International Journal of Farm Sciences **8(3):** 44-50, 2018; doi: 10.5958/2250-0499.2018.00080.0

Decontamination of cypermethrin residues in tomato fruits and cauliflower curds

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 Received: 29.1.2018/Accepted: 12.4.2018

ABSTRACT

Field and laboratory experiments were conducted to evaluate the effect of different decontamination processes like washing, cooking, washing plus cooking and dipping in chemical solutions after application of Challenger 25EC (cypermethrin 25%) on reduction of cypermethrin residues in tomato fruits and cauliflower curds. Challenger 25EC was applied twice at the rate of 0.2 ml/l at 15 days interval during each cropping season. Tomato fruits and cauliflower curds were collected at 0 (2 hours) and 3 days interval after the last spray and subjected to decontamination processes. Washing of zero day-contaminated tomato fruits provided 40.89-70.44 per cent relief from cypermethrin residues and 38.15-68.20 per cent relief in cauliflower curd samples. Cooking degraded residues up to 52.24-66.32 per cent in tomato fruits and 43.19-63.11 per cent in cauliflower curds. Washing plus cooking removed cypermethrin residues up to 70 per cent both in tomato and cauliflower as compared to other processes and proved to be the best technique for removing the residues. Washing of fruits with 2 per cent NaOH solution reduced the residues up to 64.60-70.44 per cent whereas with 0.05 per cent solution of HCl up to 65.16-68.04 per cent. Similarly in case of curds residues were reduced to 61.50-68.20 per cent after treatment with 2 per cent NaOH solution and up to 62.44-65.97 per cent with 0.05 per cent HCl solution.

Keywords: Fruits; curds; processing; tomato; cauliflower; residues; decontamination

INTRODUCTION

Vegetables are the inseparable component of Indian cuisine and are consumed throughout the country in different forms and preparations. They are the major source of vitamins and nutrients hence they fulfill the requirements of our balanced diet (Chandra et al 2015). Among the vegetables, tomato (*Solanum lycopersicum* L) and cauliflower (*Brassica oleracea* var *botrytis* L) are the important cash crops and give better return to the farmers of Himachal Pradesh and are infested by a large number of insect-pests and diseases (Sharma and Bhalla 1964, Sharma 1975, Bhalla and Pawar 1977). Tomato is attacked by many insect pests like tomato fruit borer, mites, leaf miner, aphids, whiteflies etc and the key pests of cauliflower are diamond back moth, leaf eating caterpillars and aphids (Regupathy et al 1985, Patel et al 1999) thus affecting both the quantity and quality of fruits as well as curds. In a desperate bid to save the crop farmers sometimes apply the pesticides repeatedly and at higher doses hence the repeated and intensive use of insecticides has lead to the development of resistance in insect pests (Brar et al 2017). In Himachal Pradesh cypermethrin insecticide has been used extensively by the farmers to control these major insect-pests of both tomato and cauliflower crops. Since the effect of pesticides is considered more toxic thus extra care should be taken to reduce the health hazards to the consumers (Regupathy et al 2004). The application of these pesticides near to harvest can leave residues on the fruits and curds which may be harmful to the consumers (Banshtu et al 2015).

Tomato fruits are picked frequently at short intervals and both the tomato fruits and cauliflower curds are consumed as cooked or raw vegetables hence chances of carrying pesticide residues to the consumers are more (Raj et al 1991). Hence pesticide residues in tomato and cauliflower are of major concern to

consumers due to their negative health effects. They have been found in both raw and processed fresh produce. There have been various reports suggesting use of different simple household processes in dislodging pesticide residues from food commodities thus making them safe for human consumption (Sharma et al 1994, Aktar et al 2009, Chavarri et al 2005, Dejonckheere et al 1996, Elkins 1989, Krol et al 2000, Schattenberg et al 1996). Operations such as washing, peeling, blanching and cooking play a crucial role in the reduction of residues (Elkins 1989, Kaushik et al 2009). Each operation has a cumulative effect on the reduction of the pesticides (Geisman 1975).

