

不同成熟期琯溪蜜柚果实功能成分的差异分析

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摘要:【目的】探究不同成熟期琯溪蜜柚果实不同组织部位的功能成分的差异性。【方法】超高效液相色谱法测定类黄酮、酚酸、香豆素、类柠檬苦素等营养成分, 顶空固相微萃取-气质联用仪检测挥发性物质。【结果】不同成熟期琯溪蜜柚4种主要类黄酮的含量都在成熟期呈下降的趋势; 不同成熟期琯溪蜜柚的不同部位7种酚酸的含量存在显著差异, 单一酚酸随成熟期变化趋势不同, 根据在同一成熟阶段不同部位酚酸总量来看, 呈现以下规律: 黄皮层>种子>囊衣>白皮层>果肉>果汁; 从不同部位香豆素总量来看, 黄皮层中香豆素含量最丰富; 种子类柠檬苦素随着成熟期延长而上升, 其他部位中类柠檬苦素呈现相反的趋势; 3个成熟阶段琯溪蜜柚黄皮层共检测到51种香气物质, 随着成熟期的延长香气物质总含量呈增长的趋势, 分别为7 896.769、8 664.805、10 380.399 $\mu\text{g}\cdot\text{g}^{-1}$ 。最后通过主成分分析显示, 3个成熟阶段的琯溪蜜柚果汁功能成分存在显著差异。【结论】不同成熟度对琯溪蜜柚功能成分含量影响显著, 可根据不同的开发利用目标, 选择合适的采摘期, 充分利用其有效成分。

关键词: 琯溪蜜柚; 成熟阶段; 功能成分; 主成分分析

中图分类号: S666.3

文献标志码: A

文章编号: 1009-9980(2022)06-1042-12

Analysis on the difference of functional components of Guanxi pomelo fruits during maturity

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Abstract: 【Objective】The study aimed to investigate the difference of the functional components including flavonoids, phenolic acids, coumarins, limonoids and volatile compounds in the different parts of tissue of Guanxi pomelo fruits at three mature stages (immature, semi mature and mature). 【Methods】Guanxi pomelo fruit samples were harvested from September to November, 2020. The mature stages were defined according to the peel color, that is: cyan was marked as immature, green to yellow as semi mature, and yellow as mature stage, respectively. The fresh samples were cut into four parts and manually divided into flavedo, albedo, capsules, seeds and pulps. The juices were extracted by a household juicer and filtered through four layers of fine cloth. The functional components including flavonoids, phenolic acids, coumarins and limonoids were determined by ultra-high performance liquid chromatography (UPLC) and the volatile compounds were detected by gas chromatography-mass spectrometry combined with headspace solid-phase microextraction (HS-SPME-GC-MS) to detect the difference of functional components in different parts of the fruits and at different mature stages. 【Results】The contents of four main flavonoids including naringin, rhoifolin, eriocitrin and neohesperidin in the fruits of Guanxi pomelo decreased with maturity. Naringin was the most dominant flavonoid and decreased ob-

收稿日期: 2021-12-24 接受日期: 2022-04-30

基金项目: 重庆市技术创新与应用发展专项面上项目(cstc2021jcsx-lyjsAX0007); 农业农村部现代农业(柑橘)产业技术体系建设专项(CARS-26); 国家重点研发计划项目(2019YFC1605604); 国家柑橘及热带作物产品质量安全风险评估专项(GJFP2019012, GJFP2019043)

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servably with maturity. The contents of naringin in the flavedo, albedo, capsule, seed, pulp and juice were in the ranges of 85.41%–89.28%, 94.65%–97.11%, 96.12%–97.18%, 88.41%–90.69%, 65.00%–73.73% and 75.43%–79.42%, respectively. There was a notable difference of the total contents of phenolic acid in the different parts of Guanxi pomelo fruits at the same mature stage in the order of flavedo>seed>capsule>albedo>pulp>juice. The total contents of phenolic acid in the flavedo, albedo, capsule, seed and juice at mature stage were higher than those at immature stage and the total content in the pulp decreased gradually with maturity. The total amount of coumarin in the flavedo increased with maturity, the total amount of coumarin in the albedo, juice and seed decreased with maturity and the total amount of coumarin in the pulp and capsule first increased and then decreased with maturity. The contents of coumarin in the flavedo, albedo, capsule, seed, pulp and juice were in the ranges of 133.189–397.775, 1.528–2.662, 7.589–15.673, 2.276–8.253, 3.110–4.574 and 0.745–1.679 mg·kg⁻¹, respectively. The limonoids in the seed were the most abundant. The contents of limonin, nomilin and obacunone in the seed increased by 47.11%, 60.90% and 86.47%, respectively, at the maturity stage. The limonin and nomilin in the flavedo, albedo, capsule, pulp and fruit juice decreased sharply during maturity. 51 kinds of the volatile compounds were detected in the flavedo of Guanxi pomelo fruits at three mature stages by GC-MS. Among them, 33, 27 and 43 kinds of compounds were identified at the three mature stages, respectively. The total contents of volatile compound increased gradually as the fruits matured and they were 7896.769, 8664.805 and 10380.399 μg·g⁻¹, at three stages, respectively. There was significant difference in the functional components of the juice of the Guanxi pomelo fruits at three mature stages based on principal component analysis. 【Conclusion】The contents of the functional components in the different parts of the Guanxi pomelo fruits varied with the fruit maturity. The most of the flavonoids, phenolic acids, coumarins and limonoids showed a downward trend and a few showed an upward trend with the extension of maturity. The contents of the functional components in the different parts of the fruits were different at the same maturity stage. The contents of the flavonoids, phenolic acids and coumarins in the flavedo were higher than those in the other parts and the content of limonoids in the seed was the highest. The total content of volatile compounds increased with the extension of the mature stage. The study would provide a clew for comprehensive utilization of the functional components in the fruits of Guanxi pomelo.

