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# 基于高光谱成像技术的残留氯吡脒猕猴桃无损识别

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**摘要:** 利用近红外高光谱技术,依据施药猕猴桃和空白猕猴桃中氯吡脒残留量不同、有机成分含量不同,进行了2类猕猴桃的无损识别技术研究。首先进行2类猕猴桃的田间试验,获得具有代表性的猕猴桃样品,然后通过近红外高光谱设备扫描样品,获得每个样品的近红外高光谱图像并输出每个样品平均反射光谱值,最后进行2类猕猴桃的无损识别。结果表明,PCA-LDA(主成分分析-线性判别分析)和支持向量机(SVM)2个数学模型的正确识别率都是94.4%;施药样品氯吡脒的残留量为0.03 mg/kg,空白样品的氯吡脒残留量为0 mg/kg;总糖、总酚、维生素C、可滴定酸、17种氨基酸、8种微量元素等有机成分含量存在一定的差异。因此,采用近红外高光谱技术进行植物生长调节剂猕猴桃的无损识别是可行的。

**关键词:** 猕猴桃; 氯吡脒; 高光谱; 无损识别

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## Non-destructive Identification of Kiwifruit with Forchlorfenuron Residue Based on Near Infrared Hyperspectral Imaging Technology

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**Abstract:** Based on the difference of forchlorfenuron residue and organic component, non-destructive identification method of treated and control kiwifruits was studied using hyperspectral imaging system. Firstly, the representative samples were got by field experiment, and the hyperspectral image was got. The instrument parameters of hyperspectral image were as follows: exposure time 10 ms, scanning speed 20 mm/s and object distance 20 mm/s. Secondly, the spectral regions of 1 040 ~ 1 120 nm and 1 335 ~ 1 500 nm were selected, totally 116 wave bands were produced, and the average hyperspectral value of each sample was calculated. Finally, all data of hyperspectral image were calculated by Matlab, and the non-destructive identification method of two types of kiwifruit was established. The residue of forchlorfenuron was determined by HPLC, organic component in kiwifruit was analyzed according to the national standard. The results showed that correct identification rate of PCA-LDA and support vector machine (SVM) mathematical models was 94.4%; forchlorfenuron residue level of treated kiwifruit and control kiwifruit were 0.03 mg/kg and 0 mg/kg, respectively; the content of organic components were different, such as total sugar, total phenols, vitamin C, titratable acid, 17 kinds of amino acids and eight trace elements. Therefore, the high spectrum technology was an efficient method for non-destructive identification of PGR (plant growth regulation) kiwifruit.

**Key words:** kiwifruit; forchlorfenuron; high spectrum; non-destructive identification

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## 引言

氯吡啶,1-(2-氯-4-吡啶)-3-苯基脲,是一种植物生长调节剂,广泛应用于果蔬生产中<sup>[1-4]</sup>。近些年,植物生长调节剂在猕猴桃生产中的滥用对猕猴桃的品质造成了一定的影响<sup>[5-6]</sup>,同时威胁到猕猴桃产品的安全性。目前,氯吡啶的残留检测方法有高效液相色谱法<sup>[7-8]</sup>、液相色谱-串联质谱法<sup>[9-10]</sup>、酶联免疫法<sup>[11-14]</sup>等。这些检测方法费用高,样品前处理程序多,为破坏性检测,不能实现工业化连续生产过程中的在线检测。

高光谱技术是一种将图像和光谱相结合的技术,近年来广泛应用于农业生产和农产品的无损检测。薛龙等<sup>[15]</sup>利用高光谱技术研究了脐橙表面农药残留的快速检测;索少增等<sup>[16]</sup>利用高光谱技术探讨了梨表面的毒死蜱和炔螨特2种农药的检测方法;王晓<sup>[17]</sup>以赣南脐橙为研究对象,利用高光谱技术进行了农药残留的无损检测研究;刘民法等<sup>[18]</sup>进行了长枣表面农药残留无损检测的研究。高光谱技术应用于农产品品质研究也比较多,如农产品的分类和质量管理<sup>[19]</sup>、葡萄皮中多酚含量估算<sup>[20]</sup>、苹果品质检测<sup>[21]</sup>、玉米中黄曲霉素B<sub>1</sub>的检测<sup>[20]</sup>、肉的品质检测<sup>[22]</sup>、桑叶品质检测<sup>[23]</sup>以及水果表面的污染和机械损伤<sup>[24-27]</sup>。

