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Domestic methods for the removal of pesticide residues in chilies

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Abstract
Now a days understanding of the magnitude and impact of the pesticide exposure is an important concern for the consumers. In daily life Chilies are the unavoidable food material and which are highly exposed to pesticide usage. In order to investigate the effectiveness of several cleaning methods in removing pesticide residues in chilles a study was conducted in Pesticide Management Division, NIPHM. Normally available chilies in the market are treated with five pesticides which includes Bifenthrin, Deltamethrin, Hexaconazole, Lamda cyhalothrin and Profenophos and the treated sample was subjected to four different household methods for removal of residues like T1 (Running Tap water wash, T2 (Boiling for 10 min), T3 (Soaking in 2% salt solution for 10 min), T4 (Soaking in 2% salt solution for 10 min+ boiling for 10 min). The residues present in the sample were analysed by using QTOF GC/MS instrument. Among the four methods 2% salt solution + boiling method had greatest residue removal effect for Deltamethrin (72.90%) to Hexaconazole (37.23%).

Keywords: Pesticide, domestic methods, decontamination, chili, QTOF GC/MS

Introduction
Chilli is one of the most important condiments having immense commercial and therapeutic value (Reddy *et al*. 2007a) [10]. It is an important ingredient in day-to-day foods like curries, pickles and chutneys. It is a rich source of vitamins A, C and E (Kumar *et al*. 2000) [6]. Asia produces 65.8% of world green chilies and pepper and stands at the top; Europe stands 2nd contributing 12.1% and Africa 3rd with 9.5% of world production (FAO). India is the world's largest producer, consumer and exporter of Chilli. Guntur in Andhra Pradesh produces 30% of all the chilies produced in India and the state of Andhra Pradesh as a whole contributes 75% of India's chili exports (FAO).

Pesticides are used for controlling various insect pests, pathogens and which destroy chilli crop.

It is well-known that repeated application of wide range of pesticides may lead to undesirable residues on consumable parts of vegetables (Agnihotri 1999) [2]. Therefore, the sensible suggestion of a pesticide requires that it must not only provide an effective control of pests, but at the same time its residues on the commodity must also be toxicologically acceptable.

A pesticide residue is any substance or mixture of substances in food for man or animals resulting from the use of a pesticide and includes any specified derivatives, such as degradation and conversion products, metabolites, reaction products and impurities which are considered to be of toxicological significance.(Definition given by FAO).

Residual pesticides on fresh vegetables and fruits decrease by various culinary applications or with time, depending on the type and properties of the pesticides. Several investigators have found that levels of some pesticide residues were reduced by the pre-harvest intervals and/or culinary application, Such as Washing, peeling, cooking, boiling and storage (Cengiz Metal. 2007) [4]. Moreover, these techniques in some cases are unsuitable for removal of residual pesticides adhering to surfaces of vegetables and fruits and/or present in plant tissues.

In this study efforts were made to remove the pesticide residues in chilies by following different household preparations *viz.,* Running tap water washing, boiling for 10 min, soaking in 2% salt solution for 10 min etc.

Materials and Methods
Chilli samples were collected from the organic store in Hyderabad. The collected residue free samples were treated with Bifenthrin, Deltamethrin, Hexaconazole, Lamda Cyhalothrin, and Profenophos by preparing 0.2% formulation solution for spraying. The selection of pesticides was based on the CIBRC approved list of pesticides on chilli crop.

Recovery study

Prior to the decontamination methods, recovery studies were made by using AOAC official method 2007.01 (Pesticide Residues of Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulphate) and which was slightly modified to suit to the facilities available at the laboratory and the same was validated. Fortification and recovery study was done at 0.1 and 0.5 ppm level.

To identify different insecticide residues, the retention times of the sample in the GC (using Gas Chromatography on Quadrupole Time of Flight Detector) was compared with the technical standards. For the quantification of the insecticides, the area of the sample was compared with the standard area at a particular retention time.

Decontamination methods

T_1 (Running Tap water wash)

Sample of 500 gms was taken and pesticides were sprayed on it and left to dry. Then they were

kept under running tap water for 10min and washed and allowed the water to be drained. Later it was crushed, extractions were made and analysed for residues present.

T_2 (boiling for 10 min)

Sample of 500 gms was taken and pesticides were sprayed on it. And left to dry. Then they were boiled for 10 min at 70 °**C.** Later the sample was taken for crushing and extracted for residues and analysed.

T_3 (Soaking in 2% salt solution for 10 min)

2 litres of 2% salt solution was prepared by mixing 40 g of table salt in 2 litres of water and 500 g of green chilies were soaked for 10 min, followed by analysis.

T_4 (Soaking in 2% salt solution for 10 min+ boiling for 10 min)

2 litres of 2% salt solution was prepared by mixing 40 g of table salt in 2 litres of water and 500 g of green chilies were soaked for 10 min, and then they were boiled for 10 min followed by analysis.

T_5 (Control)

Pesticides were sprayed on pesticide free organic sample and further they were analysed for residue presence. It was taken as control to compare with each treatment.

