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Comparative evaluation of decontamination processes for mitigating profenofos residues in chili (*Capsicum annuum*)

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Abstract

A study to assess the efficacy of ten decontamination processes in reducing the pesticide residue load of profenofos from chili (*Capsicum annuum* L.) was undertaken at College of Basic Science and Humanities, G. B. Pant University of Agriculture and Technology, Pantnagar, during September 2022 to April 2023. The extraction of insecticide from chili was initiated after 48 hr of pesticide formulation spray and was done using the QuEChERS (quick, easy, cheap, effective, rugged, and safe) method. The study included a control treatment too in which the pesticide residues were extracted without following any decontamination procedure. The quantitative analysis of insecticide was done by coupled gas chromatography–electron capture detection. The results depicted reduction of pesticide residues in all decontamination treatments compared with the control. Solutions of 1% NaCl and soaking in lukewarm water served as efficient decontaminants for removal (up to 90%) of profenophos residues from chili. Among commercially available decontamination products, soaking in nimwash solution showed remarkable effectiveness followed by veggie clean and Arka Herbiwash.

Key words: Decontamination; Pesticide residues; Organophosphate; GC-ECD

Chili (*Capsicum annuum* Linn) is most valuable spice crops of India as it is the largest producer, consumer and exporter of chili. The yield of any horticultural crop can be enhanced through proper irrigation practice (Halder *et al.*, 2022).

Profenofos (O,O-diethyl O-quinoxalin-2-yl phosphorothioate), is a non-systemic organophosphorus insecticide widely used to control various insect pests on chili crops. Pesticides are often used to help increase the yield of pepper crops by reducing the damage caused by pests. This improves farmer's profitability in terms of chili production (Radwan *et al.*, 2005). The risk of contamination with pesticide residues increases significantly if pesticides are not applied correctly and pre-harvest interval is not properly determined and followed (Souza *et al.*, 2019). Pesticide residues present at the time of harvest can accumulate and bio-magnify as they move up the food chain (Rawat *et al.*, 2023). Consuming raw and cooked vegetables, such as chilies, is a common route of pesticide exposure (Keikotlhaile *et al.*, 2010). Growing evidence links pesticide exposure to an increased incidence of chronic diseases in humans, such as cancer, Parkinson's, Alzheimer's, multiple sclerosis, diabetes, cardiovascular diseases, chronic kidney disease, and premature aging (Mostafalou, and Abdollahi, 2013).

Hence, removing surface pesticide residues from vegetables is crucial. Decontamination treatments for fruits and vegetables to reduce pesticide residues have proven highly effective (Bhilwadikar *et al.*, 2019; Rani *et al.*, 2019 ; Cengiz and Certel 2012). Researchers have observed that certain pesticide residues can diminish with pre-harvest intervals and/or culinary practices such as washing, peeling, cooking, boiling, and storage (Cengiz *et al.*, 2007).In recent years, the presence of pesticide residues in harvested chilli has become increasingly important, presenting not only health challenges but also serving as a significant barrier to international trade (Rawat *et al.*, 2024). Therefore, study was undertaken to investigate the impact of various washing solutions on removal of profenofos residues present on field-treated chili crops.

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MATERIALS AND METHODS

The analytical grade profenofos standard was obtained as a certified reference material from IARI, New Delhi, India. The Formulation product (celcron 50% EC) was used in the field experiment and was purchased from the local market at Rudrapur, Uttarakhand, India. All chemicals and solvents used were of analytical grade (AR) or HPLC grade, and all glassware used was sourced from Borosil (India) or Rivera (India).

A standard stock solution of profenofos of 100 mg / kg was prepared by dissolving the insecticide in n-hexane. Serial dilutions covering a range of concentrations from 0.005 to 2 mg / kg were then prepared. The working standard of profenofos was injected three times at six linear concentrations, and the calibration curve was plotted by determining each peak area with its corresponding concentration. Pesticide concentrations were subsequently determined using this calibration curve and the regression equation derived from the linearity plot. The limit of detection (LOD) and limit of quantification (LOQ) were calculated on the basis of signal-to-noise ratio.

Profenophos was analyzed by GC (Trace 1110), of Thermo Fisher Scientific, Mumbai, India make, equipped with an electron capture detector and a capillary column (30 m by 0.25 mm internal diameter and film thickness of 0.25 μm). The column temperature was initiated at 100°C, increased at a rate of 20°C per minute to 180°C for 4 minutes, then at 5°C per minute to 270°C for 18 minutes and finally at 5°C per minute to 300°C. The injection volume was 1 μL, with injector and detector temperatures set at 250°C and 300°C, respectively. Nitrogen (99.99% purity) was used as the carrier gas at a flow rate of 1.2 ml per minute.

