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苹果中苯肽胺酸残留及消解

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摘 要：【目的】评价20%苯肽胺酸水剂在苹果上应用的残留水平，确保20%苯肽胺酸水剂在苹果上的安全使用。**【方法】**采用田间试验，使用QuEChERS方法进行样品前处理，液相色谱-串联质谱法进行定量分析，对苯肽胺酸在苹果中的消解动态及最终残留进行研究。**【结果】**苯肽胺酸在苹果中的半衰期为3.7~5.8 d，药后14 d消解86.7%以上。最终残留试验表明，20%苯肽胺酸水剂按施药剂量250 mg·kg^{-1}（有效成分）和375 mg·kg^{-1}（有效成分），喷药1次，收获时苯肽胺酸在苹果中的最终残留量均＜0.01 mg·kg^{-1}。**【结论】**20%苯肽胺酸水剂在苹果上按推荐剂量使用是安全的。

关键词：苹果；苯肽胺酸；残留；消解

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Residues and dissipation dynamics of phthalanilic acid in apple

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Abstract:【Objective】Phthalanilic acid is a new type of plant growth regulator. In recent years, it has been widely used in fruit trees and vegetables. To evaluate the residual level and dissipation dynamics of 20% aqueous solutions of phthalanilic acid in apples, and ensure the safe use of 20% aqueous solutions of phthalanilic acid in apples, the residue test of 20% aqueous solutions of phthalanilic acid on apples was carried out.【Methods】Field trials of 20% aqueous solutions of phthalanilic acid were carried out in research stations at three different locations, including Penglai of Shandong province, Huaibei of Anhui province and Changshun of Guizhou province, in 2017 and 2018. Both dissipation kinetics and residue determination experiments were carried out. The field trials involved a total of 4 treatments that consisted of 3 treatments at different doses and a blank control. There were three two-trees repeated for each treatment. The apple were applied with pesticides during young fruit for the degradation dynamic trials and the final residue trials. The 20% aqueous solutions of phthalanilic acid were evenly sprayed at a dose of 1.5 times of the recommended maximum dosage (375 mg · kg^{-1}) once for the degradation dynamic trials by using knapsack sprayer and the blank control area was sprayed with water. The apple samples were collected at the 2nd hour, 1, 3, 7, 14, 21, 28, 35 and 50 day after the application of pesticides. The 20% aqueous solutions of phthalanilic acid were evenly sprayed at 250(the recommended maximum dosage) and 375 mg · kg^{-1} (1.5 times of the recommended maximum dosage) once for the final residue trials. The apples were sampled at harvest time after the pesticide application. The collected samples were cut into small pieces that were less than 1 cm. The evenly mixed samples were dispensed