本文选取成熟的施药猕猴桃和空白猕猴桃样品进行近红外高光谱无损检测的模糊识别研究,然后进行有机成分和氯吡啶残留的检测试验,确定2类样品是否存在有机成分的差别,以期对猕猴桃工业化在线无损检测提供基础数据。

## 1 试验

### 1.1 材料与仪器设备

#### 1.1.1 原料

施药猕猴桃,空白猕猴桃。

#### 1.1.2 试剂

氯吡啶(99%),色谱乙腈,色谱甲酸,西格玛公司;色谱柱,Inertsil ODS-SP,4.6 mm×250 mm,5 μm,日本岛津公司;GCB(石墨化碳),PSA(N-丙基乙二胺),天津博纳艾杰尔科技有限公司;无水醋酸钠,无水硫酸镁(分析纯),成都市科龙化工试剂厂。

#### 1.1.3 仪器设备

L-8900型全自动氨基酸分析仪,日本日立公司;HPLC-20AD型液相色谱仪,日本岛津公司;SOLAASR型原子吸收仪,美国热电公司;高光谱采集系统,北京汉光卓立公司,包括N17E型近红外光谱仪(ImSpector,Finland)、OPCA05G型CCD面阵探

测器(日本滨松),光谱波长范围900~1700 nm,光谱分辨率5 nm;HC-3018R型高速冷冻离心机,安徽中科中佳科学仪器有限公司;Milli-Q-Plus型超纯水系统,美国Millipore公司。

### 1.2 试验方法

#### 1.2.1 猕猴桃样品的制备

试验在陕西佰瑞猕猴桃研究院有限公司进行。样品制备参照NY/T 788—2004。氯吡啶的田间施药浓度(质量比)建议为5~20 mg/kg,通过调研发现种植户在施药过程中施药浓度远在这个范围之外。田间试验设计了0、5、20、40、60、80 mg/kg,共计6水平,通过试验发现5~20 mg/kg的施药浓度对于猕猴桃果实具有一定的膨大作用,但是达不到市售猕猴桃的尺寸和质量,且检测不到母药残留,2类猕猴桃理化成分未发现变化;当施药浓度为40 mg/kg时,膨大效果明显,但果实成熟后没有检测到母药残留,2类猕猴桃理化成分未发现变化;当施药浓度为60 mg/kg时检测到猕猴桃有母药残留0.03 mg/kg(NY 1500.75.1—2009中规定猕猴桃残留量为0.05 mg/kg),猕猴桃理化成分变化显著,因此选用施药浓度为60 mg/kg的猕猴桃作为无损识别的研究对象。即如果猕猴桃市场存在母药残留超标的猕猴桃,就能够采用此方法进行无损检测。选取长势大小基本一致的7年生海沃德50株,空白区和施药区各25株。氯吡啶施药浓度60 mg/kg,施药时间为盛花后15 d,浸泡时间为5 s,空白样品采用清水浸泡5 s,成熟后采收,每个处理各选100 kg果实备用。

#### 1.2.2 近红外高光谱图像数据采集及其预处理

高光谱图像采集参数设定:曝光时间10 ms,扫描速度为20 mm/s,物距为200 mm。待高光谱光源稳定后,对获得的图像进行黑白标定,以消除部分噪声影响。

近红外高光谱图像处理步骤:波段比处理(波段比计算可增强波段间的差异性,除去猕猴桃图像的阴影)、掩膜、滤波、应用掩膜、感兴趣区选取、计算并输出每个猕猴桃的近红外平均光谱值。