In the present scenario it is very important that some pragmatic solution is developed to tackle this problem of food safety. Food safety is an area of growing concern worldwide on account of its direct bearing on human health. The presence of harmful pesticide residues in food such as tomato and cauliflower has caused a great concern among the consumers. Therefore the present investigations were contemplated with the objective to study the effect of different decontamination processes in tomato fruits and cauliflower curds for the reduction of cypermethrin residues after its application on both the crops in the field. The techniques used in the present study focused on commercial and home processing of tomato and cauliflower and they included washing alone, washing with chemicals, cooking and washing followed by cooking.

MATERIAL and METHODS

Chemicals and reagents: Challenger 25EC containing 25 per cent cypermethrin was obtained from M/S Tropical Agrosystem Ltd and reagents like acetone, dichloromethane, hexane, toluene, sodium chloride, sodium sulfate anhydrous (AR grade), Celite 545 and Florisil were all procured from M/S Merck Specialities, Mumbai, Maharashtra, India. All common solvents were redistilled in an all-glass apparatus before use.

Field trials: Tomato (*Solanum lycopersicum* L) var Heem Sohna fruits and cauliflower (*Brassica oleracea* var *botrytis* L) were raised during 2009 and 2010 respectively at experimental farm of Department of Entomology, Dr YS Parmar University of Horticulture and Forestry, Nauni, Solan, Himachal Pradesh following recommended agronomic practices (Anon 2009). The experiment was conducted in randomized block design with three replications for each treatment. The first application of Challenger 25EC (cypermethrin 25%) @ 0.2 ml/l of water was made at tomato fruit and caulifloer curd formation stage followed by second application at an interval of 15 days. In control plots only water was sprayed. Pesticide was sprayed as foliar application in three replications with the help of a knapsack sprayer fitted with a hollow cone nozzle.

Sampling procedure: Fruit and curd samples (1 kg) from each replication were collected randomly at 0 (2 hours after spray) and 3 days interval after last foliar application. The samples were collected randomly, packed in bags and brought to the laboratory for processing.

Decontamination processes: Samples collected from the field were subjected to different decontamination processes viz washing, cooking and washing followed by cooking (Patyal et al 2004).

Washing

- Tomato fruits and cauliflower curds were washed under running tap water and hand-rubbed for 2 minutes.
- Samples were dipped in lukewarm water (50^{o}C) for 5 minutes and then placed on filter papers for drying.
- Samples were dipped in 2 per cent NaCl (w/v) solution for 5 minutes followed by tap water washing.
- Samples were dipped in 2 per cent lukewarm salt solution (w/v) for 5 minutes followed by water washing.
- Samples were dipped in 0.05 per cent HCl (v/v) for 5 minutes followed by water washing
- Samples were dipped in 2 per cent (w/v) sodium hydroxide solution for 5 minutes followed by water washing.

Cooking

- **Open pan cooking:** Unwashed samples from each replication were chopped and put in an open pan of one litre capacity containing 500 ml water and boiled till softness (10-15 minutes).
- **Steam cooking:** Samples were chopped and steamed for 5 minutes in a pressure cooker.
- **Microwave cooking:** Samples were kept in microwave at 1400 W power output for 5 minutes for cooking .

Washing followed by cooking

- **Washing + cooking:** Samples were washed by hand-rubbing under a stream of running tap water for 2 minutes followed by boiling in an open pan of one litre capacity containing 500 ml water till softness (10-15 minutes).
- **Washing + steam cooking:** Samples were washed under running tap water and steamed for 5 minutes in a pressure cooker.
- **Washing + microwave cooking:** Samples were washed under the tap water and placed in microwave at 1400 W power output for 5 minutes for cooking .

Extraction and cleanup: After completing decontamination process samples were extracted and cleaned up according to the method of Sharma (2007). The samples were processed and analyzed in the Department of Entomology, Dr YS Parmar University of Horticulture and Forestry, Nauni, Solan, Himachal Pradesh. Processed tomato fruits and cauliflower curd samples were homogenised in a domestic mixture.

