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Effect of different household methods for removing off insecticides residues from cabbage heads

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Abstract
Supervised field trials were carried out to investigate the effects of household processing to eliminate the imidacloprid, lambda cyhalothrin and spiromesifen residues in cabbage heads. Individual spray applications of imidacloprid, lambda cyhalothrin and spiromesifen were made at recommended dose. After spray, cabbage heads were collected at 1, 3 and 5 day interval and were subjected to various decontamination processes like tap water washing, saline water washing (2% NaCl), lukewarm water washing, open pan cooking, microwave cooking and outer leaf removal. Further, after processing the head samples were examined using QuEChERS technique and residues were estimated by GC-MS, ECD and HPLC. Among various decontamination processes, microwave cooking was found most effective which provided 92.95-100 percent relief from insecticide residues in cabbage heads followed by outer leaf removal (80.31-100%), open pan cooking (74.01-100%), saline water washing (42.45-100%), lukewarm water washing (29.24-100%) and tap water washing (23.27-100%).

Keywords: Cabbage, decontamination, imidacloprid, spiromesifen, lambda cyhalothrin

1. Introduction
Vegetables are an inseparable integral part of the human diet, and they are served in a number of different ways throughout the country. They are perhaps the most important sources of essential biochemicals and vital nutrients such as carbohydrates, carotene, proteins, vitamins, calcium, iron, ascorbic acid which is a prerequist for a healthy diet (Chandra *et al.*, 2015) [2]. Among different vegetables, crucifers are important winter crop consist of cabbage, cauliflower, broccoli, mustard and radish. Cabbage, *Brassica oleracea* var. *capitata* L. is the main temperate crucifer crop that cultivates widely in different climatic regions around the world. In worldwide production, India ranks second after China and occupy 5% of the total vegetable-growing area in India (Anonymous, 2016) [15]. As cabbage is grown more vigorously, it has a higher incidence of pest and diseases, which lowers its net production and consumption. Different major pests which attack cabbage crop are *Pieris brassicae* L. (Lepidoptera: Pieridae), *Plutella xylostella* L. (Lepidoptera: Plutellidae), *Brevicoryne brassicae* L. (Hemiptera: Aphididae) and *Trichoplusia ni*. Hübner (Lepidoptera: Noctuidae). Among all insect pests the most damaging is *P. xylostella*, which can reduce cabbage yield by 52 percent in India (Krishnamoorthy, 2004) [7].

In order to control insect pests in cabbage, use of various synthetic pesticides is necessary for the protection of vegetable crops (Shah *et al.*, 2019) [10]. However, because of the pesticides widespread usage and efficiency against a wide range of pests, farmers are tempted to employ them against pests of other crops for which they are not indicated, resulting in toxic residues on the treated crop at harvest. Before being permitted for use on crops, pesticides must pass a series of rigorous regulatory procedures. The application of pesticides on crops in an excessive and indiscriminate manner, exposing farm workers and consumers to insecticide residues. As a result, a significant amount of pesticides absorbed by different vegetable crops reaches the human body, causing various health risks. As a result, a significant amount of pesticides acquired by different crops penetrates the human body, posing a number of health risks. Removal of these residues is important before consumption of vegetables. The impact of various commercial processing on pesticide residue in food has attracted the interest of many scientists and food manufacturers (Gowda and Somashekar, 2012) [4]. Several investigations analyzed the effects of common household processing on various types of produce and found that residues were reduced as a result of various treatments (Chandra *et al.*, 2015; Joshi *et al.*, 2015) [2, 6].

So the current investigation was made to remove the pesticide residues by following different household preparations *viz.*, tap water washing, lukewarm water washing, saline water washing, open pan cooking, microwave cooking and outer leaf removal in cabbage head samples.

2. Materials and Method
2.1 Chemicals and Reagents
Certified Reference Material (CRM) for all the insecticides procured from M/S J. Kundan and Company, Mumbai, India. All the analytical grade reagents viz. acetone, acetonitrile, n-hexane, magnesium sulfate, sodium chloride, sodium sulphate utilized in the following investigation were obtained from Merck Specialties Pvt. Ltd, Mumbai, India. Primary secondary amines had been procured from M/s Agilent Technologies Pvt Ltd, Worli, Mumbai, India.