#### 1.2.3 数学模型

##### (1) PCA-LDA数学模型建立

2类果实光谱数据随机各挑选126个数据作为训练集,54个作为验证集,将光谱数据导入软件Matalab 2013a,采用PCA-PDA数学分析方法计算分析。主成分分析(Principal component analysis, PCA)是一种多元统计方法,可以降低数据的维数并表达原始数据的信息。线性判别分析(Linear discriminant analysis, LDA)更适合于分类鉴别。

## (2) 支持向量机(SVM)

支持向量机(Support vector machine, SVM)是一种新的通用的学习方法,适合应用于模式分类问题。

### 1.2.4 猕猴桃果实理化指标测定

高光谱试验完成后,称果实质量,测果形指数,然后每个果实按照四分法取样,打浆匀浆,取样测试其理化指标,包括:可滴定酸(GB/T 12456—2008)、总糖(SB/T 10203—94)、总酚(福林酚法)<sup>[28]</sup>、维生素C(GB/T 5009.86—2003)、氨基酸(全自动氨基酸分析仪)<sup>[29]</sup>、微量元素<sup>[30]</sup>。结果如表1~3所示。

表1 2类猕猴桃果实理化指标对比

Tab.1 Comparison of physicochemical indexes for two types of kiwifruit

参数	空白样品	施药样品
果型指数	1.19	1.27
质量/g	77.35	179.40
总糖质量分数/%	14	13
可滴定酸质量分数/%	1.350	1.506
总酚质量浓度/(mg·L <sup>-1</sup> )	905.5356	639.6170
维生素C质量浓度/(μg·mL <sup>-1</sup> )	888.2450	636.3727

表2 2类猕猴桃中微量元素含量的对比

Tab.2 Comparison of eight trace elements for two types of kiwifruit

样品	B	Mg	K	Ca	Mn	Fe	Cu	Zn
空白样品	2.40	138.45	4157.89	191.18	1.50	1.96	0.69	0.48
施药样品	2.21	109.98	3528.31	175.72	0.82	1.58	0.55	0.52

表3 2类猕猴桃中氨基酸含量的对比

Tab.3 Comparison of 17 kinds of amino acids for two types of kiwifruit

序号	名称	空白样品	施药样品
1	天冬氨酸	0.7240415	0.6360496
2	苏氨酸	0.3243381	0.2786498
3	丝氨酸	0.2603135	0.2300876
4	谷氨酸	0.7425779	0.6532411
5	脯氨酸	0.4477694	0.3959982
6	甘氨酸	0.3530136	0.3038750
7	丙氨酸	0.2867958	0.2424966
8	胱氨酸	0.0769423	0.0692791
9	缬氨酸	0.2788655	0.2458375
10	蛋氨酸	0.0200256	0.0123545
11	异亮氨酸	0.2716008	0.2334749
12	亮氨酸	0.3092738	0.2690421
13	络氨酸	0.2138991	0.1908624
14	苯基丙氨酸	0.2126913	0.1852087
15	赖氨酸	0.3296622	0.2891703
16	组氨酸	0.1036513	0.1011619
17	精氨酸	0.3683415	0.2015280

### 1.2.5 氯吡脞的残留测定

采用高效液相色谱法,液相条件:流动相,乙腈与水(0.1%甲酸)体积比55:45;柱温,30℃;流速:1 mL/min;色谱柱,Intersil ODS-SP 4.6~250 mm,5 μm。

样品前处理:准确称取15 g匀浆样品,置入50 mL离心管中,加入15 mL 1%醋酸乙腈,用力震荡或者涡旋1 min;加6 g无水MgSO<sub>4</sub>、1.5 g无水醋酸钠,涡旋1 min;3 800 r/min条件下离心6 min;将上层提取液4 mL转入10 mL离心管中;加入50 mg PSA、50 mg GCB、150 mg无水MgSO<sub>4</sub>,涡旋1 min;取