Key words: Guanxi pomelo; Mature stage; Functional components; Principal component analysis

柑橘是世界第一大宗水果,按种类主要包括宽皮柑橘、橙类、柚类、柠檬和金柑五大类^[1]。柚属芸香科柑橘属植物,是一类果实硕大、果肉晶莹剔透、营养丰富、风味清香独特、耐贮运的柑橘品种^[2]。柚类在我国品种多样,资源丰富,主要栽培品种有琯溪蜜柚、沙田柚、文旦柚。我国柚类产量达598.28万吨,占总柑橘产量的13.05%(2019年),其中70%为琯溪蜜柚^[3]。促进人类健康一直是科学研究的重点,柚果因其独特的感官特性以及高价值的营养功能,以及对柚果果实营养成分的探索受到越来越多的关注,包括糖、维生素C、类黄酮^[4]、酚酸^[5]、类柠檬苦素^[6]、香豆素^[7-8]、挥发性物质等多种营养成分^[9]。这些物质不仅能为人体的生命活动提供基本的能量

和营养,而且对糖尿病、心血管^[10]、骨质疏松^[11]、炎症、结肠癌等多种疾病有一定疗效^[12]。

目前针对柚子主要集中于果皮精油,果汁等加工制品中某一特定功能成分的研究,但对柚子在不同成熟期功能成分动态系统研究较少,例如张利军等^[13]对琯溪蜜柚花生长发育进行分级,并探索其生物学特性和养分动态规律;代亚兰等^[14]比较了不同成熟期琯溪蜜柚汁胞中品质指标的差异变化。笔者在本研究中测定琯溪蜜柚果实成熟的3个阶段(青色、青转黄、黄色)不同部位的黄酮类化合物、香豆素、酚酸、类柠檬苦素的含量差异以及挥发性物质的变化,评估琯溪蜜柚果实成熟的3个阶段(青色、青转黄和黄色)综合品质,为琯溪蜜柚果实的不同生产

目的选择适合的采收成熟度及其副产品的利用提供指导。

1 材料和方法

1.1 植物材料、试剂与仪器

琯溪蜜柚原料采自中国农业科学院柑桔研究所国家果树种质资源(重庆)柑橘圃,树龄为15 a左右,砧木为酸柚,具体采摘时间见表1。在3株树势较为一致果树上的不同果实着生方位(上、中、下、内、外部)均匀地采摘8~10个,成熟阶段根据果皮颜色定义如下:青色(未成熟)阶段、青转黄(半成熟)阶段和黄色(成熟)阶段,见图1。将新鲜水果样品切成四半,先手动分离黄皮层、白皮层、囊衣、种子和果肉等部位,进行样品制备;用家用榨汁机榨汁,通过4层细布过滤收集的果汁,实验使用新鲜制备的样品进行分析。笔者在本研究中共使用14种类黄酮、7种

酚酸、3种类柠檬苦素、14种香豆素等标准物质,购于上海源叶生物科技有限公司。

KQ5200DE超声波清洗器,江苏昆山市超声仪器有限公司;Sigma3K15高速冷冻离心机,美国Sigma公司;0.22 μm 有机相针式滤器,上海安谱科学仪器有限公司;RE-52AA型旋转蒸发仪,上海亚荣生化仪器厂;KS260摇床,德国IKA公司;SHZ-D型循环水式真空泵,巩义予华仪器有限公司;UPLC色谱仪(2996PDA检测器),美国Waters公司;7890A/5975C气相色谱一单四极杆质谱仪,美国Agilent公司。

表1 琯溪蜜柚样品采摘信息

Table 1 Sample information of Guanxi pomelo

序号 Number	品种名称 Variety name	资源编号 Repository number	采摘时间 Picking date
1	琯溪蜜柚	LG0038	2020-09-15
2	Guanxi pomelo		2020-10-15
3			2020-11-15



图1 不同成熟期琯溪蜜柚果实样品图

Fig. 1 Samples of Guanxi pomelo fruits at different mature stages

1.2 类黄酮测定

参考冉玥等^[5]的方法,称取样品5.00 g于离心管中,加入10 mL甲醇和二甲基亚砷体积比=1:1,超声提取30 min,10 000 $\text{r} \cdot \text{min}^{-1}$ 离心10 min,收集上清液,提取2次,定容至25 mL,经0.22 μm 微孔有机滤膜过滤后上机检测。

UPLC条件:流动相:0.2%乙酸水溶液(A)和甲醇(B);流速:0.30 $\text{mL} \cdot \text{min}^{-1}$,柱温:35 $^{\circ}\text{C}$;波长为283 nm。

1.3 酚酸含量测定

参考张静等^[6]的方法,取样品5.00 g于离心管中,加入4 $\text{mol} \cdot \text{L}^{-1}$ 10 mL NaOH溶液,振荡反应4 h;调节pH至2.0(6 $\text{mol} \cdot \text{L}^{-1}$ 盐酸,约12 mL),10 000 $\text{r} \cdot \text{min}^{-1}$ 离心5 min;取上清液,用乙酸乙酯和甲基叔丁基醚(体积比1:1)萃取3次;合并萃取液,旋转蒸

发浓缩至干,用50%甲醇定容至5 mL,经0.22 μm 微孔有机滤膜过滤后上机检测。

UPLC条件一色谱柱:ACQUITY UPLC BEH C18分析柱(2.1 mm \times 100 mm,1.7 μm);流动相:3.0%甲酸水溶液(A)和甲醇溶液(B);流速:0.30 $\text{mL} \cdot \text{min}^{-1}$;柱温:35 $^{\circ}\text{C}$;进样量为3.0 μL ;检测波长:原儿茶酸、对羟基苯甲酸和香草酸为260 nm,咖啡酸、*p*-香豆酸、芥子酸和阿魏酸为320 nm。

