酸组成分析结合化学计量学方法探索其在葡萄汁掺假鉴别中的应用效果,以期对葡萄汁产品的质量控制在提供参考。

1 材料和方法

1.1 材料与试剂

葡萄果实于2016年7月至9月采自中国农业科学院郑州果树研究所葡萄种质资源圃,每个品种在成熟期分3次采样并制汁,供试葡萄品种及其果汁样品编号见表1。

表 1 供试葡萄果汁样品及编号

Table 1 Samples and numbers of tested grape juice

编号 Number	采样批次 Harvest batch	品种 Variety	编号 Number	采样批次 Harvest batch	品种 Variety	编号 Number	采样批次 Harvest batch	品种 Variety
PT1	1	红宝石无核 Ruby seedless	PT19	1	摩尔多瓦 Moldova	PT37	1	秀玉 Xiuyu
PT2	2		PT20	2		PT38	2	
PT3	3		PT21	3		PT39	3	
PT4	1	红地球 Red globe	PT22	1	尼加拉 Niagara	PT40	1	阳光玫瑰 Shine-muscat
PT5	2		PT23	2		PT41	2	
PT6	3		PT24	3		PT42	3	
PT7	1	金手指 Gold finger	PT25	1	森田尼无核 Centennial seedless	PT43	1	早熟红 Flame
PT8	2		PT26	2		PT44	2	
PT9	3		PT27	3		PT45	3	
PT10	1	京秀 Jingxiu	PT28	1	维多利亚 Victoria	PT46	1	泽香 Zexiang
PT11	2		PT29	2		PT47	2	
PT12	3		PT30	3		PT48	3	
PT13	1	巨峰 Kyoho	PT31	1	夏黑 Summer black	PT49	1	醉金香 Zuijinxinang
PT14	2		PT32	2		PT50	2	
PT15	3		PT33	3		PT51	3	
PT16	1	巨玫瑰 Jumeigui	PT34	1	夏至红 Xiazhihong			
PT17	2		PT35	2				
PT18	3		PT36	3				

棉籽糖、蔗糖、葡萄糖、果糖、山梨醇、草酸、酒石酸、奎宁酸、苹果酸、莽草酸、乳酸、柠檬酸、富马酸标准品 美国 Sigma 公司; EDTA 二钠钙(分析纯) 美国 Aladdin 公司; 磷酸(分析纯) 天津市致远化学试剂有限公司; 磷酸氢二铵(分析纯) 天津市科密欧化学试剂有限公司。

1.2 主要仪器与设备

1525 高效液相色谱仪(配有 2707 自动进样器、

2414 示差折光检测器、2998 二极管阵列检测器) 美国 Waters 公司; 九阳料理机 九阳股份有限公司; 砂芯过滤装置 津腾实验设备有限公司。

1.3 方法

1.3.1 葡萄汁制备 按照以下工艺制备葡萄汁: 葡萄果实→去梗→破碎→榨汁→灭酶(沸水浴, 5 min)→冷却→加果胶酶酶解(0.1%, 50 °C, 40 min)→冷却→离心→过滤→葡萄汁。

制得的葡萄汁贮存于-80℃超低温冰箱,分析前取出2 mL加水定容至10 mL,用0.22 μm微孔滤膜过滤。

1.3.2 模拟掺假 葡萄汁中分别掺加20%、40%、60%、80%、100%的梨汁、苹果汁、桃汁和杏汁作为伪品,样品编号见表2。

表2 供试掺假样品及编号

Table 2 Numbers of tested adulterate samples

编号 Number	梨汁掺加量 Percent of pear juice/%	编号 Number	苹果汁掺加量 Percent of apple juice/%	编号 Number	杏汁掺加量 Percent of apricot juice/%	编号 Number	桃汁掺加量 Percent of peach juice/%
LW1	20	PW1	20	XW1	20	TW1	20
LW2	40	PW2	40	XW2	40	TW2	40
LW3	60	PW3	60	XW3	60	TW3	60
LW4	80	PW4	80	XW4	80	TW4	80
LW5	100	PW5	100	XW5	100	TW5	100

1.3.3 可溶性糖组成分析 HPLC法,以棉籽糖、蔗糖、葡萄糖、果糖、山梨醇为标准品采用外标法测定葡萄汁中可溶性糖的组成与含量。色谱条件为:Waters Sugar-pak1(6.5×300 mm)色谱柱,流动相50 mg·L⁻¹ EDTA二钠钙溶液,流速0.5 mL·min⁻¹,柱温80℃,样品池温度30℃,进样体积10 μL。

1.3.4 有机酸组成分析 HPLC法^[17],以草酸、酒石酸、奎宁酸、苹果酸、莽草酸、乳酸、柠檬酸、富马酸为标准品采用外标法测定葡萄汁中有机酸的组成与含量。色谱条件为:Ultimate® AQ-C18(4.6 mm×250 mm,5 μm)色谱柱,流动相为0.02 mol·L⁻¹磷酸氢二铵溶液(磷酸调pH值为2.4),流速1.0 mL·min⁻¹,柱温30℃,进样体积10 μL,检测波长210 nm。

1.3.5 数据处理 每一试验均进行3次重复,采用SPSS 22.0软件对数据进行统计分析。

2 结果与分析

2.1 不同品种及采收批次葡萄果汁中可溶性糖的组成与含量

葡萄果汁中的可溶性糖主要由葡萄糖和果糖组成,蔗糖含量很少,棉籽糖和山梨醇均未检出。由表3可见,不同品种以及采收批次的葡萄制成的果汁中蔗糖、葡萄糖、果糖、总可溶性糖含量及葡萄糖与果糖的比值均存在较大差异,变异系数分别为135.86%、9.89%、11.46%、10.05%和6.82%,说明原料品种和采收成熟度对葡萄果汁中可溶性糖的组成与含量存在较大影响。在供试的17个品种共51个葡萄果汁样品中,葡萄糖与果糖均为最主要的可溶性糖,二者之和占检测到的总可溶性糖含量的97.88%~99.86%,而且葡萄糖与果糖的含量相当,二者的比值在0.81~1.05。随着采收成熟度的增加,大部分品