In each treatment the residues in the sample were extracted and cleanup was done with modified QuEChERS method (Multi residue Analysis method) and finally estimated with gas chromatography (GC) equipped with Q TOF. The operating parameters have been mentioned in (Table.1)

The residues of pesticides present after each treatment were calculated.

Table 1: Operated Accurate Mass QTOF GC/MS Parameters

Instrument		**Agilent Accurate Mass QTOF GC/MS**	
GC parameters			
Equilibration time: 0.5 min			Max temperature: 325˚C
Oven programme: 60˚C for 1 min; then 60˚C/min to 170˚C for 0min; 10˚C/min to 275˚C for 7 min.			Carrier gas: He and N2 @ 2.5 and 1.5 ml/min flow rates respectively
Syringe size: 10µl	Inject volume: 2µl	Solvent A volume: 8µl	Solvent B volume: 8µl
Mode: Split less	Total flow: 64.5 ml/min		Pressure: 3.1 PSI
Heater: 250˚C	Septum purge flow to split vent: 60ml/min at 0.75 min		Flow: 1.5 ml/min
Run time: 21.25 min	Column: 30mX0.250mmX0.50µm(length X diameter X film)		
QTOF Parameters			
Ionization mode: EI	Transfer line Temperature: 290°C		Ion source temperature: 250 °C
Collision gas: He 1.5ml/mint	Acquisition time: 200 ms/Spectra		Mass range: m/z 50–600
Software for date analysis: Mass Hunter Acquisition B.06 and MassHunter Qualitative Analysis B.05			

Method validation: In order to validate the analytical method the fortification and recovery studies were done by spiking of 0.1 and 0.5 ppm concentrations of CRMs of Bifenthrin (97% purity Dr. Ehrenstorfer), Deltamethrin (98.5% purity, Dr. Ehrenstorfer), Hexaconazole (99% purity, Dr. Ehrenstorfer), Lamda cyhalothrin (98.5% purity, Dr. Ehrenstorfer), Profenophos (92% purity, Dr. Ehrenstorfer), standards to the control chilli sample. The spiked samples were processed according to the QuEChERS method along with the control sample, without spiking any standard. The study was done in replications.

Table 2: Recovery studies

Name of the Pesticide	Recovery (%) @ 0.1ppm	Recovery (%) @ 0.5ppm
Bifenthrin	110.41	92.20
Deltamethrin	105.91	82.35
Hexaconazole	89.38	86.25
Lambda Cyhalothrin	96.32	94.02
Profenphos	99.73	89.11

Reagents

Anhydrous sodium Sulphate, Primary Secondary Amine (PSA), Magnesium sulfate, anhydrous and $MgSO_4$ were obtained from Himedia, pesticide formulations from licensed dealer.

Results

Table 3: Effect of Different Decontamination Methods in the removal of Pesticide Residues in Chilli

		Control	Residue after each treatment			
S. No	Chemical	Residue in Spiked sample (Residues in mg/kg)	Tap Water Wash (Residues in mg/kg)	Boiling (Residues in mg/kg)	2% Salt Solution (Residues in mg/kg)	2% Salt Sol+ Boiling (Residues in mg/kg)
1	Bifenthrin	0.90	0.70	0.59	0.55	0.47
2	Deltamethrin	0.85	0.61	0.43	0.30	0.23
3	Hexaconazole	0.90	0.68	0.64	0.63	0.57
4	Lambda Cyhalothrin	0.79	0.56	0.46	0.45	0.31
5	Profenphos	0.88	0.60	0.51	0.45	0.35

The residues of Bifenthrin, Deltamethrin, Hexaconazole, Lamda cyhalothrin and Profenophos in chilli sample have got substantial reduction by different decontamination methods. The concentration of pesticide residues retained in chilies after various treatment methods are presented in Table 3.

In the spiked chilli sample the residue levels are reported in the range of 0.79 for Lamda cyhalothrin, 0.85 for Deltamethrin, 0.88 for Profenophos and 0.90 for Bifenthrin and Hexaconazole. Among the four household methods for pesticide removal, in the T4 (Soaking in 2% salt solution for 10 min+ boiling for 10 min) lowest pesticide residues concentrations were recorded. Compared to all treatment methods highest concentrations of residues were recorded in T1 (Tap water wash). In remaining two methods i.e., T2 (boiling for 10 min), T3 (Soaking in 2% salt solution for 10 min) intermediary concentration were reported. The reduction of pesticides in various treatments is represented in Fig 1.

In the process of running tap water washing Profenophos residues were reduced up to 31.73%, whereas Deltamethrin and Lamda cyhalothrin residues were reduced upto 28.03 and 28.67 percent respectively. Hexaconazole and Bifenthrin residues were reduced to 24.24 and 21.84 percent respectively.

With the boiling method the percentage of reduction in residues ranges from 28.46 in Hexaconazole to 49.37 in Deltamethrin. The intermittent percentages are 42.66 in Profenophos, 41.38 in Lamda cyhalothrin and 34.43 in Bifenthrin were recorded. The boiling method has shown slightly better effect when compared with Tap water washing.