1.4 香豆素含量测定

与类黄酮提取方法一致。

UPLC条件:流动相:0.1%乙酸水溶液(A)和乙酸甲醇溶液(B);流速:0.20 $\text{mL} \cdot \text{min}^{-1}$,柱温:40 $^{\circ}\text{C}$;进样量为3.0 μL ;检测波长:330 nm和300 nm。

1.5 类柠檬苦素含量测定

参考李一兵等^[7]的方法,称取样品5.00 g于离

心管中,加入10 mL二氯甲烷,高速匀浆提取5 min,10 000 r·min⁻¹离心5 min,收集上清液,再重复上述操作1次,合并2次提取的下层液,旋蒸浓缩至干,用甲醇定容至2.0 mL,经0.22 μm微孔有机滤膜过滤后上机检测。

UPLC条件——流速:0.3 mL·min⁻¹;流动相比例:乙腈、水=40:60;柱温:35℃;波长:210 nm;进样量:3 μL。

1.6 挥发性物质含量测定

参考郑洁等^[8]的方法,将采摘的柚果表面擦拭干净,四分法切开得到果皮,打碎混匀,称取3.00 g样品,置20 mL螺口样品瓶中,加入3.00 mL饱和食盐水和2.00 μL环己酮(99.5%)作为内标物,用聚四氟乙烯隔垫密封,于50℃磁力搅拌器上加热平衡20 min。用萃取头顶空吸附50 min后,将萃取头插入GC进样,解析5 min。每份样品各3个平行。

定性和半定量分析:利用Flavour 2.0、NST 08质

谱库和相关文献^[17-19]对其定性,使用内标物环己酮对挥发性成分进行半定量,含量(w)单位为μg·g⁻¹。

1.7 数据统计分析

采用SPSS Statistics 26.0软件进行单因素方差分析(ANOVA),用SIMCA软件进行主成分分析,用Excel 2019进行图表绘制及数据整理。

2 结果与分析

2.1 不同成熟期琯溪蜜柚类黄酮含量的比较

表2所示为测定不同成熟期琯溪蜜柚不同部位4种主要类黄酮的含量,新圣草次苷、柚皮苷、新橙皮苷和野漆树苷含量都在果实成熟期呈下降的趋势,且受成熟阶段的影响显著($p < 0.05$)。从总体来看,黄皮层、囊衣、果肉、果汁在青色(未成熟)阶段总含量(w,后同)高达3 753.768、5 612.382、205.217、76.925 mg·kg⁻¹,白皮层和种子则在青转黄(半成熟)阶段类黄酮总含量最高,分别为5

表2 不同成熟期琯溪蜜柚类黄酮含量

Table 2 The contents of flavonoids in Guanxi pomelo fruits at different mature stages

(mg·kg⁻¹)

部位 Position	成熟阶段 Mature stage	新圣草次苷 Neoroicitrin	柚皮苷 Naringin	新橙皮苷 Neohesperidin	野漆树苷 Rhoifolin
黄皮层 Flavedo	青色(未成熟)Cyan (immature)	27.253±1.542 a	3 317.242±5.483 a	ND	409.273±5.426 b
	青转黄(半成熟)Green to yellow (semi mature)	22.447±0.127 b	2 913.203±0.356 b	ND	475.051±0.906 a
	黄色(成熟)Yellow (mature)	21.284±0.200 b	2 221.945±1.527 c	ND	245.457±1.575 c
显著性 Significance		0.000	0.000	—	0.000
白皮层 Albedo	青色(未成熟)Cyan (immature)	19.220±0.354 b	3 887.858±17.274 b	ND	123.898±4.400 b
	青转黄(半成熟)Green to yellow (semi mature)	22.465±0.551 a	5 121.553±23.417 a	ND	266.948±10.422 a
	黄色(成熟)Yellow (mature)	17.330±1.221 c	3 918.637±3.191 b	ND	99.071±3.995 c
显著性 Significance		0.010	0.000	—	0.000
果肉 Pulp	青色(未成熟)Cyan (immature)	1.078±0.017 b	146.541±0.077 a	3.374±0.089 a	54.224±0.106 a
	青转黄(半成熟)Green to yellow (semi mature)	1.265±0.009 a	102.329±0.685 b	2.175±0.132 b	51.669±0.181 b
	黄色(成熟)Yellow (mature)	1.154±0.061 b	55.137±1.333 c	1.395±0.026 c	17.101±0.010 c
显著性 Significance		0.020	0.000	0.000	0.000
果汁 Juice	青色(未成熟)Cyan (immature)	1.023±0.019 b	58.024±2.700 a	1.943±0.185 a	15.960±0.029 a
	青转黄(半成熟)Green to yellow (semi mature)	1.298±0.022 a	56.386±3.143 ab	1.894±0.010 a	14.588±0.167 b
	黄色(成熟)Yellow (mature)	1.259±0.022 a	50.771±2.147 b	0.926±0.147 b	10.971±0.113 c
显著性 Significance		0.000	0.037	0.000	0.000
囊衣 Capsule	青色(未成熟)Cyan (immature)	54.504±0.464 a	5 395.121±7.897 a	0.457±0.013 b	162.300±2.166 a
	青转黄(半成熟)Green to yellow (semi mature)	52.944±7.324 a	5 235.807±15.171 b	0.423±0.030 b	98.513±2.790 b
	黄色(成熟)Yellow (mature)	40.266±0.067 b	3 810.625±14.171 c	0.522±0.018 a	91.312±4.267 c
显著性 Significance		0.120	0.000	0.040	0.000
种子 Seed	青色(未成熟)Cyan (immature)	8.163±0.934 b	332.939±2.341 b	10.250±0.390 b	24.911±0.106 a
	青转黄(半成熟)Green to yellow (semi mature)	10.675±1.101 a	402.87±19.516 a	22.819±0.034 a	17.138±0.128 b
	黄色(成熟)Yellow (mature)	8.260±0.463 b	283.508±15.531 c	4.131±0.028 c	16.722±0.485 b
显著性 Significance		0.021	0.000	0.000	0.000

注:“ND”代表未检出;“—”代表无。不同小写字母表示在 $p < 0.05$ 差异显著。下同。

Note: “ND” means no detected; “—” means none. Different small letters indicate significant difference at $p < 0.05$. The same below.