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into two sets of 150 g sample by using the coning and quartering method, and loaded into sample bags with proper labeling. All labeled samples were stored at −20±2 ℃ in a low-temperature freezer. We employed the QuEChERS pre-treatment method, where the samples were extracted with 0.1% (φ) formic acid in acetonitrile and purified with PSA and anhydrous magnesium sulfate, followed by gradient elution in Eclipse plus C18 (3.5 μm, 2.1 mm×100 mm) column with mobile phase consisted of formic acid (0.1%, φ) solution-acetonitrile. The instrument used for the analysis was an Agilent 1260-6420 high-performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) system equipped with electrospray ionization (ESI) source. The samples were measured in multiple reaction monitoring (MRM) modes. In order to calculate the recovery rate of spike and coefficient of variation (*CV*), the blank samples of apple were spiked with 0.01, 0.1 and 1 mg · kg^{-1} of phthalanilic acid. Each spiked amount was measured in parallel for 5 times. The samples were extracted with method as above described and tested with instrument conditions as described.【Results】The linear correlation of the method was assessed with curves constructed by measuring a series of concentrations of phthalanilic acid (0.005, 0.01, 0.05, 0.1, 0.5, and 1 mg · L^{-1}) standards in the apple matrix, The concentration was plotted on the horizontal or x-axis whereas its corresponding peak area was plotted on the vertical or y-axis to obtain a linear regression equation (Y=131 806X - 553), the measurement of phthalanilic acid in apple matrices exhibited a linear correlation (R^2=0.992 9) within the range of 0.005 mg · kg^{-1} to 1 mg · L^{-1}. We employed the matrix-based standard curve to calculate the concentration in the samples from the field in order to obtain phthalanilic acid residue data. When the spiking levels of phthalanilic acid in the samples were 0.01-1.00 mg · kg^{-1}, mean recovery values achieved for phthalanilic acid residues ranged from 79 to 91, and the relative standard deviations (RSD) were 2.5%-4.1%. The limit of detection of the analytical method was 2.5×10^{-11} g, and the limit of quantification was 0.01 mg · kg^{-1} in apple. This method has a good sensitivity, accuracy and precision that meeting the requirements for pesticide residue detection. We used the method developed to determine the residue dynamics and final residues of 20% aqueous solutions of phthalanilic acid in apple. The relationship between the residual concentration of phthalanilic acid in apple and the time interval after the pesticide application was consistent with the equation describing the first-order kinetics: $C_t=C_0e^{-kt}$. The half-life of phthalanilic acid in apple from Shandong, Anhui, and Guizhou were 3.7-4.7 days, 4.3-4.8 days and 5.7-5.8 days, respectively. The residues of phthalanilic acid in apple gradually reduced over time with up to more than 86.7% of degradation rate at 14 day. The final residue tests showed that 20% aqueous solutions of phthalanilic acid was sprayed for one time at application dose of 250 375 mg · kg^{-1}, at harvest,the final residues of phthalanilic acid in apple were all ＜ 0.01 mg · kg^{-1}. Phthalanilic acid residue was not detected in apple samples from control areas.【Conclusion】The residues of phthalanilic acid in apple were determined by HPLC-MS/MS, this method has a good sensitivity, accuracy, and precision. The half-lives of phthalanilic acid in apple were 3.7-5.8 d, phthalanilic acid was rapidly degradated in apples. At harvest, the final residues of phthalanilic acid in apple were all ＜ 0.01 mg · kg^{-1}. It is safe to used 20% aqueous solutions of phthalanilic acid at recommended dosage on apple trees.

Key words: Apple; Phthalanilic acid; Residues; Dissipation dynamics

苯肽胺酸（Phthalanilic acid，结构式见图 1），化学名称 N-苯基邻苯二甲酸单酰胺，是一种新型的植物生长调节剂，可通过叶面喷施迅速进入植物体内，缓解植物花期内源激素不足的问题，满足花芽分化对生长激素的需求，并促使营养物质向花芽移动，具有保花保果、提高抗逆性、改善果实品质、提高产量等功效，在枣、猕猴桃、苹果、辣椒、豇豆等果树和蔬菜上使用均有良好表现，近年来在农业生产