2.2 Preparation of standard solution
Both lambda cyhalothrin and spiromesifen standard stock solutions (400 mg/L) were made in acetone–hexane (5:95, v/v), while imidacloprid was prepared in acetonitrile. Working standards of 40, 10 and 1 mg/kg were obtained from each stock solution by serial dilutions in n-hexane (lambda cyhalothrin and spiromesifen) and acetonitrile (imidacloprid). Working standard solutions of 0.025, 0.05, 0.1, 0.25, 0.5, and 1 mg/L were made by serial dilution of analytical standard solution and were spiked into samples.

2.3 Pesticides and application rate
The technical grade analytical standards of imidacloprid (99.8% purity), lambda cyhalothrin (98.5% purity) and spiromesifen (98.9% purity) were procured from M/S J. Kundan and Company, Mumbai, India. Imidacloprid (Confidor® 17.8% SL), spiromesifen (Oberon® 240 SC) and lambda cyhalothrin (Reeva-5® 5% EC) formulations were used for field application and were also obtained from M/S Bayer Crop Science, Mumbai and Rallis India Ltd., respectively.

2.4 Field Experiment
The following investigation was laid out at the experimental farm of the Department of Entomology, UHF Nauni, Solan (H.P.) which is situated at 33.3° N latitude and 70.70° E longitude with an elevation of about 1,260 m. Seeds of cabbage (Golden acre) were raised and transplanted in field in November as per university standard package of practices (Anonymous, 2015) [1]. Crop was sprayed with individual insecticides (imidacloprid, lambda cyhalothrin and spiromesifen) twice at an interval of 10 days at recommended dose (25, 15 and 96 g a.i./ha) in March with the help of Knapsack sprayer. Untreated plots were sprayed with water only.

2.5 Sampling procedure
After second spray of individual insecticide about 2 kg of head sample was collected randomly at an interval of 1, 3 and 5 days from each replication separately, packed in polythene bags and bought to laboratory for further processing and were subjected to different decontamination processes as shown in Table 1

Table 1: Different decontamination processes

Treatment	Method	Time
Washing		
1. Tap water washing	Heads washed under running tap water	5 minutes
2. Lukewarm water washing	Heads dipped in lukewarm water and hand rubbed	5 minutes
3. Saline water washing	Heads dipped in water containing 2% NaCl and hand rubbed	5 minutes
Cooking		
1. Microwave cooking	Head samples cooked at 800W power output in microwave	5 minutes
2. Open pan cooking	Heads cooked in an open pan of 1 liter capacity	10-15 minutes
Removal of Outer Leaf	Outer 2 leaves removed from heads	-

2.6 Extraction and Clean up
Processed cabbage head samples were examined according to the modified QuEChERS method (Sharma, 2013) [11]. 2 kg of cabbage heads were crushed and homogenized by using high-volume homogenizer (Blixer® 6 V. V, France). A representative 15 g sample was taken in a centrifuge tube (50 ml) containing 30 ml of acetonitrile and was homogenized at 15000 rpm with the help of low volume high speed homogenizer (Heidolph Silent Crusher M.) for 3 minutes. Sodium chloride (3 g) was added into the tube containing homogenized sample and later it was shaked in Rotospin (Tarson) at 50 rpm speed for 5 minutes. At 3000 rpm the homogenized mixture was centrifuged (Eppendorf, centrifuge) for 3 minutes. 18 mL of the organic fraction from the upper layer was introduced to a 50 ml tube comprising 9 g anhydrous sodium sulphate. Thereafter, 11 ml of the filtrate was added to a 15 ml centrifuge tube containing 0.4 g PSA and 1.15 g anhydrous magnesium sulphate, which was stirred (50 rpm) and centrifuged for 3 minutes at 3000 rpm. Finally, the resultant 6 ml extract was transferred to a 30 ml turbo glass tube and allowed to evaporate in the TurboVap® at 45 °C under nitrogen current.