成熟期琯溪蜜柚不同部位均有检出,并且7种酚酸均存在极显著差异($p < 0.001$)。根据在同一成熟阶段不同部位酚酸总含量来看,呈现以下规律:黄皮层>种子>囊衣>白皮层>果肉>果汁,且肉桂酸类酚酸含量显著高于苯甲酸类酚酸。根据在同一部位不同成熟期酚酸总含量来看,黄皮层、白皮层、囊衣、种子、果汁成熟期酚酸总含量都高于未成熟期,果肉酚酸总量随成熟期延长而下降。单一酚酸含量随成熟阶段变化趋势:原儿茶酸含量随着成熟期的延长,在成熟阶段比未成熟阶段都有所增长(除种子),在果汁半成熟阶段中最丰富,达 $3.509 \text{ mg} \cdot \text{kg}^{-1}$;对羟基苯甲酸含量变化呈现先上升后下降的趋势,对羟基苯甲酸黄皮层半成熟阶段中最丰富,达 $9.592 \text{ mg} \cdot \text{kg}^{-1}$;香草酸含量成熟阶段相较其他2个成熟阶段有所下降,在半成熟阶段种子最丰富,达

$11.057 \text{ mg} \cdot \text{kg}^{-1}$;咖啡酸含量在黄皮层和果汁呈先上升后下降的趋势,果肉和种子呈下降的趋势,白皮层呈上升的趋势,果汁中呈先下降后上升的趋势,咖啡酸在果肉中最丰富,达 $7.520 \text{ mg} \cdot \text{kg}^{-1}$ (未成熟);*p*-香豆酸含量在果肉和果汁呈下降的趋势,含量变化范围分别为 $2.953 \sim 7.433$ 、 $2.207 \sim 3.478 \text{ mg} \cdot \text{kg}^{-1}$,白皮层和种子呈上升的趋势,黄皮层呈先下降后上升的趋势,囊衣呈先上升后下降的趋势;阿魏酸含量在黄皮层和囊衣呈上升的趋势,果肉和果汁呈下降的趋势,白皮层呈先上升后下降的趋势,种子呈先下降后上升的趋势,阿魏酸在黄皮层中最丰富,达 $24.555 \text{ mg} \cdot \text{kg}^{-1}$ (成熟);芥子酸在黄皮层、白皮层、果肉、囊衣呈先上升后下降的趋势,果汁中呈上升的趋势,种子中呈下降的趋势。

2.3 不同成熟期琯溪蜜柚香豆素含量的比较

表4 不同成熟期琯溪蜜柚香豆素含量

Table 4 The contents of coumarins in Guanxi pomelo fruits at different mature stages		(mg·kg ⁻¹)				
部位 Position	成熟阶段 Mature stage	伞形花内酯 Umbelliferone	橙皮内酯水合物 Meranzin hydrate	6,7-二羟基香柠檬亭 Dihydroxybergamottin	橙皮油素 Auraptene	香柠檬亭 Bergamottin
黄皮层 Flavedo	青色(未成熟)Cyan (immature)	0.813±0.071 c	63.271±1.723 b	11.124±0.516 c	51.160±2.136 c	6.821±0.221 c
	青转黄(半成熟) Green to yellow (semi mature)	2.042±0.018 a	70.387±0.009 b	20.363±0.808 b	183.369±0.042 a	25.807±0.625 a
	黄色(成熟)Yellow (mature)	1.462±0.080 b	200.030±6.192 a	35.406±1.273 a	142.505±6.044 b	18.373±1.894 b
显著性 Significance		0.000	0.000	0.000	0.000	0.000
白皮层 Albedo	青色(未成熟)Cyan (immature)	0.080±0.004 b	0.366±0.028 b	0.472±0.010 b	1.450±0.090 a	0.294±0.050 ab
	青转黄(半成熟) Green to yellow (semi mature)	0.114±0.027 a	0.309±0.102 b	0.669±0.116a	0.654±0.010 b	0.359±0.051 a
	黄色(成熟)Yellow (mature)	0.090±0.003 ab	0.512±0.015 a	0.426±0.053 b	0.268±0.024 c	0.233±0.022 b
显著性 Significance		0.740	0.015	0.016	0.00	0.027
果肉 Pulp	青色(未成熟)Cyan (immature)	0.078±0.006 c	0.281±0.021 a	4.796±0.781 b	0.719±0.031 c	3.218±0.189 b
	青转黄(半成熟) Green to yellow (semi mature)	0.087±0.005 b	0.134±0.017 b	8.099±0.093 a	1.414±0.070 a	5.939±0.709 a
	黄色(成熟)Yellow (mature)	0.119±0.002 a	0.107±0.018 b	3.125±0.027 c	0.970±0.111 b	3.268±0.044 b
显著性 Significance		0.000	0.000	0.000	0.000	0.000
果汁 Juice	青色(未成熟)Cyan (immature)	0.048±0.003 a	0.521±0.016 a	5.864±0.076 a	1.198±0.020 a	0.623±0.002 a
	青转黄(半成熟) Green to yellow (semi mature)	0.018±0.001 b	0.299±0.036 b	1.617±0.113 b	0.529±0.012 b	0.469±0.006 b
	黄色(成熟)Yellow (mature)	0.060±0.001 a	0.211±0.009 c	1.185±0.229 c	0.443±0.013 c	0.377±0.018 c
显著性 Significance		0.864	0.000	0.000	0.000	0.000
囊衣 Capsule	青色(未成熟)Cyan (immature)	0.065±0.003 b	0.628±0.033 a	1.186±0.013 c	0.469±0.011 a	0.762±0.110 c
	青转黄(半成熟) Green to yellow (semi mature)	0.049±0.001 c	0.458±0.008 b	2.295±0.033 a	0.362±0.017 b	1.409±0.010 a
	黄色(成熟)Yellow (mature)	0.157±0.002 a	0.232±0.007 c	1.519±0.108 b	0.425±0.056 ab	0.946±0.079 b
显著性 Significance		0.000	0.000	0.000	0.000	0.022
种子 Seed	青色(未成熟)Cyan (immature)	0.379±0.015 a	ND	0.371±0.014 b	0.270±0.021 a	0.658±0.043 a
	青转黄(半成熟) Green to yellow(semi mature)	0.136±0.013 b	ND	0.413±0.019 a	0.119±0.004 b	0.359±0.016 b
	黄色(成熟)Yellow (mature)	0.065±0.004 c	ND	0.359±0.002 b	0.144±0.027 b	0.177±0.011 c
显著性 Significance		0.000	—	0.014	0.000	0.000