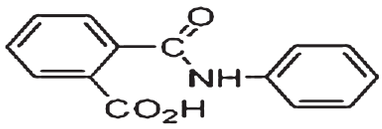

图1　苯肽胺酸化学结构式

Fig. 1　Chemical structure of phthalanilic acid

中应用广泛[1-2]。目前中国、CAC、美国、澳大利亚、韩国、日本等均未制定相应的农产品中苯肽胺酸使用的限量标准和残留限量标准。一些种植者为了提高经济效益，在生产中过度使用，高剂量的植物生长调节剂的使用可能会导致食品、环境中的高残留，危及食品安全，对人类健康产生潜在危害[3]。目前苯肽胺酸的研究主要集中在果树、蔬菜等作物上的使用效果、作用机制、毒性及其含量测定[4-11]，对苯肽胺酸在作物中的残留及消解报道较少。代玲玲等[6]研究发现，苯肽胺酸具有一定的免疫毒性，高剂量的苯肽胺酸对小鼠脾脏造成了明显的器质性病理改变。赵欢欢等[9]报道了土壤中苯肽胺酸的测定及淋溶特性研究。Zhao 等[11]报道了利用 QuEChERS 方法和超高效液相色谱—质谱法对豆类、水果和蔬菜中苯肽胺酸残留量的检测方法，但未对苯肽胺酸在这些作物中的消解动态、最终残留量及安全性进行研究。笔者采用改进的 QuEChERS 方法进行提取、净化，结合较高精确度和灵敏度的液相色谱-质谱联用仪(HPLC-MS/MS)检测，建立苹果中苯肽胺酸残留的检测方法，并对苯肽胺酸在苹果中的消解动态和最终残留量进行分析，以期为该药在苹果生产上的合理使用及其在苹果上残留限量值的确定提供理论支撑。

1　材料和方法

1.1　试验材料

供试农药：20%苯肽胺酸水剂（陕西上格之路生物科学有限公司提供）。

供试材料：苹果，品种为‘嘎拉’‘寒富’和‘富士’。

化学试剂：甲酸、氯化钠、无水硫酸镁均为分析纯，乙腈为色谱纯；PSA（乙二胺-N-丙基硅烷）；苯肽胺酸标准品（99%，上海安谱实验科技股份有限公司）。

仪器设备：Agilent1260-6420 液相色谱-三重串联四级杆质谱联用仪、匀浆机、离心机、多管涡旋混合器、电子天平。

1.2　方法

1.2.1　*田间试验方法*　参照农业行业标准《农作物中农药残留试验准则》[12]和《农药登记残留田间试验标准操作规程》[13]，于 2017—2018 年分别在山东省蓬莱市、贵州省长顺县、安徽省淮北市三地进行了20%苯肽胺酸水剂在苹果上的消解试验和最终残留试验。设置 3 个剂量处理和一个空白对照，每个处理 3 次重复，每小区 2 株树。所有小区都按正常的田间管理。

消解动态试验和最终残留试验施药时期为苹果长到成熟个体一半大小时，消解动态试验 20%苯肽胺酸水剂按施药质量分数 375 mg·kg^{-1}（有效成分）均匀喷雾 1 次，施药后 0.083、1、3、7、14、21、28、35、50 d 后取苹果样品。最终残留试验 20%苯肽胺酸水剂按施药浓度 250 mg·kg^{-1}（有效成分）和 375 mg·kg^{-1}（有效成分）分别均匀喷雾 1 次，收获期采集苹果样品。施药器械为高压喷雾器，空白对照区喷施清水。取样方法为随机在试验小区内不同方向及上、中、下、里、外等不同部位采集 12 个以上（不少于 2 kg）生长正常、无病害的苹果果实，装入样品袋中做好标记。

1.2.2　*分析样品的制备与保存*　田间样本在采集后，8 h 内运回实验室，将苹果除去果柄后，用不锈钢刀具沿纵向切成均匀的四瓣，取对角的两瓣，切成 1 cm 以下的碎块，在不锈钢盆中充分混合均匀，四分法缩分，取 150 g 样品 2 份，分别装入封口样品袋中，贴好标签，贮存于−20 ℃冰柜中。

1.2.3　*样品的提取和净化*　称取 5 g 捣碎的苹果样品于 50 mL 离心管中，加入 5 mL 水，10 mL 乙腈（0.1%甲酸，φ），匀浆 2 min，加入 5 g 氯化钠，涡旋 1 min，5 000 r·min^{-1} 离心 5 min，取 2 mL 上清液转移至装有 0.025 g PSA 和 0.05 g 无水硫酸镁的 15 mL 离心管中，涡旋 1 min，5 000 r·min^{-1} 离心 5 min，取上清液过 0.22 μm 滤膜，待测。

