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A STUDY ON DECONTAMINATION OF PESTICIDES IN GRAPES

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ABSTRACT: The effects of washing with tap and lemon water, acetic acid and sodium bicarbonate on different pesticide residue levels in grapes were investigated at 10 min processing time. An analysis of these pesticides was conducted using easy Household washing methods. The processing factor (PF) for each pesticide in each processing technique was determined. Washing with Bicarbonate was demonstrated to be more effective than washing with tap water. Bicarbonate decreased the residues of the most compounds, with reductions ranging from 35 to 85.9 %. The data indicated that cleaning and washing were the most effective treatments for the reduction of pesticide residues on grapes, resulting in a lower health risk to pesticide exposure. To investigate the levels of pesticides in grape samples and their properties, a principal component analysis (PCA) was performed.

Key words: Grapes, Pesticide washing, Fungicide and insecticide residues

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INTRODUCTION

Pesticide residue refers to the pesticides lest over on a substrate. In case of food, pesticides may remain on or in food after they are applied to food crops. The levels of these residues in foods are often stipulated by regulatory bodies in many countries (Battino et al. 2009; McDougall and Stewart 2012). Many of these chemical residues, especially derivatives of chlorinated pesticides, exhibit bioaccumulation and biomagnifications which could build up to harmful levels in the body as well as in the environment (Fernandes et al. 2012; Wołejko et al. 2014). Persistent chemicals can be bio-magnified through the food chain and food web. They have been detected in products ranging from meat, poultry, and fish, to vegetable oils, nuts, and various fruits and vegetables. Indian agriculture has progressed a long way and became a significant exporter of different agricultural commodities from the regime of food shortages (Christensen et al. 2003; Angioni et al. 2004; Kim and Huat 2010). In recent years India is facing a great challenge in exporting because of rejection of many food commodities that contain residues higher than maximum residue limits (MRL's) specification. The US rejected as many as 256 food export consignments from India in August 2015. Basmati rice which fetches twice the price of non-basmati is rejected because of high pesticide residues. Residues of four pesticides which are banned in India are detected by analysis as aldrin in brinjal, chlordane in apple, chlorfenvinpfos in bitter gourd, and heptachlor in brinjal. Many health problems are associated with the intake of pesticide in the diet (Shabeer et al. 2015; Kentish and Feng 2014; Kaushik et al. 2009; Keikotlhaile et al. 2010).

Hence the present study was taken up to study the effect of different washes on the insecticide residues present ongrapes of Telangana fruit markets.

Methodology

A field trial was conducted during 2011-14 in order to study the Effect of House hold Processing Methods in the removal of certain pesticides in Grapes which are picked up from fruit markets resulting Spray application of Profenophos 50EC@ 2ml/litres, Chlorpyrifos 20EC@ 2ml/litres, Dimethoate 30EC @ 4ml/litres, Malathion 50EC@3ml/litres, Phosalone 35EC@3ml/litres, Quinalphos 25EC@ 2ml/litres, Triazophos 40EC @ 2.5ml/litres, Lamdacyhalothrin 5EC@ 0.6ml/litres. Single spray was given and collected the Grapes samples after 2 hours and brought to the Laboratory for further analysis.

Treatments: Household Processing Methods

T1 (Direct):

2 Kg of grapes fruits were directly taken for the analysis.

T_1 (Tap water wash):

Four litres of tap water was taken into the plastic tub of 7 litresers capacity and 2 Kg of grapes fruits were dipped in the tub for 10 min, followed by the tap water wash for 30 sec, further the fruits were kept for air drying on tissue paper for 5 min.

T_2 (Soaking in 2% salt solution for 10 min followed by tap water wash):

Four litres of 2 % salt solution was prepared by mixing 80 g of table salt in 4 litres of water in plastic tub of 7 litres capacity and 2 Kg grapes fruits were dipped in the tub for 10 min, followed by the tap water wash for 30 sec, further the fruits were kept for air drying on tissue paper for 5 min, followed by analysis.

T3 (Soaking in 2% Tamarind solution for 10 min followed by tap water Wash):

Four liters of 2 % Tamarind solution was prepared by mixing 80 g of tamarind in 4 litres of water in plastic tub of 7 lit capacity and 2 Kg grapes fruits were dipped in the tub for 10 min, followed by the tap water wash for 30 sec, further the fruits were kept for air drying on tissue paper for 5 min, followed by analysis.