由表4可知,不同成熟阶段琯溪蜜柚成熟期间5种主要香豆素组分的含量存在极显著差异($p < 0.001$)。从同一成熟阶段不同部位香豆素总含量来看,未成熟期香豆素总量呈黄皮层>果肉>果汁>囊衣>白皮层>种子的规律;半成熟阶段和成熟阶段香豆素总含量呈相同的规律:黄皮层>果肉>囊衣>果汁>白皮层>种子。从同一部位不同成熟期香豆素总含量来看,黄皮层中香豆素总含量随着成熟阶段的延长而上升,白皮层、果汁、种子香豆素含量随着成熟阶段的延长而下降,果肉、囊衣香豆素总含量先上升后下降,其中黄皮层、白皮层、果肉、果汁、囊衣、种子含量变化范围分别为:133.189~397.775、1.528~2.662、7.589~15.673、2.276~8.253、3.110~4.574、0.745~1.679 $\text{mg} \cdot \text{kg}^{-1}$ 。单一香豆素在不同成熟期不同部位含量变化不尽相同,伞形花内酯在6个部位含量变化差异不大;橙皮内酯水合物在果肉、果汁、囊衣随着成熟期延长呈下降的趋势,黄皮层呈上升的趋势,含量变化范围为63.271~

200.030 $\text{mg} \cdot \text{kg}^{-1}$,种子中未检出;6,7-二羟基香柠檬亭在白皮层、果肉、囊衣、种子随着成熟阶段的延长呈现先上升后下降的趋势,黄皮层呈上升的趋势,果汁呈下降的趋势;橙皮油素在黄皮层和果肉随着成熟期延长呈先上升后下降的趋势,囊衣、种子、白皮层和果汁呈下降的趋势;香柠檬亭在黄皮层、白皮层、果肉、囊衣呈先上升后下降的趋势,果汁和种子呈下降的趋势。

2.4 不同成熟期琯溪蜜柚类柠檬苦素含量的比较

表5所示为不同成熟期琯溪蜜柚不同部位类柠檬苦素的含量,显著差异分析显示琯溪蜜柚在成熟过程中的柠檬苦素、诺米林和黄柏酮含量的差异非常显著($p < 0.001$)。由表5可知,种子中类柠檬苦素含量最丰富,黄柏酮只在种子检出,其中种子中柠檬苦素、诺米林和黄柏酮随着成熟期的延长,含量不断上升,分别增长了47.11%、60.90%、86.47%。黄皮层、白皮层、囊衣、果肉、果汁中柠檬苦素和诺米林在成熟期间急剧下降,柠檬苦素分别下降了25.99%、

表5 不同成熟期琯溪蜜柚类柠檬苦素含量

Table 5 The contents of limonoids in Guanxi pomelo fruits at different mature stages

($\text{mg} \cdot \text{kg}^{-1}$)

部位 Position	成熟阶段 Mature stage	柠檬苦素 Limonin	诺米林 Nomilin	黄柏酮 Obacunone
黄皮层 Flavedo	青色(未成熟)Cyan (immature)	488.366±3.825 a	20.833±1.741 a	ND
	青转黄(半成熟)Green to yellow (semi mature)	377.928±0.065 b	12.284±0.107 b	ND
	黄色(成熟)Yellow (mature)	361.457±1.298 c	11.370±0.131 b	ND
显著性 Significance		0.000	0.000	—
白皮层 Albedo	青色(未成熟)Cyan (immature)	9.230±0.245 a	7.546±0.073 a	ND
	青转黄(半成熟)Green to yellow (semi mature)	2.820±0.118 b	5.328±0.044 b	ND
	黄色(成熟)Yellow (mature)	0.515±0.032 c	1.918±0.044 c	ND
显著性 Significance		0.000	0.000	—
果肉 Pulp	青色(未成熟)Cyan (immature)	83.375±0.212 a	26.892±0.436 b	ND
	青转黄(半成熟)Green to yellow (semi mature)	47.948±0.564 b	31.966±1.339 a	ND
	黄色(成熟)Yellow (mature)	48.944±0.202 c	15.188±0.514 c	ND
显著性 Significance		0.000	0.000	—
果汁 Juice	青色(未成熟)Cyan (immature)	49.509±0.207 a	13.969±0.610 a	ND
	青转黄(半成熟)Green to yellow (semi mature)	25.203±0.421 b	14.765±0.992 a	ND
	黄色(成熟)Yellow (mature)	16.441±0.080 c	11.598±0.030 b	ND
显著性 Significance		0.000	0.001	—
囊衣 Capsule	青色(未成熟)Cyan (immature)	342.338±0.447 a	427.095±0.094 a	ND
	青转黄(半成熟)Green to yellow (semi mature)	331.835±0.061 b	412.882±0.474 b	ND
	黄色(成熟)Yellow (mature)	199.764±1.045 c	195.752±0.392 c	ND
显著性 Significance		0.000	0.000	—
种子 Seed	青色(未成熟)Cyan (immature)	576.389±2.417 c	439.280±0.159 c	1.637±0.078 c
	青转黄(半成熟)Green to yellow (semi mature)	689.443±2.918 b	652.332±1.734 b	5.265±0.467 b
	黄色(成熟)Yellow (mature)	1 089.852±2.365 a	1 123.570±5.726 a	12.122±0.030 a
显著性 Significance		0.000	0.000	0.000