A representative 100 g homogenised sample was taken with 200 ml acetone in a 500 ml conical flask and kept overnight. The extract was filtered through Buchner funnel by fitting a Whatman # 1 filter paper. An aliquot of 60 ml (30 g equivalent) of sample was transferred to 1 litre separatory flask and extracted with 200 ml mixture of hexane and dichloromethane (1:1, v/v). The lower aqueous phase was transferred to another 1 litre separatory funnel containing ten millilitre saturated sodium chloride solution and partitioned twice with 100 ml dichloromethane. Lower aqueous phase was discarded and upper organic phase was pooled with first organic fraction. Pooled organic phase was passed through anhydrous sodium sulfate and evaporated to dryness at 45°C by using vacuum rotary evaporator. Finally the residues were taken up in 3 ml (1 + 2) acetone for cleanup. Samples for cypermethrin residues were cleaned up on Florisil column.

Sample fraction of 1 ml was diluted with 10 ml of acetone:hexane (1:9) mixture loaded on the 4 g activated Florisil column overlaid with 2 g sodium sulfate. The column was eluted with 50 ml eluent (50% dichloromethane:48.5% hexane:1.5% acetonitrile). Eluant was evaporated to dryness, residues were dissolved in 1 ml n-hexane and injected into gas chromatograph.

Residue estimation: Residues of cypermethrin were estimated by using gas-chromatograph (Agilent 6890N) having ECD detector and DB-5 ultra performance capillary column (cross-linked methyl silicon, length 30 m, 0.25 mm internal diameter with 0.25 µm film thickness). Oven temperature was programmed at 100°C for 1 minute, 30°C/minute up to 150°C, 3°C/minute up to 205°C and finally 260°C at rate of 10°C/minute. Injection port and electron capture detector (ECD) temperature were kept at 250 and 300°C respectively.

Cypermethrin residues (mg/kg) were determined for each replication and mean residues were calculated. Per cent relief from residues in each treatment was calculated from the mean residues by the following equation:

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

Validation of analytical method: The analytical method employed to estimate cypermethrin residues was spiked at 0.05, 0.10, 0.20, 0.50 and 1.00 mg/kg concentrations. Recovery of cypermethrin was between 88.00-90.00 per cent with relative standard deviation (RSD) of 0.034-0.738 per cent in fruits of tomato and for cauliflower curds recovery was between 87.00-92.00 per cent with RSD 0.049-1.003 per cent (Table 1).

RESULTS and DISCUSSION

Effect of washing

Washing is the most common form of processing which is a preliminary step in both household and commercial preparations. Loosely-held residues of several pesticides are removed with reasonable efficiency by varied types of washing processes (Street 1969). Washing of 0-day samples under running tap

water provided 40.89 per cent relief from cypermethrin residues for tomato fruits whereas 33.70 per cent relief was observed in 3-day old samples of tomato fruits (Fig 1). Similar observations were recorded after washing of cauliflower curds treated with cypermethrin (Fig 2). Aktar et al (2010) reported that washing of cabbage head under running tap water removed 27.72-32.48 per cent quinalphos residues. Similarly Singh et al (2004) also found that washing of okra fruits with tap water could remove the residues of cypermethrin to the extent of 36.25-42.76 per cent. Cengiz et al (2006) found that the initial diazinon residue level (0.822 ppm) on cucumbers was decreased by 22.3 per cent by rubbing under running water for 15 seconds.

Lukewarm water washing of 0-day sampled tomato fruits provided 46.39 per cent relief whereas 35.39 per cent relief from cypermethrin residues was observed in 3-day old samples (Fig 1). Similar observations were recorded after washing of cauliflower curds treated with cypermethrin (Fig 2). Kumari (2008) also reported 32-100 per cent reduction of organophosphorus insecticide residues by lukewarm water of cauliflower.