2.7 Residue estimation
Dried imidacloprid residues were diluted in 3ml of acetonitrile: water (30:70) mixture and injected (20 µl) into a High Performance Liquid Chromatography (HPLC) (SHIMADZU LC-20AT) equipped with DGU-20A5 degasser, auto injector SIL-20 AHT, SPD-M20A Photodiode Array Detector (PDA) and C_{18} (5 µm) column (2.1 mm X 30 cm) for further analysis. Imidacloprid was detected at 270 nm wavelength.
Residues of lambda cyhalothrin and spiromesifen were dissolved in 3 mL n-hexane and injected (1 µl) into a gas chromatograph (GC) (Shimazdu GC 2010) equipped with capillary column (DB-5, 30m long, 0.25mm ID. and 0.25 µm film thickness), coupled with mass spectrometer (GCMS-QP 2010 plus) for spiromesifen analysis and an electron capture detector (ECD) for lambda cyhalothrin with different operating parameters. For spiromesifen, initial temperature was kept at 175 °C for 1 minute, raised to 250 °C @ 20 °C/min and hold for 5 minutes. For lambda cyhalothrin,

initially temperature was kept at 170 °C for 5 minutes, raised to 220 °C at the rate of 30 °C/min with a hold time of 10 minutes. Then, again temperature of oven was raised to 280 °C at the rate of 4 °C/min with a hold of 7 minutes. Under such system parameters, retention time of imidacloprid, lambda cyhalothrin and spiromesifen was 6.90, 24.81 and 24.74, respectively. For imidacloprid and lambda cyahlothrin the limit of determination (LOD) was 0.05 mg/kg and 0.025 mg/kg in case of spriomesifen.

From each replication, imidacloprid, lambda cyhalothrin and spiromesifen residues were assessed and mean residues were calculated. The following calculation was used to compute the percent relief from residues in each treatment based on the mean residues:

$$\text{Percent relief} = \frac{100 - \text{Residue in processed sample (mg/kg)}}{\text{Residue in unprocessed sample (mg/kg)}} \times 100$$

3. Results and Discussion

3.1 Method validation

In order to validate the analytical method, recovery experiments were carried out at different levels. Cabbage head control samples were spiked in the range of 0.05-1.00 mg/kg, for imidacloprid and lambda cyhalothrin and 0.025-1.00 mg/kg for spiromesifen, respectively. The average recovery values of imidacloprid, lambda cyhalothrin and spiromesifen were in range of 97.10-109.00, 95.20-108.00 and 94.00-100.00 percent, respectively in cabbage heads. The recovery obtained in all the insecticides in cabbage heads and soil was in the acceptable range of 80-120 percent as per SANTE guidelines (SANTE, 2019) [9], thus establishing the suitability of method for further studies.

3.2 Decontamination

3.2.1 Effect of washing

Washing is one of the household activities which play an important role in reduction of the insecticide residues. In the present study effect of different washing was recorded to see their effect in reducing the insecticide residue. Data in the Table 2 showed that when the treated cabbage heads were collected from the experimental field at 1, 3 and 5 days interval and treated under running tap water for 5 minutes, the imidacloprid and lambda cyhalothrin (1 and 3 days) and spiromesifen (1, 3 and 5 days) residues reduced to 0.189 and 0.081 mg/kg, 0.105 and 0.067 mg/kg, 0.552, 0.279 and 0.178 mg/kg, respectively for the given days and also showed the percent relief from residues as 25.59 and 23.58 percent, 25.53 and 23.86 percent, 25.20, 28.64 and 23.27 percent on respective days. Imidacloprid residues reduced upto 0.121 mg/kg after treating okra fruits under running tap water (Srivastava *et al.*, 2021) [14]. Washing of field bean with tap water washing reduced lambda cyhalothrin and imidacloprid by 28.77 and 22.74 percent, respectively (Srinivasa *et al.*, 2018) [13]. Washing of tomato fruits under running tap water reduced lambda cyhalothrin residues by 39.0 and 30.0 percent from 0 day and 3rd day samples, respectively (Jayakrishnan *et al.*, 2005) [5] . In other method of washing cabbage heads were dipped in lukewarm water (36-40 ℃) for 5 minutes which reduced the imidacloprid, lambda cyhalothrin and spiromesifen residues to 0.170 and 0.075 mg/kg, 0.085 and 0.055 mg/kg and 0.449, 0.233 and 0.139 mg/kg, respectively for above given days with percent relief of 33.07 and 29.24 percent, 39.71 and 37.50 percent and 39.15, 40.40 and 40.08 percent on respective days.