种葡萄果汁中葡萄糖、果糖、蔗糖含量呈升高趋势,说明延迟采收可提高葡萄果汁中可溶性糖含量。但对于同一葡萄品种,采收批次对其果汁中各种可溶性糖含量占总可溶性糖的比例以及相互之间的比值影响不大,说明葡萄果汁中各种可溶性糖之间的比值主要受原料品种影响,采收成熟度主要影响可溶性糖的含量。

2.2 不同品种及采收批次葡萄果汁中有机酸的组成与含量

葡萄果汁中的有机酸主要由酒石酸、苹果酸、柠檬酸和少量的莽草酸、富马酸组成,草酸、奎宁酸、乳酸均未检出。由表4可见,不同品种及采收批次葡萄果汁中酒石酸、苹果酸、柠檬酸、莽草酸和富马酸含量均存在较大差异,其变异系数分别为16.93%、41.13%、34.89%、76.65%和64.82%,高于葡萄糖和果糖,说明葡萄果汁中有机酸的组成与含量受原料的影响较可溶性糖大。在供试的17个品种共51个葡萄果汁样品中,酒石酸均是最主要的有机酸,质量浓度在4 721.77~9 670.75 mg·L⁻¹,占检测到的总有机酸含量的57.95%~92.37%,其次为苹果酸,占5.26%~36.11%,柠檬酸占2.08%~6.67%,莽草酸与富马酸仅占0.05%~0.31%。李佳秀等^[17]研究表明,苹果汁、梨汁、桃汁、樱桃汁、蓝莓汁、石榴汁等果汁中均未检出酒石酸,樱桃果汁中有机酸组成以苹果酸为主,苹果汁、梨汁、桃汁、蓝莓汁中含有奎宁酸,石榴汁含有草酸,表现出与葡萄汁不同的有机酸组成特征,因此可以利用有机酸组成来辨别葡萄汁与其他果汁以及在其他果汁中添加葡萄汁或者在葡萄果汁中添加其他果汁的掺假行为。

2.3 掺假鉴别

2.3.1 主成分分析 按照1.3.2方法制备模拟掺假

表 3 不同品种及采收批次葡萄果汁中可溶性糖的组成与含量

Table 3 Composition and content of soluble sugars in grape juices of different varieties and harvest batches (g·L⁻¹)