Chemical washing

Washing of treated tomato fruits with sodium hydroxide and hydrochloric acid provided a good relief from cypermethrin in comparison to washing with sodium chloride and lukewarm sodium chloride solution. It may be due to hydrolytic property of cypermethrin in strong acids and alkalies (Tomlin 1997). Sodium hydroxide provided 70.44 and 64.60 per cent relief from cypermethrin in 0- and 3-day processed fruits respectively. Dip treatment of tomato fruits with hydrochloric acid gave 68.04 and 65.16 per cent relief from cypermethrin residues. Similar observations were recorded after chemical washing of cauliflower curds treated with cypermethrin. The present findings are in agreement with those of Patyal et al (2004) who found that washing of treated apple fruits with 2 per cent (w/v) NaOH and 0.05 per cent (v/v) HCl gave 77.06 and 75.96 per cent relief respectively from endosulfan residues. Dipping of brinjal fruits in hydrochloric acid solution, acetic acid solution and sodium hydroxide solution reduced 40-45 per cent residues of synthetic pyrethroids namely cypermethrin and deltamethrin (Awasthi 1986).

Dipping of tomato fruits samples in 2 per cent sodium chloride solution (w/v) reduced cypermethrin residues to 48.45 and 43.82 per cent whereas lukewarm sodium chloride solution reduced residues to 62.54 and 55.05 per cent respectively which is in agreement with the findings of Mukherjee et al (2006) who also observed that dipping of cauliflower curds in 1 per cent brine solution followed by washing reduced the residues by 39.6 per cent while in case of 1 per cent hot brine solution the reduction was 55.0 per cent. Similar observations were recorded after chemical washing of cauliflower curds treated with cypermethrin.

Cooking

Application of heat to the food commodities is commonly done through ordinary cooking, pressure cooking, microwave cooking, frying, sterilization and canning. The effect of different cooking processes on removal of cypermethrin residues in tomato and cauliflower were studied (Figs 1, 2). In all of the processes cooking with pressure was found to be more effective than others. Pressure cooking reduced the residues up to 66.32 per cent. These results are in accordance with the findings of Muthukumar et al (2010) who also reported that pressure cooking was the most effective in reducing both alpha- and beta-

Table 1. Recovery of cypermethrin from tomato fruits and cauliflower curds

Crop	Fortification level (mg/kg)	Mean recovery (%)	Relative standard deviation (%)
Tomato fruits	0.05	88.00	0.738
	0.10	88.00	0.286
	0.20	89.00	0.202
	0.50	89.00	0.147
	1.00	90.00	0.034
Cauliflower curds	0.05	90.00	1.003
	0.10	88.00	0.933
	0.20	90.00	0.704
	0.50	87.00	0.104
	1.00	92.00	0.049