In another method, treated cabbage heads when dipped in saline water solution (2% NaCl) for 5 minutes the imidacloprid, lambda cyhalothrin and spriromesifen residues reduced to 0.132 mg/kg and 0.061 mg/kg (1 and 3 days), 0.051 mg/kg (1 day) and 0.368, 0.196 and 0.119 mg/kg (1, 3 and 5 days), respectively which give the percent relief of 48.03 and 42.45, 63.82 percent and 50.13, 49.87 and 48.70 percent on respective days. Similar to this, a study was conducted which revealed that washing of okra with lukewarm water and dipping in NaCl solution reduced imidacloprid residues upto 0.103 mg/kg and 0.074 mg/kg (Srivastava *et al.*, 2021) [14]. Dipping of brinjal fruits in 2 percent NaCl solution for 10 minutes reduced lambda cyhalothrin by 48 percent which was observed by Cherukuri *et al.* (2014) [3].

3.2.2 Effect of cooking

Another important household practice is cooking which helps in reducing the residues to greater extent. Open pan cooking of treated cabbage heads for 10-15 minutes showed that the imidacloprid residues reduced to 0.066 mg/kg in 1 day and reduced to below determination limit (BDL) in 3rd day samples. On the other side lambda cyhalothrin residues reduced to below the limit of determination in 1st, 3rd and 5th day of treated heads and spiromesifen residues to 0.111 and 0.079 mg/kg from 1 and 3 day old samples which showed 84.95 and 81.84 percent relief from spiromesifen residues, respectively. Another method in which the treated head samples were cooked in microwave (800 W) for 5 minutes reduced imidacloprid and lambda cyhalothrin residues to below determination limit from 1 day old treated samples and reduced spiromesifen residues to 0.052 mg/kg in 1 day sampled heads and showed 92.95 percent relief whereas residues were below the determination limit in 3rd and 5th day treated samples. Present study showed that microwave cooking had better effect in reducing the insecticides residues in comparison to the open pan cooking. In relation to this study, Sharma (2016) [12] also concluded that chlorpyriphos residues in capsicum fruits were reduced when cooked under microwave conditions (43.45-53.11%) in comparison to cooking in open pan (36.23-49.90%).

In another method i.e. removal of outer leaf revealed the imidacloprid residues reduced upto 0.05 mg/kg from 1st day samples and below the limit of determination in 3rd day samples and showed percent relief of 80.31 and 100.00 percent, respectively. In case of spiromesifen residues were reduced to 0.132 mg/kg in 1st day samples and below the limit of determination in 3rd and 5th day samples. Lambda cyhalothrin residues were below the limit of determination in 1st, 3rd and 5th day samples. Some studies showed that peeling is effective in reducing the insecticide residues both in fruits and vegetables. Liu *et al.* (2014) [8] reported that peeling process yielded 87.3 percent loss in carbendazim residues from tomato fruits.

Table 2: Effect of culinary processes on imidacloprid, lambda cyhalothrin and spiromesifen residues in cabbage heads