T_4 (Dipping in 0.1% baking soda) ($NaHCo_3$):

Four lit of 0.1% baking soda solution was prepared by mixing 4 g of baking soda in 4 lit of water in plastic tub of 7 lit capacity and 2 Kg grapes fruits were dipped in the tub for 10 min, followed by the tap water wash for 30 sec, further the fruits were kept for air drying on tissue paper for 5 min, followed by analysis.

T_5(Soaking in 4% acetic acid solution for 10 min followed by tap water wash):

Four lit of 4% acetic acid solution was prepared by mixing 160 ml of acetic acid glacial 100% in 4 lit of water in plastic tub of 7 litres capacity, mixture was kept for 1 min and 2 Kg of grapes fruits were dipped in the tub for 10 min, followed by the tap water wash for 30 sec, further the fruits were kept for air drying on tissue paper for 5 min, followed by analysis.

T_6 (Dipping in Lemon water):

Four litres of Lemon water solution was prepared by mixing 12ml in 4 litres of water in plastic tub of 7 litres capacity and 2 Kg grapes fruits were dipped in the tub for 10 min, followed by the tap water wash for 30 sec, further the fruits were kept for air drying on tissue paper for 5 min, followed by analysis.

T7 (Biowash):

2 Kg grapes fruits were dipped in the Biowash solution for 10 min, further the fruits were kept for air drying on tissue paper for 5 min, followed by analysis.

Extraction and clean up for Grape samples

After 10min of each treatment, Grape samples were taken out and air dried for 5 min. Grape samples were analyzed for Dimethoate, Profenophos, Chlorpyrifos, Malathion, Phosalone, Quinalphos, Triazophos, Lambda cyhalothrin pesticide residues following the AOAC official method 2007.01 (QuEChERS) after validation of the method at the laboratory. The samples were homogenized with robot coupe blixer, and homogenized 15±0.1g sample was taken in 50ml centrifuge tube. The sample tube is then added with 30±0.1 ml acetonitrile. The sample is homogenized at 14000-15000 rpm for 2-3 min using Heidolph silent crusher. The samples is then added with 3±0.1g sodium chloride and mixed by shaking gently followed by centrifugation for 3 min at 2500-3000 rpm to separate the organic layer. The top organic layer of about 16 ml was taken into the 50 ml centrifuge tube and added with 9±0.1g anhydrous sodium sulphate to remove the moisture content. 8 ml of extract was taken in to 15 ml tube, containing 0.4±0.01gr PSA sorbent (for dispersive solid phase d-SPE cleanup) and 1.2±0.01gr anhydrous magnesium sulphate. The sample tube was vortexed for 30sec then followed by centrifugation for 5min at 2500-3000rpm. The extract of about 2ml was transferred into test tubes and evaporated to dryness using turbovap with nitrogen gas and reconstituted with 1ml n-Hexane for GC analysis with ECD detector.

RESULTS AND DISCUSSION

The data obtained revealed that the percentage reduction of various pesticides such as Dimethoate, Chlorpyrifos, Quinolphos, Profenophos, Phosalone, Lamdacyhalothrin, Malathion. After the tap water wash represented in (table 2), the percentage reduction was found to be in the range of 28.0 – 56.1% for various pesticides. Washing with Lemon water for 10 min is represented in (table 3), the percentage reduction was found to be in the range of 24.4-56.5% for various pesticides. Washing with 2% Tamarind solution for 10 min is represented in (table 4), the percentage reduction was found to be in the range of 25.8-80.4% for various pesticides. Washing with 2% Salt solution for 10 min is represented in (table 5),the percentage reduction was found to be in the range of 23.5-66.3% for various pesticides. Washing with 0.1% sodium bicarbonate for 10 min is represented in (table 6),the percentage reduction was found to be in the range of 39.0-77.0% for various pesticides. Washing with 4% Acetic acid for 10 min is represented in (table 7), the percentage reduction was found to be in the range of 36.5-79.5% for various pesticides. Washing with Bio-wash for 10 min is represented in (table 8), the percentage reduction was found to be in the range of 23.6-58.4% for various pesticides, Cooking in Pressure cooker for 10 min is represented in (table 9), the percentage reduction was found to be in the range of 15.7-57.8% for various pesticides.