1.2.4　*仪器条件*　色谱柱：Agilent C18（3.5 μm，2.1mm×100 mm），柱温：35 ℃，流动相:乙腈/0.1%甲酸水溶液=80/20，流速：0.4 mL·min^{-1}，进样量：5 μL，离子源：电喷雾离子源 ESI，扫描方式：负离子源，毛细管电压：4 kV(-)，脱溶剂温度：300 ℃，脱溶剂气流量：10 L·min^{-1}，雾化器压力，35 psi；检测方式：多重反应监测（MRM）。其他质谱参数见表 1。

表1 苯肽胺酸的的主要质谱参数

Table 1 Optimized working parameter of ESI-MS/MS for phthalanilic acid

参数 Parameter	保留时间 Retention time/min	定性离子对 qualitative Ion-pair/(m/z)	定量离子对 Quantitative ion-pair/(m/z)	滞留时间 Retention time/ms	碰撞能量 Collision energy/eV
数值 Figure	0.9	240.1/196;240.1/118	240.1/196	150	3;15

2 结果与分析

2.1 苯肽胺酸检测方法分析

2.1.1 检测方法的线性关系 用苹果空白配制0.005、0.01、0.05、0.1、0.5、1 mg·L^{-1}系列质量浓度的苯肽胺酸苹果基质标样，在上述仪器条件下进样，以浓度为横坐标，以其相应的峰面积为纵坐标作图，在0.005~1 mg·L^{-1}范围内，苯肽胺酸的标准曲线为Y=131 806X−553，相关系数为0.992 9，说明该标准曲线线性好，可以单点定量用于样品中苯肽胺酸含量的检测。

2.1.2 检测方法的灵敏度、准确度及精密度 本试验条件下，苯肽胺酸的定量限为0.01 mg·kg^{-1}，说明本方法有较好的灵敏度，符合农药残留检测要求。空白苹果中添加0.01、0.1、1 mg·kg^{-1}三档质量分数的苯肽胺酸标准溶液，设空白对照，每档5次重复，用上述分析方法测定回收率。苯肽胺酸在苹果中的添加回收率为79%~91%，相对标准偏差为2.5~4.1%(表2)。说明本方法有较好的准确度及精密度，符合农药残留检测要求。

表2 苯肽胺酸在苹果中的添加回收结果

Table 2 Fortified recoveries in apple

添加量 Fortification level/(mg·kg^{-1})	回收率Recoveries/%					平均回收率 Average recovery/%	相对标准偏差 Relative standard deviation/%
	1	2	3	4	5		
0.01	80	79	79	74	83	79	4.1
0.10	86	80	81	88	85	84	4.0
1.00	93	80	93	88	89	91	2.5

2.2 苯肽胺酸在苹果中的残留消解动态

由表3可知，山东、安徽和贵州两年试验中，苹果中苯肽胺酸的残留浓度与施药后间隔时间的关系符合一级动力学方程C_t=C_0e^{-kt}，式中：C_t为施药后t时的农药残留量(mg·kg^{-1})，C_0为施药后的原始沉积量(mg·kg^{-1})，k为消解速率常数，t为施药后的天数。山东试验，苯肽胺酸在苹果中的半衰期为3.7~4.7 d，药后14 d降解率超过92.4%。安徽试验，苯肽胺酸在苹果中的半衰期为4.3~4.8 d，药后14 d降解率超过88.9%。贵州试验，苯肽胺酸在苹果中的半衰期为5.7~5.8 d，药后14 d降解率超过86.7%。

2.3 苯肽胺酸在苹果中的最终残留量

2017—2018年，20%苯肽胺酸水剂250 mg·kg^{-1}(有效成分)，施药一次，山东、安徽和贵州收获期苹果样品中苯肽胺酸残留量未检出(＜0.01 mg·kg^{-1})。20%苯肽胺酸水剂375 mg·kg^{-1}(有效成分)，施药一