Treatments	Interval (Days)	Imidacloprid		Lambda cyhalothrin		Spiromesifen	
		Residues (mg/kg) Mean±SD	Percent Relief	Residues (mg/kg) Mean±SD	Percent Relief	Residues (mg/kg) Mean±SD	Percent Relief
Unprocessed	1	0.254±0.042	-	0.141±0.006	-	0.738±0.022	-
	3	0.106±0.010	-	0.088±0.005	-	0.391±0.011	-
	5	BDL	-	0.065±0.004	-	0.232±0.011	-
Tap Water washing	1	0.189±0.006	25.59	0.105±0.003	25.53	0.552±0.004	25.20
	3	0.081±0.005	23.58	0.067±0.005	23.86	0.279±0.004	28.64
	5	-	-	BDL	100.00	0.178±0.007	23.27
Lukewarm Water Washing	1	0.170±0.005	33.07	0.085±0.004	39.71	0.449±0.008	39.15
	3	0.075±0.003	29.24	0.055±0.004	37.50	0.233±0.008	40.40
	5	-	-	BDL	100.00	0.139±0.004	40.08
Saline Water Washing	1	0.132±0.005	48.03	0.051±0.001	63.82	0.368±0.006	50.13
	3	0.061±0.004	42.45	BDL	100.00	0.196±0.002	49.87
	5	-	-	BDL	100.00	0.119±0.004	48.70
Open Pan Cooking	1	0.066±0.004	74.01	BDL	100.00	0.111±0.004	84.95
	3	BDL	100.00	BDL	100.00	0.071±0.003	81.84
	5	-	-	BDL	100.00	BDL	100.00
Microwave Cooking	1	BDL	100.00	BDL	100.00	0.052±0.002	92.95
	3	BDL	100.00	BDL	100.00	BDL	100.00
	5	-	-	BDL	100.00	BDL	100.00
Outer Leaf Removal	1	0.05±0.001	80.31	BDL	100.00	0.132±0.004	82.11
	3	BDL	100.00	BDL	100.00	BDL	100.00
	5	-	-	BDL	100.00	BDL	100.00