Table-1: Pesticide Residues (mg kg-1) in Grape Samples collected at 2 hrs after spray Control

Pesticide	Residues (mg kg-1)				SDEV	% RSD	MRL (mg kg-1)	
	R1	R2	R3	AVERAGE			FSSAI	CODEX
Dimethoate	1.81	1.46	1.78	1.68	0.083	4.885	2	NA
Chlorpyriphos	4.35	4.52	4.44	4.44	0.034	0.763	0.5	0.5
Quinolphos	0.93	0.88	0.86	0.89	0.017	1.931	NA	NA
Profenophos	1.41	1.39	1.37	1.39	0.012	0.890	NA	NA
Phosalone	1.59	1.57	1.57	1.58	0.007	0.420	5	NA
Lamda cyhalothrin	2.27	2.28	2.26	2.27	0.006	0.242	NA	0.3
Malathion	0.14	0.15	0.15	0.15	0.006	3.546	4	5
Triazophos	1.98	1.89	1.86	1.91	0.064	3.343	NA	NA

Tap water: Results showed after decontamination of Grapes reduction of Dimethoate to 0.902mg kg-1 of 53.4 % over control , reduction of Chlorpyrifos to 1.241mg kg-1 of 28.0% over control , reduction of Quinalphos to 0.501mg kg-1 of 56.1 % over control, reduction of Profenophos to 0.696mg kg-1 of 49.8 % over control, reduction of Phosalone to 0.878mg kg-1 of 55.4 % over control, reduction of Lambda cyhalothrin to 0.978mg kg-1 of 43.0 % over control, reduction of Malathion to 0.079mg kg-1 of 50.9 % over control.

Fig 1: Tap water wash

Table 2: Pesticide Residues (mg kg-1) in Grape Samples after Dipping in tap water

Pesticide	R1	R2	R3	AVERAGE	SDEV	% RSD	% Removal over control
Dimethoate	0.805	0.969	0.932	0.902	0.086	9.536	53.394
Chlorpyriphos	1.044	1.421	1.259	1.241	0.189	15.235	27.960
Quinolphos	0.494	0.511	0.498	0.501	0.009	1.774	56.103
Profenophos	0.602	0.786	0.699	0.696	0.092	13.231	49.845
Phosalone	0.745	0.993	0.897	0.878	0.125	14.237	55.427
Lamda cyhalothrin	0.905	1.004	1.026	0.978	0.064	6.588	42.997
Malathion	0.080	0.076	0.081	0.079	0.003	3.349	50.858
Triazophos	0.771	0.803	0.734	0.769	0.035	4.488	40.139

Lemon water: Results showed after decontamination of Grapes reduction of Dimethoate to 0.760mg kg-1 of 45.0 % over control , reduction of Chlorpyrifos to 1.084mg kg-1 of 24.4% over control , reduction of Quinalphos to 0.505mg kg-1 of 56.5 % over control, reduction of Profenophos to 0.644mg kg-1 of 46.1 % over control, reduction of Phosalone to 0.798mg kg-1 of 50.3 % over control, reduction of Lambda cyhalothrin to 1.002mg kg-1 of 44.0 % over control, reduction of Malathion to 0.069mg kg-1 of 44.6 % over control.

Table 3: Pesticide Residues (mg kg-1) in Grapes Samples after Dipping in Lemon Water for 10min

Pesticide	R1	R2	R3	AVERAGE	SDEV	% RSD	% Removal over control
Dimethoate	0.754	0.721	0.804	0.760	0.042	5.501	44.968
Chlorpyriphos	1.145	1.101	1.007	1.084	0.070	6.501	24.424
Quinolphos	0.511	0.469	0.534	0.505	0.033	6.531	56.514
Profenophos	0.644	0.61	0.677	0.644	0.034	5.205	46.119
Phosalone	0.776	0.797	0.82	0.798	0.022	2.759	50.337
Lamda Cyhalothrin	1.001	0.997	1.007	1.002	0.005	0.502	44.023
Malathion	0.068	0.074	0.066	0.069	0.004	6.005	44.635
Triazophos	0.638	0.598	0.688	0.641	0.045	7.031	33.461

2% Tamarind solution: Results showed after decontamination of Grapes reduction of Dimethoate to 0.994mg kg-1 of 58.8 % over control , reduction of Chlorpyrifos to 1.146mg kg-1 of 25.8% over control , reduction of Quinalphos to 0.718mg kg-1 of 80.4 % over control, reduction of Profenophos to 0.804mg kg-1 of 57.6 % over control, reduction of Phosalone to 1.055mg kg-1 of 66.6 % over control, reduction of Lambda cyhalothrin to 1.387mg kg-1 of 61.0 % over control, reduction of Malathion to 0.122mg kg-1 of 78.5 % over control.