品种 Variety	采样批次 Harvest batch	蔗糖 Sucrose	葡萄糖 Glucose	果糖 Fructose	总可溶性糖 Total sugar	葡萄糖/果糖 Glu/Fru
红宝石无核 Ruby seedless	1	0.26±0.00	85.29±0.39	94.39±0.66	179.94±1.05	0.90±0.00
	2	0.27±0.00	85.69±1.35	96.21±1.46	182.17±2.81	0.89±0.00
	3	0.28±0.00	86.05±0.57	95.89±0.61	182.21±1.18	0.90±0.00
红地球 Red globe	1	0.27±0.00	72.30±0.75	75.08±0.78	147.64±1.52	0.96±0.00
	2	0.30±0.00	79.65±0.11	84.97±0.16	164.93±0.26	0.94±0.00
	3	0.35±0.00	86.17±0.14	90.48±0.13	176.99±0.27	0.95±0.00
金手指 Gold finger	1	0.49±0.00	73.25±0.53	85.00±0.61	158.74±1.15	0.86±0.00
	2	0.57±0.00	77.78±0.63	90.36±0.82	168.70±1.44	0.86±0.00
	3	0.69±0.00	90.60±0.36	105.05±0.43	196.34±0.79	0.86±0.00
京秀 Jingxiu	1	0.29±0.00	81.19±0.38	90.30±0.43	171.78±0.81	1.03±0.00
	2	0.30±0.00	82.38±0.24	92.86±0.27	175.54±0.52	1.04±0.00
	3	0.34±0.00	90.22±0.34	100.60±0.39	191.16±0.73	1.02±0.00
巨峰 Kyoho	1	0.40±0.00	98.42±0.39	102.28±0.43	201.10±0.81	0.93±0.00
	2	0.41±0.00	102.27±1.03	107.45±1.02	210.13±2.05	0.95±0.00
	3	0.46±0.01	105.90±0.48	112.08±0.52	218.44±1.01	0.94±0.00
巨玫瑰 Jumeigui	1	0.37±0.00	93.22±0.36	90.73±0.34	184.31±0.71	1.03±0.00
	2	0.47±0.00	95.74±0.58	94.97±0.57	191.18±1.15	1.01±0.00
	3	0.55±0.01	106.11±0.34	103.41±0.33	210.08±0.67	1.03±0.00
摩尔多瓦 Moldova	1	0.22±0.00	88.15±0.13	85.30±0.17	173.67±0.30	0.90±0.00
	2	0.19±0.00	87.96±1.35	84.41±1.23	172.56±2.57	0.89±0.00
	3	0.20±0.00	86.00±0.46	83.60±0.42	169.79±0.87	0.90±0.00
尼加拉 Niagara	1	2.20±0.03	70.97±0.50	77.36±0.61	150.53±1.14	0.92±0.00
	2	3.67±0.03	81.68±0.80	87.66±0.85	173.01±1.69	0.93±0.00
	3	3.43±0.01	84.14±1.94	91.19±0.35	178.76±1.58	0.92±0.02
森田尼无核 Centennial seedless	1	0.27±0.00	82.61±0.36	102.44±0.44	185.32±0.80	0.81±0.00
	2	0.26±0.00	82.41±0.76	99.03±0.94	181.70±1.70	0.83±0.00
	3	0.28±0.00	87.12±0.30	103.52±0.35	190.93±0.66	0.84±0.00
维多利亚 Victoria	1	0.30±0.00	82.92±0.60	84.00±0.60	167.22±1.20	0.99±0.00
	2	0.32±0.00	80.27±0.51	82.81±0.53	163.41±1.04	0.97±0.00
	3	0.33±0.00	87.50±1.03	91.61±0.44	179.44±1.11	0.96±0.01
夏黑 Summer black	1	0.30±0.00	78.00±0.56	75.64±0.55	153.94±1.10	1.03±0.00
	2	0.32±0.00	78.66±1.06	76.42±1.00	155.40±2.06	1.03±0.00
	3	0.32±0.00	77.58±0.69	77.15±0.70	155.06±1.40	1.01±0.00
夏至红 Xiazhihong	1	0.24±0.00	73.65±0.27	69.99±0.27	143.87±0.54	1.05±0.00
	2	0.25±0.00	75.27±1.32	72.21±1.14	147.73±2.47	1.04±0.00
	3	0.25±0.00	75.91±0.36	74.06±0.25	150.22±0.61	1.03±0.00
秀玉 Xiuyu	1	0.32±0.00	89.09±0.71	89.29±0.73	178.71±1.44	1.00±0.00
	2	0.38±0.00	92.11±0.24	92.92±0.16	185.41±0.38	0.99±0.00
	3	0.41±0.00	97.26±0.47	98.20±0.48	195.87±0.94	0.99±0.00
阳光玫瑰 Shine-muscat	1	0.34±0.00	93.79±0.21	103.46±0.26	197.59±0.46	0.91±0.00
	2	0.34±0.00	98.82±1.33	110.50±1.50	209.66±2.84	0.89±0.00
	3	0.32±0.00	91.05±0.10	104.50±0.14	195.87±0.23	0.87±0.00
早熟红 Flame	1	0.27±0.00	78.79±0.81	82.44±0.89	161.50±1.70	0.96±0.00
	2	0.31±0.00	90.52±1.46	94.98±1.47	185.81±2.93	0.95±0.00
	3	0.36±0.00	98.07±1.60	103.93±1.63	202.36±3.23	0.94±0.00
泽香 Zexiang	1	0.37±0.00	83.12±0.06	84.43±0.05	167.93±0.12	0.98±0.00
	2	0.37±0.00	87.98±0.18	88.99±0.19	177.33±0.37	0.99±0.00
	3	0.40±0.00	89.76±0.35	91.43±0.40	181.59±0.74	0.98±0.00
醉金香 Zuijinxinag	1	0.29±0.00	83.45±0.36	80.25±0.39	163.99±0.74	1.04±0.00
	2	0.32±0.00	90.30±0.33	87.39±0.31	178.01±0.64	1.03±0.00
	3	0.36±0.00	100.58±0.84	94.76±0.82	195.69±1.66	1.06±0.00
平均值 Mean		0.50	86.62	90.94	178.06	0.96
标准差 Standard deviation		0.68	8.57	10.43	17.89	0.07
变异系数 Coefficient of variation/%		135.86	9.89	11.46	10.05	6.82

表 4 不同品种及采收批次葡萄果汁中有机酸的组成与含量

Table 4 Composition and content of organic acids in grape juices of different varieties and harvest batches (mg·L⁻¹)