94.47%、41.65%、30.41%、66.79%,诺米林分别下降了45.42%、74.57%、54.17%、43.51%、21.41%。

2.5 不同成熟期琯溪蜜柚果皮香气物质含量的比较

由表6可知,利用GC-MS在3个成熟阶段琯溪蜜柚黄皮层共检测到51种香气物质,其中在青

色(未成熟)阶段、青转黄(半成熟)阶段、黄色(成熟)阶段中分别检测到34、25、42种;随着成熟期的延长,琯溪蜜柚黄皮层中共测得香气物质总含量呈增长的趋势,分别为7 896.769、8 664.805和10 380.399 $\mu\text{g}\cdot\text{g}^{-1}$ 。在3个成熟阶段琯溪蜜柚果皮所

的香气物质中,单萜类物质和倍半萜类物质含量较

表6 不同成熟期琯溪蜜柚果皮挥发物成分含量

Table 6 The contents of volatile compounds in Guanxi pomelo flavedo at different mature stages ($\mu\text{g}\cdot\text{g}^{-1}$)

成熟阶段 Mature stage	挥发性成分物质分类 Classification of volatile compounds							合计 Sum
	单萜类 Monoterpenes	倍单萜类 Sesquiterpenes	醛类 Aldehydes	醇类 Alcohols	酯类 Esters	酮类 Ketones	其他 Others	
青色(未成熟)Cyan (immature)	7 131.510(7)	297.979(8)	47.717(3)	375.213(10)	17.546(4)	12.916(2)	ND	7 896.769(34)
青转黄(半成熟) Green to yellow (semi mature)	7 829.534 (5)	454.985(9)	102.937 (3)	270.889 (5)	1.606(1)	1.995(1)	2.859(1)	8 664.805(25)
黄色(成熟)Yellow (mature)	9 171.815 (9)	502.440 (11)	189.838(9)	464.719(8)	27.746(3)	20.632(1)	3.209(1)	10 380.399(42)

注:“ND”代表未检出。Note: “ND” means no detected.

高,其次为醇类和醛类物质。黄色(成熟)阶段的单萜类、倍单萜类、醛类、醇类、酯类、酮类物质含量均高于其他2个成熟期,其中柠檬烯占比高达总含量的78.48%。

根据香气活度值理论,食品中香气浓度高而阈值低的成分很可能是食品的特征香气,物质浓度高不一定对样品的香气有很大的贡献(如柠檬烯),而含量低的物质也有可能对整体香气有大的影响。OAV的水平可以反映挥发性风味成分对样品特征香气的贡献,以往的研究表明OAV越高,化合物的个体贡献和影响越大。查阅相关文献得到香气物质的香气阈值^[19-21],结合GC-MS的定量结果,计算所得OAV值。表7所示为3个成熟阶段琯溪蜜柚黄皮层中OAV > 1的香气物质总共35种,随着成熟期延长,OAV值也呈上升的趋势,其中醇类、醛类、酮类、酯类、单萜类和倍半萜类分别有9、9、2、4、7和4种。黄色(成熟)阶段包含最多的特征香气物质,为30种,其次为未成熟,包含24种,半成熟最少,为15种。

2.6 主成分分析

主成分分析(principal component analysis, PCA)是一种利用降维技术将多个变量转化为主成分的统计方法,是最重要的降维方法之一^[22]。水平和垂直坐标为主成分,每个向量代表原始特征,向量在主成分上的投影可以表示两者之间的相关程度^[23]。笔者在本研究中对3个成熟阶段的琯溪蜜柚果汁样品的选取功能成分共18个进行主成分分析,

F1主成分的贡献率为70.3%,F2主成分的贡献率为29.7%,2个主成分的累计贡献率已经达到100%。青色(未成熟)、青转黄(半成熟)、黄色(成熟)分别位于第2象限、第4象限、第1象限,表明3个成熟阶段琯溪蜜柚果汁样品可以很好地区分,其不同成熟期的功能成分具有显著差异。

通过双标图(bi-plot)同时显示得分图与载荷图之间的关系,结合分析不同成熟期琯溪蜜柚综合品质。如图2,其中芥子酸、伞形花内酯位于第1象限,说明对主成分1和主成分2有较大的影响。阿魏酸、6,7-二羟基香柠檬亭、柠檬苦素、橙皮内酯水合物等物质位于第2象限,说明与主成分2呈正相关。柚皮苷、新橙皮苷、野漆树苷、诺米林、对羟基苯甲酸等物质位于第3象限,说明对主成分1和主成分2呈负相关。原儿茶酸、香草酸、新圣草次苷等物质位于第4象限,说明与主成分1有较大的相关性。主成分1和主成分2对3个成熟阶段琯溪蜜柚区分明显,青色(未成熟)位于第2象限,与柠檬苦素、阿魏酸、6,7-二羟基香柠檬亭等物质有较大的相关性,青转黄(半成熟)阶段与新圣草次苷、香草酸、原儿茶酸相关性显著,黄色(成熟)阶段与芥子酸、伞形花内酯相关性显著。综上所述,不同成熟期对功能成分的影响大小高低不一致,不同成熟期琯溪蜜柚功能成分具有显著差异。