上清液2 mL移入样品瓶中,用氮气吹干,再用1 mL甲醇复溶;上样分析。

## 2 结果与讨论

### 2.1 氯吡脞残留量测定

施药样品的氯吡脞残留量为0.03 mg/kg,空白样品的氯吡脞残留量为0 mg/kg。

### 2.2 猕猴桃果实的有机成分

将2类猕猴桃有机成分进行比较,如果2类猕猴桃的有机成分含量差异较小或者没有差异,说明近红外光谱值贡献较小;反之,近红外光谱值贡献较大。

由表1可以看出,施药样品的果实质量是空白样品的2.3倍,总糖、总酚、维生素C含量均低于空白样品,可滴定酸质量分数大于空白样品。糖酸比是影响猕猴桃口感风味的重要指标,由表1明显可以看出,施药样品的糖酸比小于空白样品。6种理化指标差异显著。由表2可以得出2种猕猴桃的微量元素差异比较大。由表3可知施药样品中17种氨基酸的含量均低于空白样品,差异显著。因此通过近红外高光谱技术可以实现施药果实的判断与识别。

### 2.3 近红外高光谱无损识别

#### 2.3.1 高光谱图像的预处理

高光谱采集系统所得到的图像周围有杂点和阴影,在整个猕猴桃平均光谱输出之前,对其进行一系列图像处理,以去除干扰,获得理想的光谱值。由图1可以看出,图1a存在阴影,如果直接选取感兴趣区域并提取计算平均波长,可能会影响到平均波长的数据,甚至失真。经过波段比处理后得到图1b,波段比Band 65(1 077.590 0 nm): Band 175

(1 442.790 0 nm), 阴影部分基本去除, 创建掩膜, 发现有杂点, 采用滤波去除杂点, 然后应用掩膜得到图 1e, 选择整个猕猴桃图像为感兴趣区, 计算输出每个果实的近红外平均反射波长。空白样品和施药

样品的图像前处理步骤相同。根据图 2, 选择 PCA-LDA 模型的波长数据, 去除样品波长紧密的波段, 选取 1 040 ~ 1 120 nm 和 1 335 ~ 1 500 nm 范围内共 116 个波长的 2 类猕猴桃平均光谱值。