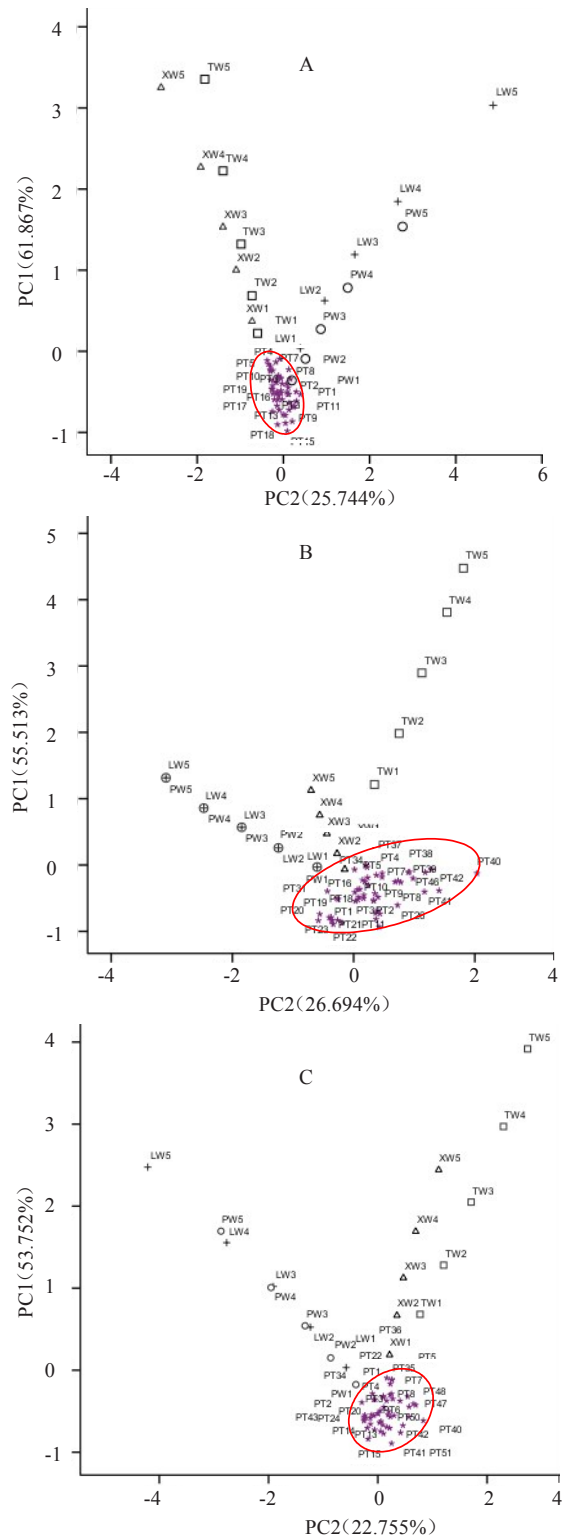
品种 Variety	采样批次 Harvest batch	酒石酸 Tartaric acid	苹果酸 Malic acid	莽草酸 Shikimic acid	柠檬酸 Citric acid	富马酸 Fumaric acid	总酸 Total acid
红宝石无核 Ruby seedless	1	5 978.11±23.20	1 094.30±6.46	9.01±0.09	298.25±6.24	6.40±0.04	7 386.07±35.07
	2	6 022.48±5.63	1 347.77±5.80	12.60±0.13	294.63±3.81	9.83±0.10	7 687.31±4.67
	3	5 899.45±131.30	1 091.83±2.63	10.52±0.07	281.46±4.31	8.08±0.01	7 291.34±128.11
红地球 Red globe	1	4 749.26±7.93	2 303.52±22.40	7.27±0.04	530.66±7.85	8.55±0.00	7 599.26±31.34
	2	4 937.48±4.22	2 194.63±17.19	7.54±0.11	471.75±2.71	9.47±0.07	7 620.87±22.17
	3	4 721.77±38.24	2 215.32±15.46	7.28±0.08	479.27±13.94	7.40±0.05	7 431.05±43.04
金手指 Gold finger	1	6 288.51±90.84	2 809.22±11.74	4.43±0.05	401.18±0.87	8.67±0.02	9 512.01±86.32
	2	6 313.64±108.19	2 854.75±40.85	5.08±0.08	405.68±4.63	9.21±0.06	9 588.36±74.99
	3	6 573.48±99.63	2 842.56±50.61	5.45±0.06	401.46±2.26	9.47±0.13	9 832.41±64.22
京秀 Jingxiu	1	7 140.10±100.15	1 718.12±54.47	13.81±0.21	290.85±5.09	9.02±0.11	9 171.89±126.90
	2	7 182.19±192.22	1 652.41±61.56	12.21±0.28	254.32±3.14	8.02±0.04	9 109.15±145.50
	3	7 148.56±61.86	1 702.08±34.03	10.22±0.06	244.83±5.52	7.60±0.03	9 113.28±61.12
巨峰 Kyoho	1	6 836.43±35.28	2 116.01±6.40	6.17±0.04	448.03±3.17	3.93±0.06	9 410.57±43.78
	2	6 675.21±72.57	1 814.02±29.60	5.69±0.08	424.50±4.48	4.25±0.04	8 923.66±102.37
	3	6 504.87±37.36	1 993.80±26.34	6.23±0.01	451.53±8.75	5.19±0.03	8 961.62±2.32
巨玫瑰 Jumeigui	1	5 924.61±151.95	2 368.22±43.79	3.45±0.01	476.95±4.41	3.63±0.01	8 776.85±107.39
	2	5 719.11±88.13	2 207.30±10.19	3.20±0.03	492.48±2.27	4.31±0.02	8 426.40±78.11
	3	5 949.02±89.53	2 047.40±32.43	4.13±0.03	525.24±9.81	3.73±0.03	8 529.52±111.83
摩尔多瓦 Moldova	1	7 922.54±38.85	1 347.57±14.95	15.38±0.22	405.04±1.78	0.20±0.01	9 690.72±54.40
	2	8 173.52±284.11	993.18±23.31	14.86±0.29	376.59±2.49	0.11±0.00	9 558.27±303.38
	3	8 874.70±71.68	1 610.38±24.28	14.71±0.24	427.46±6.15	0.49±0.02	10 927.74±95.80
尼加拉 Niagara	1	6 807.51±163.71	853.71±26.94	5.68±0.04	177.65±2.46	1.27±0.02	7 845.83±186.45
	2	7 428.39±81.44	982.24±6.76	6.02±0.08	183.00±0.93	0.99±0.03	8 600.64±75.23
	3	7 500.66±358.97	970.36±38.16	6.28±0.23	180.06±3.83	0.94±0.02	8 658.29±379.41
森田无核 Centennial seedless	1	6 655.36±175.49	703.26±9.20	2.94±0.05	294.92±0.94	8.28±0.08	7 664.76±177.52
	2	6 772.17±243.79	1 002.45±20.68	2.99±0.08	308.12±5.96	10.75±0.13	8 096.48±113.75
	3	7 332.01±284.78	890.39±14.48	3.26±0.07	268.68±3.97	7.76±0.11	8 502.10±268.20
维多利亚 Victoria	1	5 792.75±229.36	2 319.11±8.33	19.81±0.09	228.21±5.16	8.63±0.05	8 368.53±222.24
	2	5 577.86±218.30	2 219.18±41.62	15.27±0.15	229.89±8.77	8.23±0.13	8 050.43±214.80
	3	5 882.14±236.15	2 176.62±24.18	13.65±0.18	206.22±1.08	8.15±0.06	8 286.79±249.82
夏黑 Summer black	1	7 011.58±275.40	2 486.97±36.60	21.63±0.33	323.77±6.48	2.23±0.02	9 846.17±256.62
	2	7 910.79±299.85	2 413.87±50.07	19.93±0.27	326.97±4.28	2.52±0.01	10 674.08±291.72
	3	7 690.52±355.33	2 209.67±22.14	18.88±0.31	338.72±3.38	2.96±0.03	10 260.75±371.05
夏至红 Xiazhihong	1	5 063.98±34.79	2 741.43±31.37	19.00±0.31	472.50±13.21	7.15±0.09	8 304.06±42.57
	2	5 215.80±158.54	2 923.16±121.17	18.15±0.10	501.43±9.10	8.45±0.12	8 666.99±46.52
	3	4 862.00±55.90	3 029.62±88.79	16.26±0.08	474.37±4.47	8.05±0.19	8 390.30±139.25
秀玉 Xiuyu	1	6 436.20±65.23	3 467.92±27.07	8.12±0.06	698.27±4.21	6.32±0.02	10 616.83±72.03
	2	6 481.33±105.85	3 341.35±78.67	5.66±0.14	703.52±7.54	9.81±0.17	10 541.66±155.70
	3	6 726.58±33.18	2 933.06±50.22	5.28±0.02	641.76±14.09	10.82±0.07	10 317.50±60.91
阳光玫瑰 Shine-muscat	1	5 774.79±245.82	2 213.54±35.79	1.19±0.02	370.36±11.65	21.67±0.20	8 381.55±270.28
	2	6 315.74±43.37	1 684.69±17.43	1.37±0.03	294.87±8.57	13.99±0.11	8 310.65±54.26
	3	6 210.34±65.79	1 502.89±20.97	1.18±0.01	297.26±4.04	16.49±0.19	8 028.16±74.74
早熟红 Flame	1	6 887.17±296.12	392.53±8.58	2.11±0.02	172.95±1.32	1.59±0.03	7 456.36±303.45
	2	6 834.30±259.70	635.98±17.58	2.30±0.01	227.84±4.06	1.98±0.10	7 702.40±272.08
	3	7 215.44±117.08	619.27±12.34	2.38±0.07	206.74±1.09	2.32±0.02	8 046.15±113.75
泽香 Zexiang	1	5 263.57±38.44	2 877.39±83.12	1.67±0.04	500.38±14.18	11.01±0.19	8 654.01±123.73
	2	5 547.32±97.23	2 871.73±4.86	1.39±0.03	443.68±9.87	13.11±0.24	8 877.22±86.77
	3	5 320.58±73.79	2 882.65±67.78	1.39±0.01	454.17±8.55	14.27±0.32	8 673.06±110.33
醉金香 Zuijinxiang	1	8 230.44±198.04	1 599.08±35.70	1.74±0.01	487.66±10.24	4.77±0.14	10 323.68±243.47
	2	8 907.45±279.91	1 514.27±21.64	1.66±0.01	493.65±2.30	4.28±0.04	10 921.30±294.86
	3	9 760.75±269.12	1 319.82±33.27	1.40±0.02	531.95±3.28	4.13±0.09	11 618.05±277.49
平均值 Mean		6 567.66	1 923.58	8.00	380.82	6.87	8 886.92
标准差 Standard deviation		1 111.72	791.23	6.13	132.85	4.45	1 079.43
变异系数 Coefficient of variation/%		16.93	41.13	76.65	34.89	64.82	12.15