3 讨论

笔者在本研究中对不同成熟阶段的琯溪蜜柚不

表 7 不同成熟期琯溪蜜柚果皮香气物质的 OAV

Table 7 OAV of volatile compounds in Guanxi pomelo flavedo at different mature stages

特征香气物质 Compound	阈值 Odor threshold/ ($\mu\text{g}\cdot\text{g}^{-1}$)	香味描述 Odor description	OAV		
			青色(未成熟) Cyan (immature)	青转黄(半成熟) Green to yellow (semi mature)	黄色(成熟) Yellow (mature)
α -蒎烯 α -pinene	0.190	松香、树脂香 Woody, terpenic	—	—	940.024
β -蒎烯 β -pinene	1.500	松香、树脂香 Woody, terpenic	230.011	312.120	353.816
罗勒烯 Ocimene	0.034	青草味, 树脂香 Green, terpenic	3 951.370	5 872.744	5 519.833
水芹烯 α -phellandrene	0.036	黑胡椒香、薄荷香 Bitter black pepper, sweet lotus	—	—	1 389.057
γ -松油烯 γ -terpinene	0.260	柠檬味 Lemon	67.688	—	81.635
α -松油烯 α -terpinene	0.080	柑橘香, 柠檬香 Citrus, lemon	26.482	29.166	33.291
异松油烯 Terpinolene	0.410	酸橙味, 树脂香 Lime, terpenic	103.818	123.018	128.974
β -榄香烯 β -elemene	0.150	木香味 Woody	—	716.174	—
α -石竹烯 α -caryophyllene	0.160	木香, 青草味 Woody, green	—	—	124.172
巴伦西亚橘烯 Valencene	10.500	柑橘香 Citrus	—	—	6.873
金合欢烯(Z)- β -farnesene	0.160	—	7.705	11.342	33.513
己醛 Hexenal	0.100	青草味, 香脂味 Green, terpenic	—	—	118.200
反式-2-己烯醛 <i>Trans</i> -2-hexenal	0.050	—	—	—	257.897
苯甲醛 Benzaldehyde	0.350	果味, 杏仁 Fruity, almond	—	—	12.486
香茅醛 Citronellal	0.035	花香 Floral	—	—	92.844
月桂醛 Lauric aldehyde	0.055	柑橘香 Citrus	—	—	35.099
十一醛 Undecanal	0.010	—	691.622	1 009.143	963.794
顺式-柠檬醛(Z)-citral	0.085	柠檬味 Lemon	175.932	421.032	614.364
反,反-2,4-癸二烯醛 <i>Trans, trans</i> -2,4-decadienal	0.010	—	2 584.656	5 705.746	9 084.033
柠檬醛 Citral	0.041	柠檬味 Lemon	—	—	73.773
顺式-3-己烯醇 <i>Cis</i> -3-hexenol	3.625	青草味 Green	16.578	—	—
辛醇 Octanol	0.875	肥皂味 Soap	29.796	46.672	42.411
芳樟醇 Linalol	0.037	花香、木香 Floral, woody	2 193.269	2 273.082	2 460.214
正壬醇 1-nonanol	1.000	青草味, 香脂味 Green, terpenic	1.612	—	2.382
4-萜烯醇 4-Carvomenthenol	6.400	木香 Woody	1.786	1.846	1.786
α -松油醇 α -terpineol	15.000	木质, 柑橘 Woody, citrus	3.087	—	2.956
香叶醇 <i>Cis</i> -geraniol	0.010	玫瑰, 天竺葵味 Rosy, Geranium	141.972	—	13 720.689
橙花醇(Z)-geraniol	0.680	甜花香, 柠檬香 Fresh, lemon	191.568	190.640	202.276
正癸醇 1-decanol	0.180	—	76.960	—	—
乙酸冰片酯 Borneol acetate	0.075	松木香, 樟脑香 Woody	21.155	21.412	—
乙酸辛酯 Octyl acetate	0.300	—	23.169	—	11.345
乙酸香叶酯 Geranyl acetate	0.150	松木香, 玫瑰香 Woody, rosy	39.767	—	124.093
乙酸橙花酯 Nerol acetate	2.000	玫瑰花香 Rosy	1.522	—	2.865
诺卡酮 Nootkatone	0.180	柚子味 Grapefruit	38.615	11.083	114.623
香芹酮 Carvone	0.025	留兰香味, 花香 Cooling, spearmint	238.601	—	—

同部位的功能成分综合评价,结果显示:不同成熟期琯溪蜜柚4种主要类黄酮的含量都在成熟期呈下降的趋势,柚皮苷和野漆树苷是琯溪蜜柚果实占主导地位的两种黄酮类化合物。胡阳等^[24]对不同生长期琯溪蜜柚中柚皮苷含量进行测定,结果表明随着成熟期的延长,果实柚皮苷含量也呈下降趋势;吴雪霞等^[25]的研究表明茄子生长发育过程中类黄酮和总酚

含量均呈现下降的趋势,这与琯溪蜜柚果实中的规律相似。不同成熟期琯溪蜜柚的不同部位7种酚酸的含量存在显著差异,酚酸随成熟期变化趋势不尽相同,根据在同一成熟阶段不同部位酚酸总含量来看,呈现以下规律:黄皮层>种子>囊衣>白皮层>果肉>果汁,且肉桂酸类酚酸含量显著高于苯甲酸类酚酸,这与汤春甫^[26]的研究结果类似;根据在同一

