Analysis of Pesticide Residues in Chayote, *Sechium edule* by GC/MS/MS with QuEChERS

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INTRODUCTION

The chayote is an edible plant of the family Cucurbitaceae. Originally native to Mexico, it was introduced worldwide. It is among the ten most consumed vegetables in Brazil. Nutritionally it is a good source of fibers, potassium, vitamins A, B1, C and amino acids. It has low calorie count, mild flavor and easy digestibility. It is an alternative to increase the consumption of fruit and vegetable in Brazilian governmental initiatives. Many diseases affect cucurbit crops, requiring application of pesticides.

OBJECTIVES

The aim of this study was to implement an analytical multiresidue method for determination of pesticides in chayote by gas chromatography coupled with mass spectrometry triple quadrupole (GC-MS/MS) and the QuEChERS sample preparation method.

METHOD

The method used was based on that described by Association of Official Analytical Chemists (AOAC), AOAC 2007. 01.

Analytical procedure and instrumentation

A 15 g of chayote sample, previously homogenized, was weighed into a 50 mL centrifuge tube, followed by the addition of 15 mL of 1% acetic acid in acetonitrile (ACN). The tube was shaken for 1 minute and 6 g of MgSO4 and 1.5 g of sodium acetate were added. It was shaken for 1 minute and centrifuged at 2000 rpm for 20 minutes. A total of 8 mL of upper ACN layer were transferred to a dispersive tube containing (PSA, C18EC, and MgSO4). The mixture was shaken for 1 minute and centrifuged at 2000 rpm for 20 minutes. Then, 1 mL of the supernatant was transferred to an autosampler vial and evaporated to almost dryness. After this, it was diluted with ethyl acetate and adjusted to volume of 1 mL, followed by the injection into GC/MS/MS system with PTV, equipped with back flush valve (BKF), with a column Thermo Scientific TR-Pesticide II of 30 m × 0.25 mm × 0.25 µm with a deactivated silica guard column 2 m x 0,53 mm.

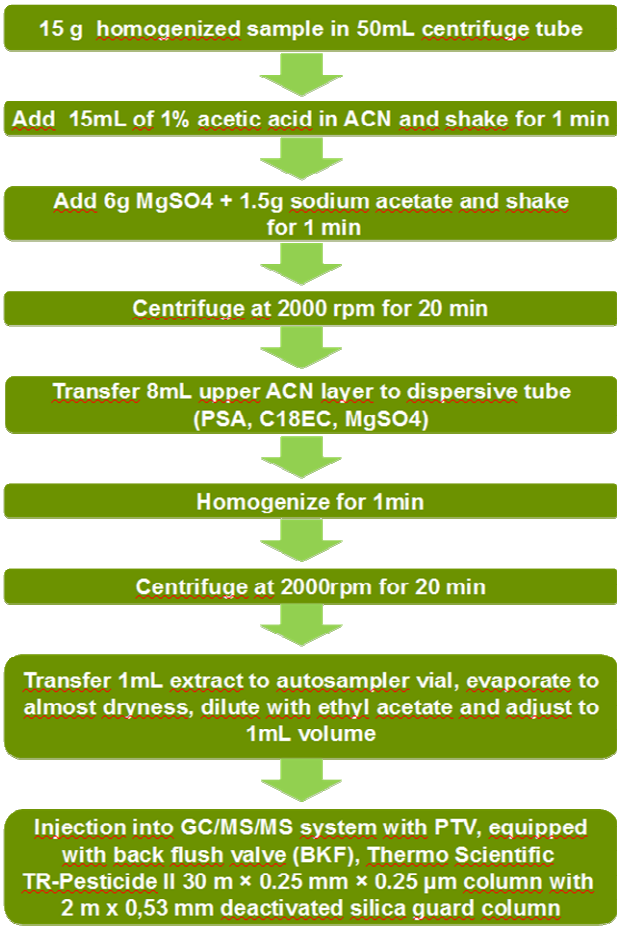

Figure. Flow diagram for pesticide extraction from chayote sample.

RESULTS AND DISCUSSION

The classic multiresidue method of sample preparation is based on liquid-liquid partition which requires large sample volumes and solvents generating environmental impact. The QuEChERS extraction method based on Association of Official Analytical Chemists (AOAC, 2007) was studied for chayote analysis and it was considered suitable for analysis of 38 active ingredients, including isomers and metabolites. This is a simple, quick technique, inexpensive, uses little solvent and is quite efficient in pesticide multiresidues extraction in the analysis of different matrices.

The quantification and confirmation were performed by GC-MS/MS. Blank samples showed no interference of the studied active ingredients (matrix effect). The limits of detection ranged from 0.001 to 0.005 mg/kg and quantification was 0.012mg/kg. Mean recoveries obtained under repeatability conditions (N = 5) ranged from 79 to 103% for the 1LOQ level and from 71 to 108% for 2LOQ level. The method presented good precision with relative standard deviations in the range of 2-14% for 1LOQ and 3-12% for 2LQ (Table). These results comply with the minimum criteria established by the validation procedures adopted by the European Community/SANTE (EC/SANTE, 2015).