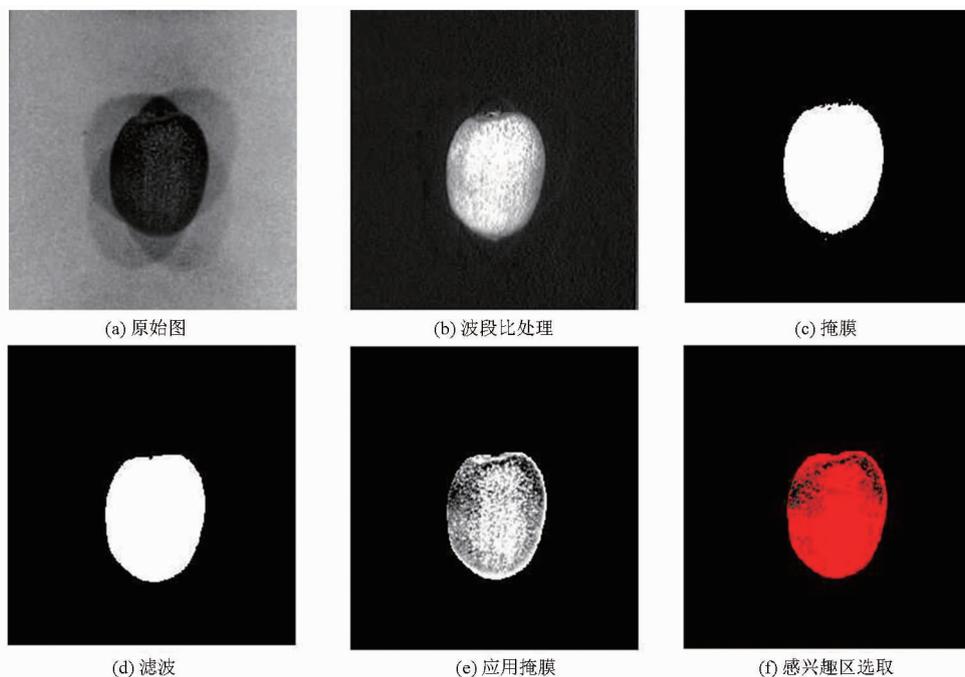


图 1 近红外高光谱图像处理

Fig. 1 Image process of near infrared hyperspectral imaging

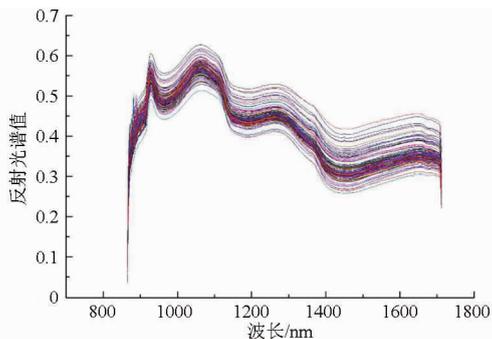


图 2 2 类猕猴桃样品近红外反射光谱图

Fig. 2 Reflectance spectra of two types of kiwifruit

### 2.3.2 数学模型建立

#### (1) PCA-LDA 数学模型

根据 Matlab 2013a 的函数及运算命令, 首先采用主成分分析对所有样品的 116 个波长进行降维, 由图 3 可以得出前 3 个主成分对模型整体数据的贡献较大, 考虑到数据的保真, 试验选取了前 10 个主成分作为线性判别分析(LDA)建模的数据。LDA 识别模型选取 2 类猕猴桃各 126 个, 将其平均光谱值作为建模数据, 另外再各取 54 个猕猴桃作为验证。通过 LDA 识别运算, 得出 54 个空白猕猴桃的正确识别率为 100%, 54 个施药猕猴桃正确识别 48 个, 误判 6 个, 识别率为 88.9%, 整体正确识别率为 94.4%。模型误判的主要原因分析有 2 个: 施药猕

猴桃表面不规整或者绒毛密度不一致, 影响到采集的光谱数据以及模型本身存在的误差。

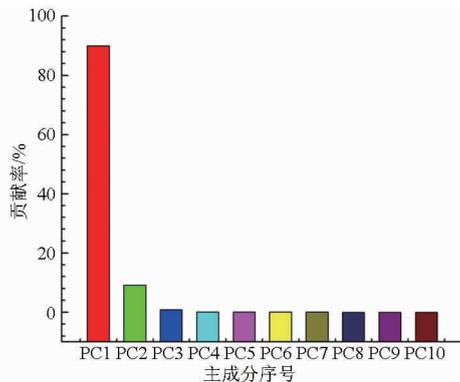


图 3 前 10 个主成分的贡献率

Fig. 3 Contribution rate of the first 10 principal components

#### (2) 支持向量机

根据 Matlab 2013a 的函数及运算命令, 2 类猕猴桃各取 126 个猕猴桃光谱数据用于建模, 另外再各取 54 个作为验证。通过支持向量机函数运算得出空白猕猴桃的正确识别率为 100%, 54 个施药猕猴桃正确识别 48 个, 误判 6 个, 识别率为 88.9%, 整体正确识别率为 94.4%。模型误判的主要原因同 PCA-LDA。

### 3 结束语

采用高光谱技术, 通过一系列的图像处理, 得到

2 类样品近红外高光谱的平均反射光谱值,然后通过 Matlab 2013a 的函数及运算命令建立 PCA-LDA 和支持向量机(SVM)2 个数学模型,正确识别率都是 94.4%,计算结果保持一致,说明 2 个数学模型运行稳定,数据可靠。通过化学计量的方法,得到了

2 类猕猴桃的有机成分差别和氯吡啶母药含量的差别,进一步确认近红外高光谱技术是适合于氯吡啶猕猴桃的一种无损快速识别的分析技术,为猕猴桃工业化连续化生产过程中的品质分析和安全监测提供了一种新方法。

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