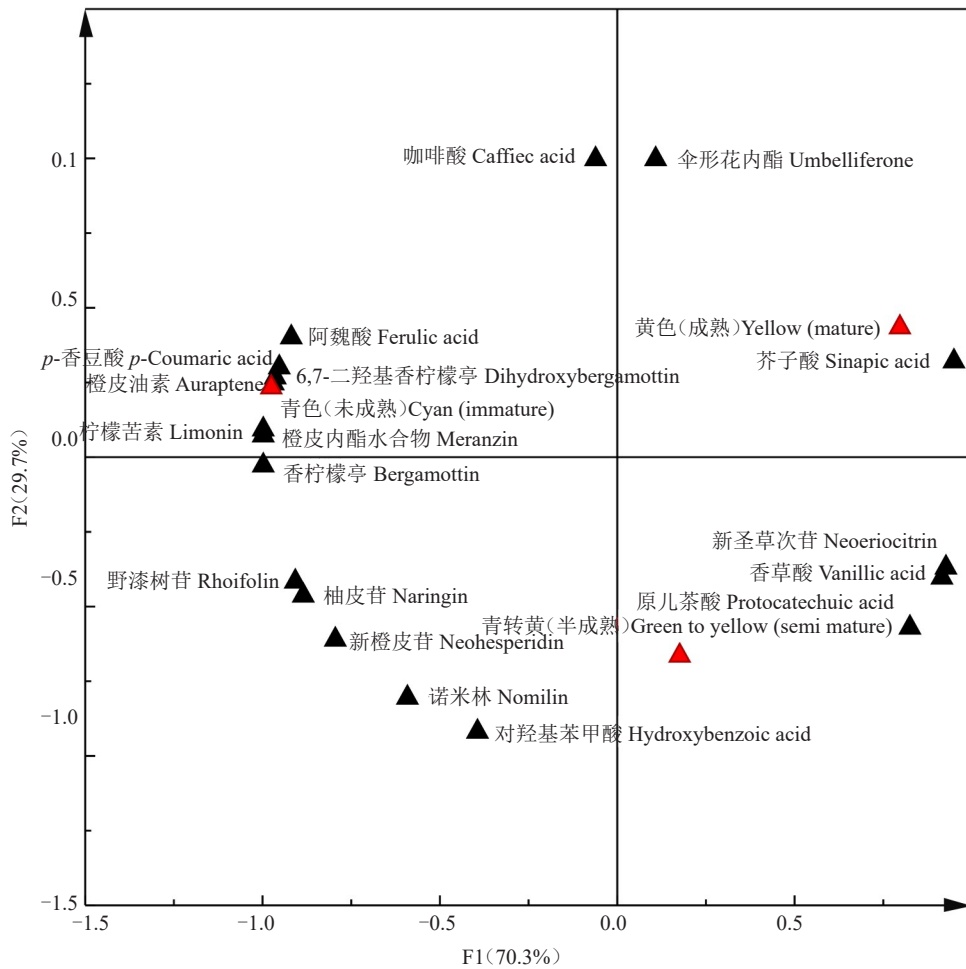


图2 基于不同成熟期琯溪蜜柚果汁功能成分的主成分分析

Fig. 2 Principal component analysis based on functional components in Guanxi pomelo juice at different mature stages

部位不同成熟期酚酸总含量来看,黄皮层、白皮层、囊衣、种子、果汁成熟期酚酸总含量高于未成熟期,果肉酚酸总含量随成熟期延长而下降,符合徐贵华等^[27]采用 HPLC-PDA 测定了不同成熟期南丰蜜橘中酚酸的含量,结果显示,果肉中酚酸含量随成熟度提高而减少。在不同成熟阶段不同部位香豆素总含量来看,黄皮层中香豆素含量最丰富,可作为提取香豆素的良好来源。类柠檬苦素随着成熟期延长而下降,其中黄皮层和囊衣下降最明显,其次是果肉、果汁、白皮层;种子中类柠檬呈现相反的趋势,且种子中类柠檬苦素最丰富,文献表明^[28-29],种子中的诺米林和柠檬苦素在10月和11月显示出最高浓度,果实完全成熟后,种子中柠檬苦素浓度在12月达到最大值,并且在种子中远高于果皮,与本文研究结果相同。3个成熟阶段琯溪蜜柚黄皮层共检测到51种香气物质,随着成熟期的延长挥发性成分总含量呈增

长的趋势。对3个成熟阶段的琯溪蜜柚果汁样品的18个功能成分进行主成分分析,结果表明不同成熟阶段琯溪蜜柚功能成分存在显著差异。

4 结论

对不同成熟期琯溪蜜柚果实不同部位功能成分(类黄酮、酚酸、香豆素、类柠檬苦素和挥发性成分)的组成进行比较分析,结果表明,成熟度对琯溪蜜柚不同部位功能成分有显著性影响。同一部位,大多数类黄酮、酚酸、香豆素和类柠檬苦素含量随着成熟期延长呈现下降的趋势,少部分随着成熟期延长呈现上升的趋势;同一成熟期,黄皮层中类黄酮、酚酸和香豆素含量较高,种子中类柠檬苦素含量最丰富,分别可作为相应功能成分的天然来源;挥发性成分总含量随着成熟期的延长呈增长的趋势。基于本研究结果,笔者可选择合适的采摘期,进而充分利用某

一成熟期某一部位的有效成分,比如未成熟时囊衣部位的柚皮苷、成熟时黄皮层部位的橙皮内酯水合物和成熟时种子部位的类柠檬苦素都是相对丰富的。这为不同成熟阶段的琯溪蜜柚采后资源综合利用及工业化提供方向,同时也为开发医药以及保健品提供理论指导。

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