果汁,分析其中的可溶性糖和有机酸组成,与51个葡萄原汁的分析结果一起组成数据矩阵,用SPSS22.0软件进行主成分分析,根据各样品在前两个主成分上的得分作图,得到得分图(图1)。

图1-A为利用可溶性糖组成进行主成分分析的得分图。前两个主成分方差贡献率分别为61.867%和25.744%,所有葡萄原汁样品主要集中于第一主成分的负方向,随着掺入苹果汁、梨汁、桃汁、杏汁量的增加,掺假样品逐渐向正方向偏移,掺假量越高,距离葡萄原汁的集中分布区越远。除掺加20%苹果汁的样品(PW1)外,其他掺假样品均可与葡萄原汁区分开来。当以有机酸组成进行主成分分析时,前两个主成分方差贡献率分别为55.513%和26.694%,葡萄原汁样品同样主要位于第一主成分的负方向,但分布较以可溶性糖组成进行主成分分析时分散(图1-B)。随着掺入苹果汁、梨汁、桃汁、杏汁量的增加,掺假样品逐渐偏离葡萄原汁分布区。除掺加20%杏汁的样品(XW1)外,其他掺假样品均可与葡萄原汁区分开来。为了进一步改善区分效果,尝试利用可溶性糖和有机酸组成进行主成分分析。由图1C可见,以可溶性糖和有机酸组成进行主成分分析得到的得分图中,葡萄原汁的分布比较集中,所有掺假样品都可与葡萄原汁区分开来。这说明利用可溶性糖和有机酸组成进行主成分分析用于识别掺入苹果汁、梨汁、桃汁、杏汁的掺假葡萄汁样品优于单一利用可溶性糖或有机酸组成。

2.3.2 线性判别分析 线性判别分析(linear discriminant analysis, LDA)是一种有监督降维分析方法,常与主成分分析法配合使用,广泛应用于食品真伪鉴别^[18-19]。将51个葡萄原果汁以及20个掺假样品的可溶性糖和/或有机酸分析数据用SPSS22.0软件进行线性判别分析,根据前两个典型判别函数计算各样品的得分并作图,得到线性判别图(图2)。

图2-A所示为以可溶性糖组成进行线性判别分析的结果。由图可见,51个葡萄原汁样品在线性判别图中分布较集中,但与掺加苹果汁的掺假样品有较多重叠。当以有机酸组成进行线性判别分析时,得到的判别图中葡萄原汁分布较为分散,而且与掺加杏汁的样品相距较近(图2-B)。将可溶性糖和有机酸组成结合进行线性判别分析,得到的判别图中葡萄原汁分布较集中,而且避免了与掺加苹果汁的掺假样品的重叠(图2-C)。说明综合利用可溶性糖