By washing with 2% salt water solution, Deltamethrin was reduced to 64.51% and Profenophos was reduced to 48.68%. 42.57% of reductions in Lamda cyhalothrin, 38.67% reduction in Bifenthrin are obtained. Hexaconazole has got reduction up to 30.33%. A prominent effect has got with 2% salt water washing when compared with above two methods.

In washing with 2% salt water solution followed by Boiling method distinguished reduction in residues are recorded. Highest percentage of residue reduction recorded in Deltamethrin (72.90%) followed by Lamda cyhalothrin (61.26%). Where as in Profenophos and Bifenthrin 59.97% and 47.81% of residue reduction reported respectively. In Hexaconazole 37.23% of reduction reported through this method.

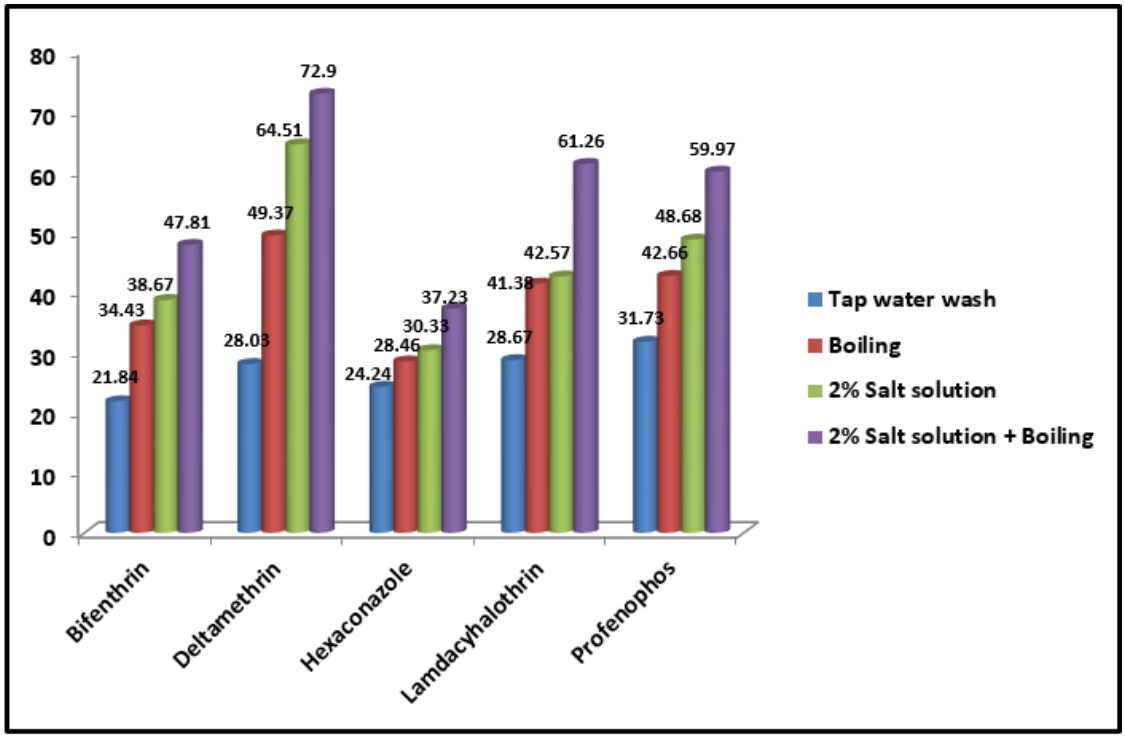

Fig 1: Percentage Removal of Pesticide Residues in Green Chilli by various Domestic Decontamination Methods

Discussion

Pesticides are used indiscriminately and excessively throughout the globe, and these residues remain in the food materials, water, fruits and vegetables (Baptista *et al*., 2008, Lazic *et al*., 2009) [3, 7] and in total diet. Excessive use of pesticides, their toxic residues has been reported in various environmental commodities (Patel *et al*., 1999, Lazic *et al*., 2009) [9, 7]. These pesticide residues enter in to the human

body by consumption of the pesticide contaminated food which leads to the chronic disorders. Thus the removal of these residues from food commodities utilizing different household processing methods is very essential. The different household preparations such as washing, cooking, washing plus cooking, salt water washing play a role in reduction of pesticide residues (Wasim Aktar *et al*., 2010) [12].

In the present study, it was found that tap water wash was the less effective treatment among all the decontamination methods followed and the findings of present investigations are in agreement with the findings of Parmar *et al*. (2012) [8] Shashi Bhushan *et al*. (2014) [11] reported that washing with tap water reduced the residues in tomato. Kumari *et al*. (2008) [5] reported that boiling process can completely remove organ phosphorus insecticides.

In the present study it was found that 2% salt solution + boiling was found to be most effective in the removal of pesticide residues and said to be safe for human consumption.

The results of earlier workers (Aurore *et al*., 2012, Wasim Atkar *et al*., 2010, Kumari B., 2008, Shasi Bhushan *et al*., 2014) [1, 12, 5, 11] have shown similar results reducing the pesticide residues from other vegetables.

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