The development of investigations in this field is relevant; in Brazil there is no systematic monitoring of quality assessment for chayote, and nearly, there is no registration of pesticides for this crop. The studied methodology is appropriate to acquire data for pesticide residue programs in this matrix. In addition, this study is significant for Good Agricultural Practices verification, ensuring food safety and to estimate the risk of human exposure to illegal residues including chlorinated hydrocarbon insecticide residues.

Table. Pesticides residues analyzed in chayote with MRM transitions, retention times, limits of detection (LOD), limits of quantitation (LOQ), RSD, recovery (REC)

Pesticides	Transition MRM	RT (min)	Working linear range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Recoveries Level: 1 LOQ		Recoveries Level: 2 LOQ	
						Media (%)	RSD (%)	Media (%)	RSD (%)
Mevinphos	127.03-109.02	9.12	0.002-0.1	0.003	0.012	103	7	71	7
Molinate	187.10-126.07	10.15	0.002-0.1	0.003	0.012	87	11	91	4
Trifluraline	306.10-264.09	11.23	0.002-0.1	0.002	0.012	82	6	71	7
Lindane (gama-HCH)	180.91-144.93	11.73	0.002-0.1	0.005	0.012	81	8	90	11
Hexaclorobenzene (HCB)	283.81-248.84	11.84	0.002-0.1	0.005	0.012	87	18	92	11
Simazine	201.08-138.05	12.05	0.005-0.1	0.004	0.012	96	10	95	7
Atrazine	200.09-104.05	12.15	0.003-0.1	0.003	0.012	93	9	95	7
alpha-HCH	180.91-144.93	12.20	0.002-0.1	0.005	0.012	99	15	100	11
Clomazone	125.04-89.03	12.26	0.002-0.1	0.004	0.012	97	12	96	9
beta-HCH	180.91-144.93	12.45	0.002-0.1	0.005	0.012	84	17	90	10
Diazinone	304.10-179.06	12.59	0.002-0.1	0.002	0.012	87	6	89	6
delta-HCH	180.91-144.93	13.03	0.002-0.1	0.005	0.012	91	14	108	4
Vinclozoline	212.00-172.00	13.98	0.002-0.1	0.002	0.012	93	5	97	4
Alachlor	188.08-160.07	14.11	0.002-0.1	0.003	0.012	97	8	89	12
Heptachlor	269.87-234.89	14.39	0.002-0.1	0.002	0.012	90	7	95	6
Chlorpiriphos	198.96-170.96	15.25	0.002-0.1	0.002	0.012	95	6	103	5
Aldrin	262.91-192.93	15.43	0.002-0.1	0.002	0.012	86	7	93	5
Pendimentaline	252.12-162.08	16.26	0.003-0.1	0.002	0.012	95	5	77	11
cis-Heptachlor epoxide	352.83-262.87	16.59	0.002-0.1	0.002	0.012	92	6	98	6
trans-Heptachlor epoxide	217.00-182.00	16.71	0.003-0.1	0.002	0.012	93	7	93	6
Procimidone	283.02-96.01	16.87	0.002-0.1	0.002	0.012	96	5	102	5
gama-Chlordane	372.81-265.87	17.3	0.002-0.1	0.002	0.012	96	5	106	3
o,p-DDE	245.95-175.97	17.38	0.002-0.1	0.002	0.012	93	5	101	3
alpha-Endossulfan	240.89-205.91	17.72	0.002-0.1	0.002	0.012	90	7	99	5
alpha-Chlordane	372.81-265.87	17.72	0.002-0.1	0.002	0.012	90	5	100	4
trans-Nonachlor	406.78-299.84	17.82	0.002-0.1	0.002	0.012	89	6	98	6
p,p-DDE	245.95-175.97	18.41	0.002-0.1	0.001	0.012	84	2	94	3
Dieldrin	262.91-192.93	18.59	0.002-0.1	0.001	0.012	83	5	89	6
o,o-DDD	234.97-164.98	18.64	0.002-0.1	0.001	0.012	85	5	91	3
Endrin	262.91-192.93	19.26	0.002-0.1	0.002	0.012	86	8	90	3
p,p-DDD	236.97-164.98	19.77	0.002-0.1	0.002	0.012	85	6	91	3
o,p-DDT	234.94-164.96	19.88	0.002-0.1	0.001	0.012	79	5	84	8
Epoxiconazole	192.04-138.03	21.98	0.002-0,1	0,001	0,012	85	4	88	5
Bifentrine	181.05-166.05	22.79	0.003-0.1	0.001	0.012	85	3	84	4
Tetradifone	226.93-198.94	23.61	0.002-0.1	0.002	0.012	85	6	87	4
Fenarimol	139.01-111.01	24.87	0.03-0.1	0.001	0.012	89	3	79	3

CONCLUSIONS

This multiresidue method using QuEChERS in the sample preparation step and chromatographic technique GC/MS/MS is suitable and highly sensitive for the active ingredients of pesticides studied. This study contributes to development of monitoring programs and researches, insufficient for residues of pesticides used on chayote.

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