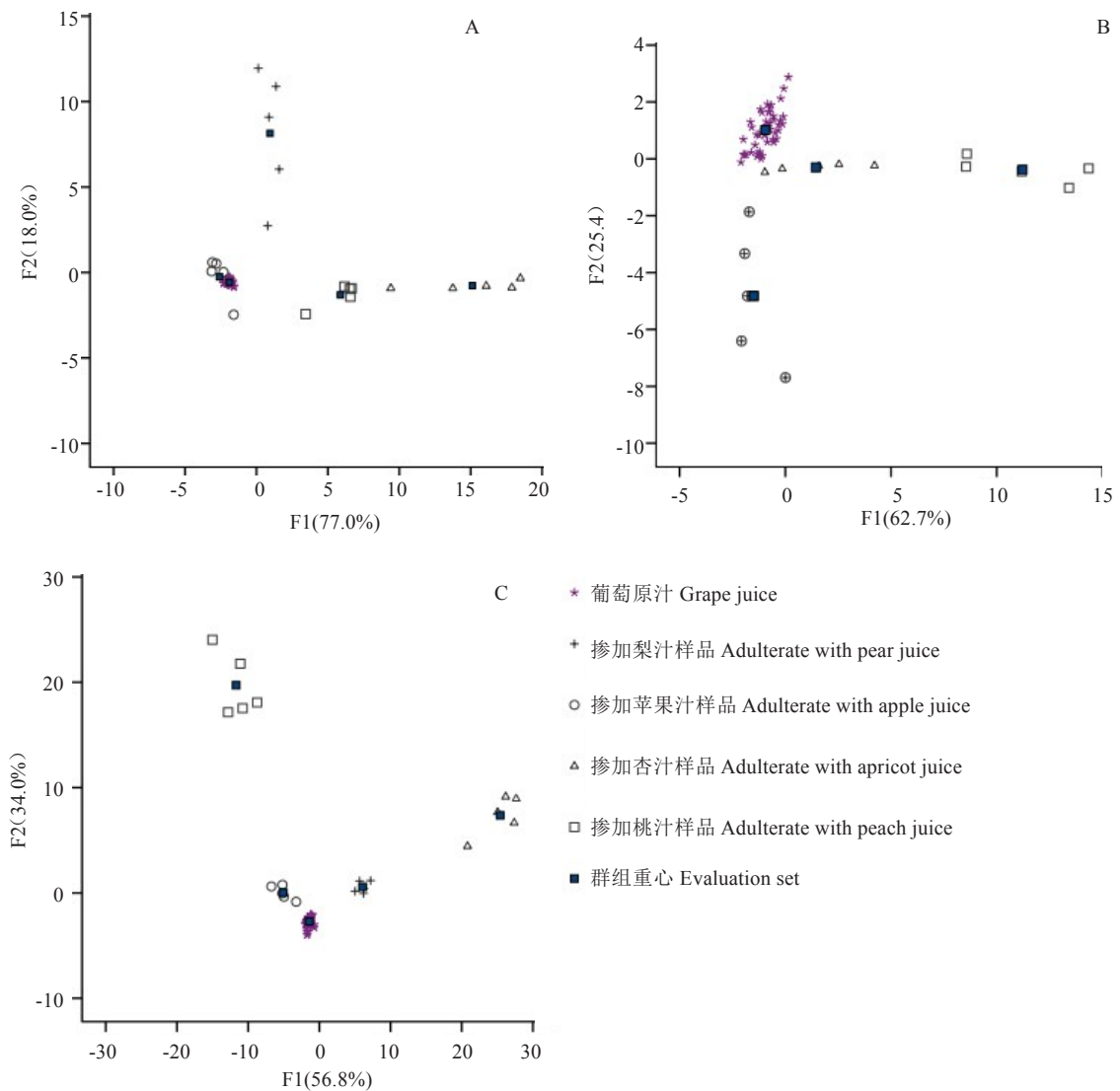


A. 以可溶性糖组成进行主成分分析; B. 以有机酸组成进行主成分分析; C. 以可溶性糖和有机酸组成进行主成分分析。*葡萄原汁; +掺加梨汁样品; O掺加苹果汁样品; Δ掺加杏汁样品; □掺加桃汁样品; ■群组重心。

A. PCA with soluble sugar composition; B. PCA with organic acid composition; C. PCA with soluble sugar and organic acid composition. * Grape juice; + Adulterate with pear juice; O Adulterate with apple juice; Δ Adulterate with apricot juice; □ Adulterate with peach juice; ■ Evaluation set.

图1 主成分分析得分图

Fig. 1 Score plots obtained from the principal component analysis (PCA)



A. 以可溶性糖组成进行判别分析;B. 以有机酸组成进行判别分析;C. 以可溶性糖和有机酸组成进行判别分析。

A. LDA with soluble sugar composition; B. LDA with organic acid composition; C. LDA with soluble sugar and organic acid composition.

图 2 线性判别

Fig. 2 Score plot of linear discriminant analysis (LDA)

和有机酸组成进行线性判别分析用于识别掺入苹果汁、梨汁、桃汁、杏汁的掺假葡萄汁样品优于单一利用可溶性糖或有机酸组成。这也与主成分分析的结果相一致。

表 5 为利用可溶性糖进行线性判别分析输出的预测分类结果。由表可见,在原始判别中分别有一个掺加苹果汁和梨汁的掺假样品被错误地分在葡萄原汁组,而在交叉验证中一个掺加桃汁的样品也被错误地分在葡萄原汁组,而且一个葡萄汁样品被错误地分入掺加苹果汁的掺假样品组,原始判别和交叉验证的正确率分别为 95.8%和 93.0%。当利用有机酸组成进行线性判别分析时,在原始判别和