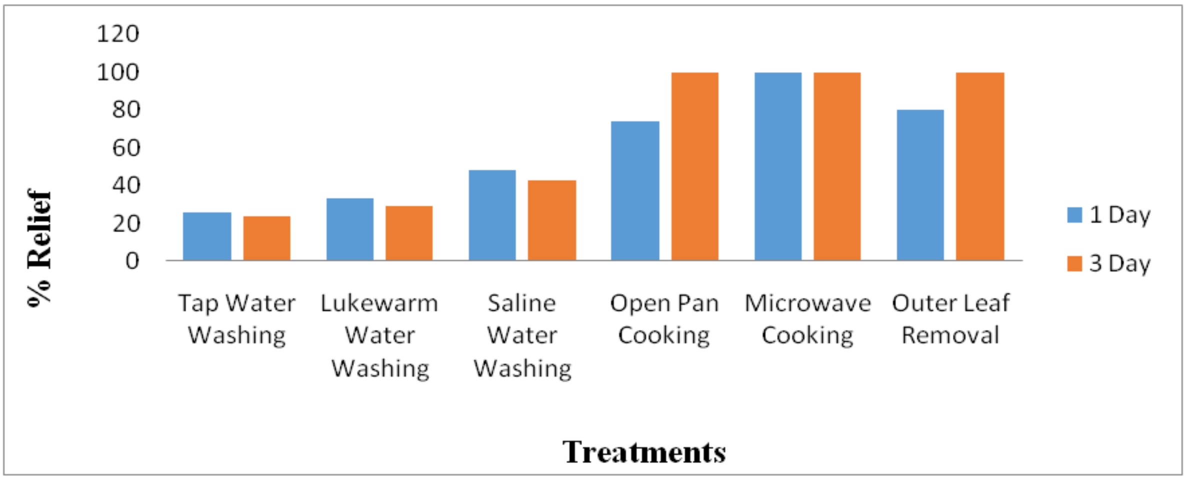

Fig 1a: Effect of culinary processes on imidacloprid residues in cabbage heads

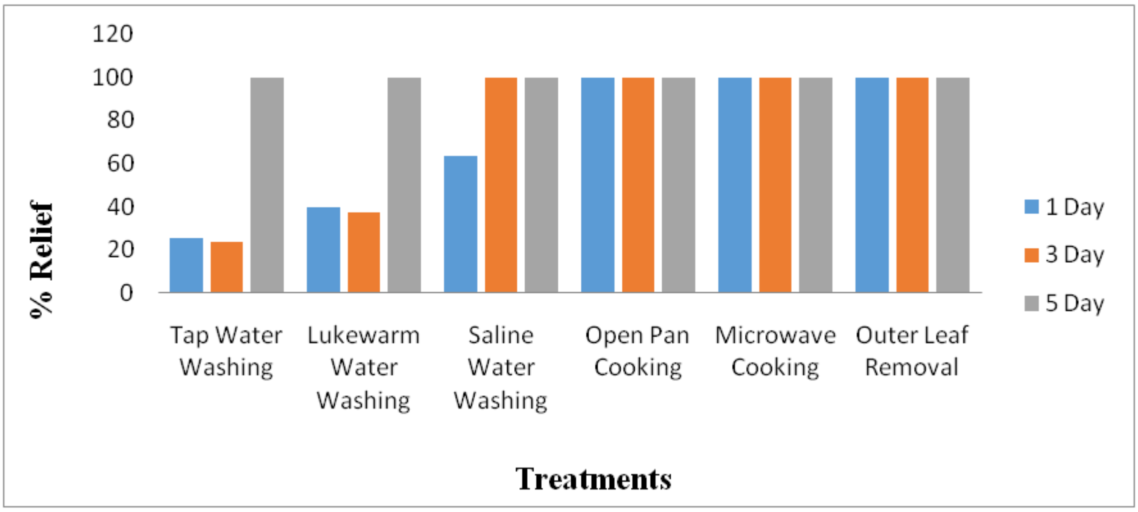

Fig 1b: Effect of culinary processes on lambda cyhalothrin residues in cabbage heads

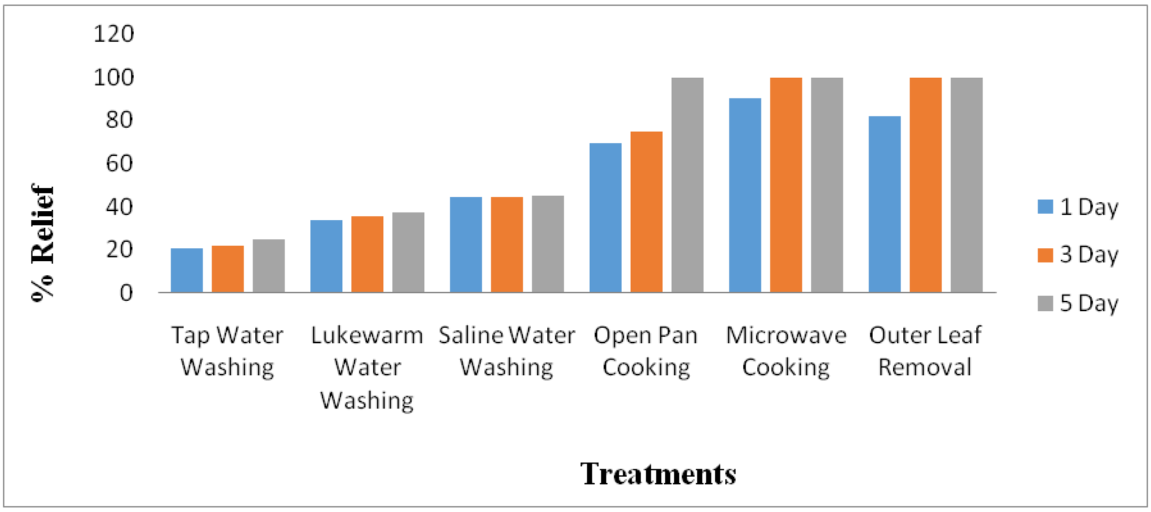

Fig 1c: Effect of culinary processes on spiromesifen residues in cabbage heads

4. Conclusion

Traditional processing was found to be an effective strategy for reducing pesticide residues, implying that traditional processing procedures can successfully reduce pesticide residues in vegetables. Pesticide residues in food are affected by storage, handling and processing which occurs after the harvest of raw agricultural commodities but before the consumption of prepared foodstuffs. Processing results in significant reductions in residue levels in prepared foods, particularly through washing and cooking operations. According to a critical study of complete decontamination data it was concluded that microwave cooking eliminated far more residues from contaminated cabbage heads than simple washings.

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