Table 4 : Pesticide Residues (mg kg-1) in Grapes Samples after Dipping in 2% Tamarind Solution for 10 min

Pesticide	R1	R2	R3	AVERAGE	SDEV	% RSD	% Removal Over control
Dimethoate	0.998	0.977	1.007	0.994	0.015	1.549	58.840
Chlorpyriphos	1.322	1.112	1.005	1.146	0.161	14.068	25.820
Quinolphos	0.779	0.699	0.677	0.718	0.054	7.473	80.440
Profenophos	0.801	0.799	0.811	0.804	0.006	0.800	57.583
Phosalone	1.08	1.006	1.078	1.055	0.042	3.997	66.554
Lamda cyhalothrin	1.339	1.401	1.421	1.387	0.043	3.083	60.958
Malathion	0.111	0.134	0.121	0.122	0.012	9.453	78.541
Triazophos	0.912	0.887	0.851	0.883	0.031	3.471	46.087

2% Salt solution: Results showed after decontamination of Grapes reduction of Dimethoate to 0.746mg kg-1 of 44.1 % over control , reduction of Chlorpyrifos to 1.044mg kg-1 of 23.5% over control , reduction of Quinalphos to 0.592mg kg-1 of 66.3 % over control, reduction of Profenophos to 0.600mg kg-1 of 43.0 % over control, reduction of Phosalone to 0.706mg kg-1 of 44.5 % over control, reduction of Lambda cyhalothrin to 0.963mg kg-1 of 42.3 % over control, reduction of Malathion to 0.081mg kg-1 of 52.4 % over control.

Table 5: Pesticide Residues (mg kg-1) in Grapes Samples after Dipping in 2% Salt Solution for 10 min

Pesticide	R1	R2	R3	AVERAGE	SDEV	% RSD	% Removal over control
Dimethoate	0.754	0.741	0.742	0.746	0.007	0.970	44.140
Chlorpyriphos	1.111	1.025	0.995	1.044	0.060	5.769	23.508
Quinolphos	0.632	0.601	0.544	0.592	0.045	7.536	66.331
Profenophos	0.64	0.628	0.532	0.600	0.059	9.866	42.990
Phosalone	0.707	0.711	0.699	0.706	0.006	0.866	44.531
Lamda cyhalothrin	1.002	0.987	0.899	0.963	0.056	5.780	42.309
Malathion	0.086	0.084	0.074	0.081	0.006	7.905	52.361
Triazophos	0.639	0.652	0.601	0.631	0.027	4.202	32.904

0.1% Sodium bicarbonate solution: Results showed after decontamination of Grapes reduction of Dimethoate to 0.983mg kg-1 of 58.2 % over control , reduction of Chlorpyrifos to 1.733mg kg-1 of 39.0% over control , reduction of Quinalphos to 0.688 mg kg-1 of 77.0 % over control, reduction of Profenophos to 0.865mg kg-1 of 62.0 % over control, reduction of Phosalone to 1.037mg kg-1 of 65.4 % over control, reduction of Lambda cyhalothrin to 1.353mg kg-1 of 59.5 % over control, reduction of Malathion to 0.087mg kg-1 of 56.2 % over control.