交叉验证中均分别有一个掺加梨汁、苹果汁和杏汁的掺假样品被错误地分在葡萄原汁组,正确率分别为 90.1%和 83.1%(交叉验证中一个掺加桃汁的样品被错误地分在掺加杏汁的样品组)(表 6)。将可溶性糖和有机酸组成结合进行线性判别分析,在原始判别中所有样品均得到正确的分类,正确率为 100%,交叉验证中分别有一个掺加苹果汁和梨汁的掺假样品被错误地分在葡萄原汁组,正确率为 95.8%(表 7)。再次验证了综合利用可溶性糖和有机酸组成用于识别掺入苹果汁、梨汁、桃汁、杏汁的掺假葡萄汁样品优于单一利用可溶性糖或有机酸组成。

表 5 以可溶性糖组成进行判别分析的预测分类结果

Table 5 Results of discriminant analysis with soluble sugar

		composition						
		果汁种类 Juice	预测组成员 Predicted group membership					合计 Total
			PT	LW	PW	XW	TW	
初始 Original	计数 Count	PT	51	0	0	0	0	51
		LW	1	4	0	0	0	5
		PW	1	0	4	0	0	5
		XW	0	0	0	4	1	5
		TW	0	0	0	0	5	5
	%	PT	100.0	0.0	0.0	0.0	0.0	100.0
		LW	20.0	80.0	0.0	0.0	0.0	100.0
		PW	20.0	0.0	80.0	0.0	0.0	100.0
		XW	0.0	0.0	0.0	80.0	20.0	100.0
		TW	0.0	0.0	0.0	0.0	100.0	100.0
交叉验证 Cross-validation	计数 Count	PT	50	0	1	0	0	51
		LW	1	4	0	0	0	5
		PW	1	0	4	0	0	5
		XW	0	0	0	4	1	5
		TW	1	0	0	0	4	5
	%	PT	98.0	0.0	2.0	0.0	0.0	100.0
		LW	20.0	80.0	0.0	0.0	0.0	100.0
		PW	20.0	0.0	80.0	0.0	0.0	100.0
		XW	0.0	0.0	0.0	80.0	20.0	100.0
		TW	20.0	0.0	0.0	0.0	80.0	100.0

注:PT. 葡萄原汁;LW. 掺加梨汁的样品;PW. 掺加苹果汁的样品;XW. 掺加杏汁的样品;TW. 掺加桃汁的样品。

Note: PT. Grape juice; LW. Adulterate with pear juice; PW. Adulterate with apple juice; XW. Adulterate with apricot juice; TW. Adulterate with peach juice.

表 6 以有机酸组成进行判别分析的预测分类结果

Table 6 Classification results of discriminant analysis with

		organic acid composition						
		果汁种类 Juice	预测组成员 Predicted group membership					合计 Total
			PT	LW	PW	XW	TW	
初始 Original	计数 Count	PT	51	0	0	0	0	51
		LW	1	3	1	0	0	5
		PW	1	3	1	0	0	5
		XW	1	0	0	4	0	5
		TW	0	0	0	0	5	5
	%	PT	100.0	0.0	0.0	0.0	0.0	100.0
		LW	20.0	60.0	20.0	0.0	0.0	100.0
		PW	20.0	60.0	20.0	0.0	0.0	100.0
		XW	20.0	0.0	0.0	80.0	0.0	100.0
		TW	0.0	0.0	0.0	0.0	100.0	100.0
交叉验证 Cross-validation	计数 Count	PT	51	0	0	0	0	51
		LW	1	0	4	0	0	5
		PW	1	4	0	0	0	5
		XW	1	0	0	4	0	5
		TW	0	0	0	1	4	5
	%	PT	100.0	0.0	0.0	0.0	0.0	100.0
		LW	20.0	0.0	80.0	0.0	0.0	100.0
		PW	20.0	80.0	0.0	0.0	0.0	100.0
		XW	20.0	0.0	0.0	80.0	0.0	100.0
		TW	0.0	0.0	0.0	20.0	80.0	100.0

注:PT. 葡萄原汁;LW. 掺加梨汁的样品;PW. 掺加苹果汁的样品;XW. 掺加杏汁的样品;TW. 掺加桃汁的样品。

Note: PT. Grape juice; LW. Adulterate with pear juice; PW. Adulterate with apple juice; XW. Adulterate with apricot juice; TW. Adulterate with peach juice.

表 7 以可溶性糖和有机酸组成进行判别分析的预测分类结果

Table 7 Results of discriminant analysis with soluble sugar and organic acid composition

		果汁种类 Juice	预测组成员 Predicted group membership					合计 Total
			PT	LW	PW	XW	TW	
初始 Original	计数 Count	PT	51	0	0	0	0	51
		LW	0	5	0	0	0	5
		PW	0	0	5	0	0	5
		XW	0	0	0	5	0	5
		TW	0	0	0	0	5	5
	%	PT	100.0	0.0	0.0	0.0	0.0	100.0
		LW	0.0	100.0	0.0	0.0	0.0	100.0
		PW	0.0	0.0	100.0	0.0	0.0	100.0
		XW	0.0	0.0	0.0	100.0	0.0	100.0
		TW	0.0	0.0	0.0	0.0	100.0	100.0
交叉验证 Cross-validation	计数 Count	PT	51	0	0	0	0	51
		LW	1	4	0	0	0	5
		PW	1	0	4	0	0	5
		XW	0	0	0	5	0	5
		TW	0	0	1	0	4	5
	%	PT	100.0	0.0	0.0	0.0	0.0	100.0
		LW	20.0	80.0	0.0	0.0	0.0	100.0
		PW	20.0	0.0	80.0	0.0	0.0	100.0
		XW	0.0	0.0	0.0	100.0	0.0	100.0
		TW	0.0	0.0	20.0	0.0	80.0	100.0

注:PT. 葡萄原汁;LW. 掺加梨汁的样品;PW. 掺加苹果汁的样品;XW. 掺加杏汁的样品;TW. 掺加桃汁的样品。

Note: PT. Grape juice; LW. Adulterate with pear juice; PW. Adulterate with apple juice; XW. Adulterate with apricot juice; TW. Adulterate with peach juice.