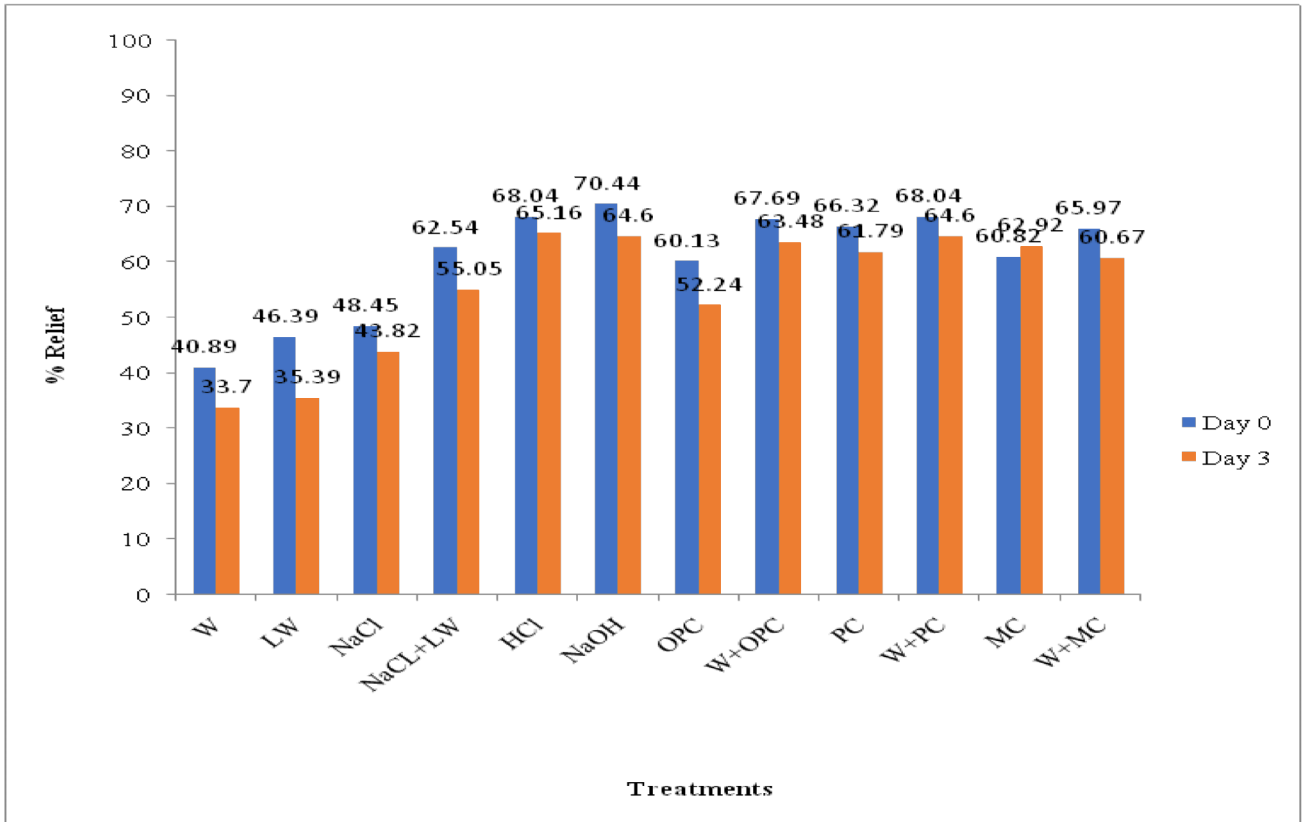

Fig 1. Per cent relief from cypermethrin residues from tomato fruits using different decontamination processes (W= Tap water washing, LW= Luke warm, OPC= Open pan cooking, PC= Pressure cooking, MC= Microwave cooking)