Table 6: Pesticide Residues (mg kg-1) in Grapes Samples after Dipping in 0.1% Sodium bicarbonate Solution for 10 min

Pesticide	R1	R2	R3	AVERAGE	SDEV	% RSD	% Removal over control
Dimethoate	1.009	0.986	0.953	0.983	0.028	2.864	58.169
Chlorpyriphos	1.773	1.884	1.543	1.733	0.174	10.034	39.042
Quinolphos	0.722	0.696	0.645	0.688	0.039	5.696	77.006
Profenophos	0.932	0.874	0.788	0.865	0.072	8.379	61.954
Phosalone	1.104	1.022	0.984	1.037	0.061	5.916	65.419
Lamda cyhalothrin	1.432	1.094	1.533	1.353	0.230	16.993	59.464
Malathion	0.097	0.086	0.079	0.087	0.009	10.390	56.223
Triazophos	0.911	0.898	0.799	0.869	0.061	7.046	45.357

4% acetic acid solution: Results showed after decontamination of Grapes reduction of Dimethoate to 1.010 mg kg-1 of 59.8 % over control , reduction of Chlorpyrifos to 1.620mg kg-1 of 36.5% over control , reduction of Quinalphos to 0.710mg kg-1 of 79.5 % over control, reduction of Profenophos to 0.837mg kg-1 of 60.0 % over control, reduction of Phosalone to 1.208mg kg-1 of 76.2 % over control, reduction of Lambda cyhalothrin to 1.535mg kg-1 of 67.5% over control, reduction of Malathion to 0.109mg kg-1 of 70.0 % over control.

Table 7: Pesticide Residues (mg kg-1) in Grapes Samples after Dipping in 4% Acetic Acid Solution for 10 min

Pesticide	R1	R2	R3	AVERAGE	SDEV	% RSD	% Removal over control
Dimethoate	1.003	1.044	0.983	1.010	0.031	3.079	59.787
Chlorpyriphos	1.661	1.599	1.601	1.620	0.035	2.174	36.497
Quinolphos	0.705	0.711	0.715	0.710	0.005	0.709	79.545
Profenophos	0.803	0.904	0.804	0.837	0.058	6.933	59.971
Phosalone	1.112	1.204	1.307	1.208	0.098	8.078	76.210
Lamda Cyhalothrin	1.411	1.773	1.422	1.535	0.206	13.411	67.477
Malathion	0.101	0.122	0.103	0.109	0.012	10.666	69.957
Triazophos	0.953	1.094	0.919	0.989	0.093	9.386	51.583

BIO WASH Solution: Results showed after decontamination of Grapes reduction of Dimethoate to 0.871mg kg-1 of 51.5 % over control , reduction of Chlorpyrifos to 1.046mg kg-1 of 23.6% over control , reduction of Quinalphos to 0.522mg kg-1 of 58.4% over control, reduction of Profenophos to 0.650mg kg-1 of 46.6% over control, reduction of Phosalone to 0.804mg kg-1 of 50.7 % over control, reduction of Lambda cyhalothrin to 1.045mg kg-1 of 45.9% over control, reduction of Malathion to 0.083mg kg-1 of 53.2 % over control.

Table 8: Pesticide Residues (mg kg-1) in Grapes Samples after Dipping in BIOWASH Solution for 10 min

Pesticide	R1	R2	R3	AVERAGE	SDEV	% RSD	% Removal over control
Dimethoate	0.912	0.899	0.801	0.871	0.061	6.970	51.539
Chlorpyriphos	1.011	1.134	0.993	1.046	0.077	7.337	23.560
Quinolphos	0.533	0.52	0.512	0.522	0.011	2.032	58.417
Profenophos	0.685	0.661	0.605	0.650	0.041	6.313	46.597
Phosalone	0.801	0.812	0.799	0.804	0.007	0.871	50.736
Lamda cyhalothrin	1.088	1.065	0.983	1.045	0.055	5.280	45.942
Malathion	0.084	0.089	0.075	0.083	0.007	8.582	53.219
Triazophos	0.643	0.614	0.601	0.619	0.022	3.472	32.313

CONCLUSION

The studied washing methods for removal of pesticides was successful. Washing with tap and other materials were used to determine the effectiveness of the removal of pesticide residues in grapes. Concentration changes of pesticide residues after 10min treatments were observed, and a gradual reduction was noted. The effect of the long treatment time, had a significant effect on the reduction of several pesticide residues in all procedures. The results show that water treatments could be useful for the partial removal of several pesticide residues from grapes under both household and industrial conditions.To the best of our knowledge, this paper reports for the first time the effectiveness of water technologies for the removal of several pesticide residues from grapes. The data from this study helps in the estimation of processing factors for pesticides in specific processes. These values will complement the limited databases and aid in risk assessments of processed grapes. With the growing need to identify food safety hazards, this type of study is required for a more realistic estimation of the dietary intake of the pesticides.

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