表3 苯肽胺酸在苹果中的残留消解动态

Table 3 First-order kinetic equation of phthalanilic acid dissipation in apple

试验地点 Test site	年份 Year	一级动力学方程式C_t=C_0e^{-kt} First-order kinetic equation	相关系数 Correlation coefficient, R^2	半衰期 Half-life/d	消解率 Dissipation rate/%
山东	2017	$0.391\ 8e^{-0.187t}$	0.943 6	3.7	93.1
Shandong	2018	$0.640\ 2e^{-0.149t}$	0.978 8	4.7	92.4
安徽	2017	$0.299\ 3e^{-0.160t}$	0.965 6	4.3	88.9
Anhui	2018	$0.515\ 8e^{-0.143t}$	0.973 6	4.8	93.1
贵州	2017	$0.184\ 9e^{-0.121t}$	0.893 3	5.7	92.6
Guizhou	2018	$0.360\ 4e^{-0.119t}$	0.918 0	5.8	86.7

注：消解率为苹果用药后14 d值。

Note: The dissipation rate of phthalanilic acid in apple was tested at 14 days after spraying.

次，山东、安徽和贵州收获期苹果样品中苯肽胺酸残留量未检出（＜ 0.01 mg · kg^{-1}）。对照区样品均未检出苯肽胺酸。

3 讨 论

本试验经 QuEChERS 方法样品前处理，结合液相色谱-串联质谱法完成了苯肽胺酸在苹果中残留的检测。张文等[14]报道了液相色谱–串联质谱法同时测定浆果类、瓜果类水果中 19 种植物生长调节剂的残留量。孙涛等[15]报道了 QuEChERS-UPLC-MS/MS 法测定蔬菜中 5 种植物生长调节剂。在日常检测中可以实现采用 QuEChERS-LC-MS/MS 法可同时完成苯肽胺酸和其他生长调节剂在蔬菜和水果中的残留量检测，缩短检测周期，降低检测成本。

目前生长调节剂在食品中的残留问题引起公众的关注，肖国军等[16]报道了超高效液相色谱–串联质谱法测定果蔬中 6 种植物生长调节剂残留及其污染水平分析，部分果蔬样品测出 2,4-D，多效唑等残留，存在一定的膳食暴露风险。林纯忠等[17]报道了果蔬中植物生长调节剂残留量调查分析，在番茄、葡萄等样品中检测出矮壮素、氯吡脲等残留。但有关苯肽胺酸在果蔬中的残留及消解动态未见报道。本试验中，20%苯肽胺酸水剂，在苹果上的消解半衰期为 3.7~5.8 d，药后 14 d 消解 86.7%以上，总的来说，苯肽胺酸在苹果中半衰期较短，消解速度较快。欧盟默认苯肽胺酸在苹果中的最大残留限量为 0.01 mg · kg^{-1}[18]。暂以 0.01 mg · kg^{-1} 为依据，据山东、安徽及贵州两年三地的残留试验结果，20%苯肽胺酸水剂以 250、375 mg · kg^{-1}（有效成分），施药一次，收获时苹果中苯肽胺酸的最终残留量均＜ 0.01 mg · kg^{-1}，因此，20%苯肽胺酸水剂按推荐剂量在苹果上使用是安全的。目前各国均未制定苯肽胺酸在苹果中的最大残留限量(MRL)，本试验数据为中国制定苯肽胺酸在苹果中的残留限量提供数据基础。本试验结果只是针对苯肽胺酸在苹果中的残留和安全性评价，对在其他作物的安全性，还需进一步试验验证。

4 结 论

采用液质联用法检测苹果中的苯肽胺酸的残留量。苯肽胺酸在苹果中的半衰期为 3.7~5.8 d。苯肽胺酸在苹果中的最终残留量均＜ 0.01 mg · kg^{-1}，20%苯肽胺酸水剂按推荐剂量在苹果上使用是安全的。

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