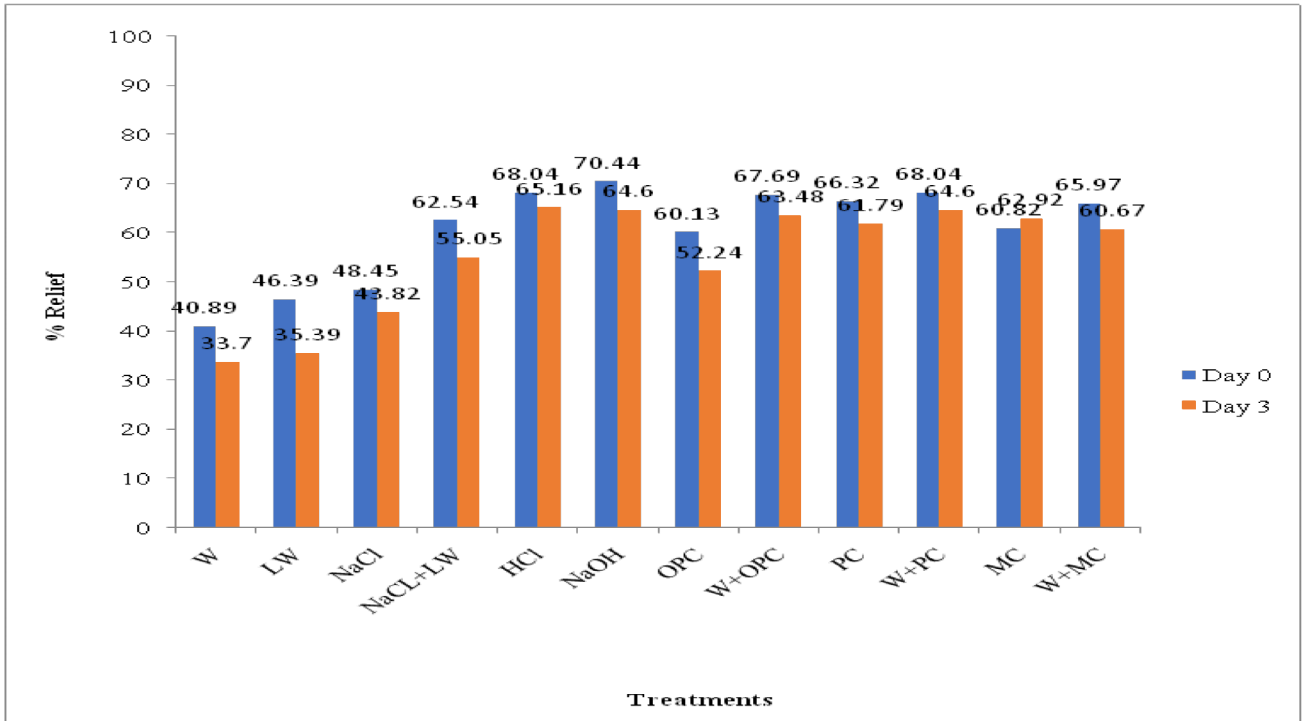

Fig 2. Per cent relief from cypermethrin residues from cauliflower curds using different decontamination processes (W= Tap water washing, LW= Luke warm, OPC= Open pan cooking, PC= Pressure cooking, MC= Microwave cooking)

endosulfan by 64.59 and 61.60 per cent respectively as compared to boiling and microwave cooking.

Cooking of tomato fruits and cauliflower curds in open pan or under pressure or in the microwave resulted in 50-65 per cent relief from cypermethrin residues. The findings are in agreement with the work of Dikshit (2001) who observed that process of steaming dislodged the cypermethrin residues by 63-74 per cent on stored pulses treated at 3 and 5 mg/kg levels. The disappearance of pesticide residues from boiling extract could be due to decomposition by the effect of heat, the stronger adsorption of pesticide onto plant tissues and/or the poor solubility of pesticides in water (Abou-Arab and Abou-Donia 2001, Ali 1983). Walia et al (2010) reported that microwave cooking reduced cypermethrin residues to the extent of 40.89 per cent in brinjal sprayed at 0.001 per cent concentration. Hence processes involving heat can increase volatilization, hydrolysis or other chemical degradation and thus reduce residue levels (Holland et al 1994).

Washing followed by cooking

Washing is generally the first step in various types of treatments which are given to food commodities in combinations like washing followed by cooking, washing and drying, washing and peeling and washing, peeling and juicing to allow for effective decontamination from pesticides (Kaushik et al 2009).

Washing of tomato fruits and cauliflower curds followed by cooking lead to more than 65 per cent removal of cypermethrin residues (Figs 1, 2). Similarly Mukherjee et al (2006) also reported that the washing of cauliflower heads under running tap water removed 27.9 per cent chlorpyrifos residues, cooking reduced residues to 41.4 per cent and washing + cooking further reduced residues to 66.7 per cent. Aktar et al (2010) also reported that washing plus cooking of cabbage heads reduce more quinalphos residues (66.45-68.19%) in comparison to washing alone (41.30-45.20%).

CONCLUSION

A critical analysis of whole decontamination data revealed that the washing plus pressure cooking removed much higher residues from contaminated tomato fruits and cauliflower curds as compared to the simple washings. Although sodium hydroxide and hydrochloric acid treatments were superior over all other decontamination processes but such treatments can be used in the industries where large quantities of vegetables are processed for decontamination. Washing of vegetables with water followed by pressure cooking removed maximum residues up to 79 per cent as compared to the other processes and proved a good household practice.

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