3 讨 论

如果没有人为干预,果汁中可溶性糖和有机酸的组成及含量主要与水果原料有关。原料水果的种类是决定其可溶性糖和有机酸组成的首要因素,因此可根据果汁的糖酸组成特征对果汁种类与真伪进行鉴别和区分^[14-17]。但由于品种、采收成熟度以及栽培条件等可对水果中各种可溶性糖和有机酸的含量产生影响,使得不同品种及原料来源的同一种类果汁中可溶性糖和有机酸组成与含量存在较大差异,明确不同品种及原料来源的果汁中可溶性糖和有机酸组成的共有特征以及与其他种类果汁的差异性成为利用糖酸组成特征进行果汁鉴伪的关键。本研究对17个常见葡萄品种共51个采收批次的果实榨汁,分别分析其可溶性糖和有机酸组成,证实不同品种及采收成熟度的葡萄汁中可溶性糖和有机酸组成与含量存在较大差异,但含有可溶性糖和有机酸的种类与基本特征具有较好的一致性。如:各葡萄汁样品中主要的可溶性糖均主要是葡萄糖和果糖,而且二者含量相当,而有机酸主要是酒石酸和苹果酸,尤其是酒石酸,占检测到的总有机酸含量的57.95%~92.37%。已有研究表明,苹果汁中可溶性糖主要是果糖、蔗糖和葡萄糖,而且果糖与葡萄糖的比值在2.0以上^[20],有机酸主要是苹果酸^[17];梨果汁具有较高含量的山梨醇^[21],而且有机酸主要是苹果酸、奎宁酸和柠檬酸^[17];桃果实中可溶性糖主要是蔗糖,葡萄糖和果糖含量很少,有机酸主要是苹果酸和奎宁酸^[22];杏果实中可溶性糖主要是蔗糖,且葡萄糖含量均高于果糖(二者比值为1.35~1.73),而有机酸则以苹果酸或柠檬酸为主(因品种而异)^[23]。这些水果或果汁的可溶性糖和/或有机酸组成与葡萄汁均存在一定的差别,在葡萄果汁中掺假以上果汁会导致可溶性糖和/或有机酸组成特征发生变化,从而可利用糖酸组成特征鉴别葡萄汁掺假苹果汁、梨汁、桃汁、杏汁的行为。

利用食品或农产品的化学组成对食品品质进行评价或鉴别必须借助一定的算法或模式识别方法才能获得好的效果。Abad-Garcia等^[24]分析了83个柑橘汁样品中的酚类物质组成与含量,采用聚类分析和主成分分析等化学计量学方法根据酚类物质组成实现了甜橙、橘子、柠檬、葡萄柚等四类柑橘汁的区分,并成功预测了甜橙汁中掺假葡萄柚汁的比例;

Guo等^[25]分析了陕西省5个品种、6个产地的58个苹果汁样品中酚类物质组成,结合逐步线性判别分析(stepwise linear discriminant analysis, SLDA)方法可实现品种和产地的鉴别;黎海红等^[26]采用主成分分析法识别芝麻油掺假菜籽油、豆油、葵花油、花生油;Navarro-Pascual-Ahuir等^[14-15]在利用可溶性糖或有机酸组成对苹果、葡萄、柑橘、橙、菠萝等5种果汁以及掺入50%葡萄汁的橙汁、菠萝汁进行区分时也应用了线性判别分析。本研究采用主成分分析和线性判别分析对葡萄果汁及其模拟掺假样品的可溶性糖和有机酸组成进行降维处理,实现了葡萄果汁与大部分掺假样品的区分。其中,综合利用可溶性糖和有机酸组成结合主成分分析的区分效果最好,所有掺假样品都可与葡萄原汁区分开来。在进行线性判别分析时,原始判别中所有样品均得到正确的分类,正确率为100%,但交叉验证中分别有一个掺加苹果汁和梨汁的掺假样品被错误地分在葡萄原汁组,正确率为95.8%。单一利用可溶性糖或有机酸组成进行主成分分析或线性判别分析,对掺假样品的识别效果均差于二者综合应用。这说明模式识别方法以及采用的数据矩阵不同也可影响鉴伪效果。这是因为不同的模式识别方法,对数据的处理和算法不同,所得到的结果可能会存在差异。Martelo-Vidal等^[27]在对红葡萄酒原产地的鉴别中也发现不同的模式识别方法得到的区分效果存在较大差异。Papapetros等^[28]在对甜樱桃品种和产地的鉴别中发现综合采用多组数据矩阵(常规品质指标与挥发性成分以及矿物质)进行线性判别分析得到的分组正确率明显高于单一利用常规品质指标、挥发性成分或矿物质含量。这与本研究结果相似。因此,在未来的研究中,也可尝试进一步优化数据矩阵和模式识别方法以提高鉴伪效果。

4 结 论

葡萄汁中可溶性糖主要由葡萄糖和果糖组成,二者之和占总糖含量的97.88%~99.86%。酒石酸是其中最主要的有机酸,占总酸含量的57.95%~92.37%。不同品种及采收成熟度的葡萄汁中可溶性糖和有机酸含量存在较大差异,但其基本组成特征具有较好一致性,可用于葡萄汁的掺假鉴定和质量控制。综合应用可溶性糖和有机酸组成的鉴别效果优于单一利用可溶性糖或有机酸组成。

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更正

由于本人的工作疏漏,把2019年第10期1273页“新中国果树科学研究70年——梨”一文中的作者项中王文辉没体现出来,他完成的是论文中贮藏加工内容。特此恳请编辑部予以更正。王文辉,中国农业科学院果树研究所,排名第一,英文同。谢谢!

更正如下。

本文通信作者

新中国果树科学研究70年——梨

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