Chili crop (cv. Pant1) was planted in plots (5 m by 5 m) in triplicate. All the recommended cultural practices were followed. Upon maturity, plants were sprayed with Celcron 50% EC at a rate of 800 g a.i/ha. The control plots were not subjected to any spraying, and vegetable samples weighing approximately 250–300 g were harvested at 50%–60% fruiting stage for recovery studies. Recovery studies involved spiking untreated/control chili samples at three different concentrations i.e. 0.02, 0.1 and 0.2 mg/kg (LOQ, 5 LOQ and 10 LOQ respectively). Subsequent extraction and clean-up procedures for profenofos residues were conducted following modified QuEChERS method (Srivastava *et al.*2021) The percent recovery was calculated using the specified formula.

Recovery (%) = [Recovered concentration/Actual concentration] × 100

Accuracy was determined by performing five replicates at each recovery level, and the % relative standard deviation (RSD) was then calculated.

% RSD = (Standard Deviation/Mean) × 100

The extraction procedure followed was a s follows : Each 10 g portion of chopped vegetable sample was placed in triplicate into a 50 mL centrifuge tube along with 10 mL of distilled water and 10 mL of n-hexane. After vortexing for 2–3 min, the mixture was allowed to stand for 10 minutes. Then, 3 g of anhydrous magnesium sulfate and 1 g of sodium chloride were added, followed by shaking for an additional minute. The tubes were vortexed again and centrifuged at 3000 rpm for 5 min. For cleanup, dispersive solid-phase extraction (dSPE) was utilized. This involved adding 1 g of $MgSO_4$, 150 mg of PSA (primary secondary amine) reagent, and graphitized carbon black to the mixture, which was then centrifuged at 3000 rpm for another 5 min. The top supernatant layer was extracted using a pipette and passed through a dispersive solid-phase extraction (SPE) cartridge for further cleanup. The samples were then filtered through 0.45-μm-pore-size polytetrafluoroethylene disc filter membrane for analysis by GC-ECD.

For decontamination studies samples were collected from the plot, 48 hr after the application of Celcron 50% EC formulation and divided into eleven equal portions. Every portion was subjected to different cleansing treatment to investigate the impact of different culinary processes on reducing profenofos residues. These included washing with running tap water, soaking in lukewarm water, soaking in a 1% NaCl solution, soaking in a 5% $NaHCO_3$ aqueous solution, soaking in a 2% CH_3COOH solution, soaking in a 0.01% $KMnO_4$ solution, and dipping in commercially available decontaminant formulations (such as Veggi Clean, Nimwash, and Arka Herbiwash) for 10 min. Each treatment was replicated three times. Samples of chili without any processing (Control) were also analyzed for residues alongside the processed samples.

RESULTS AND DISCUSSION

The recovery of different pesticides at three concentrations in chilli crop ranged between 83.2 and 87.3% (Table 1), which denotes the acceptability of extraction method. The correlation coefficient (r) value of $r^2 \geq 0.99$ was obtained, which indicated a good linear relationship. The limit of detection and limit of quantification was 0.005 and 0.02 mg/kg^{-1} respectively. Additionally, retention times of profenofos remained consistently at 16.7 minutes.

Table 1: Validation data of standardized analytical method

Pesticide	Linear range (mg/ kg^{-1})	Variation coefficient	LOD (mg/ kg^{-1})	LOQ (mg/ kg^{-1})	% Recovery with RSD
Profenofos	0.005-2	0.999	0.005	0.02	83.2 ± 2.3 to 87.3 ± 1.7

Profenofos residues obtained in different treatments were calculated relative to pesticide residues in T0 where no treatment was done and the % decontamination was zero. As evident from the data, treating chili crop with 1% NaCl was the most effective method for removing profenofos residues (Table 2). The strong electrolytic properties of salt solutions with a net charge enable them to interact with pesticides, creating an attractive force that facilitates their removal from the vegetable matrix (Rasolonjatovo *et al.*

2017). Washing with lukewarm water was the second most effective treatment for removing residues, slightly more effective than using running tap water. This effectiveness is likely due to slight solubility of profenofos in water, influenced by its high oil-water partition coefficient and low vapor pressure. Soaking in an acetic acid solution was also effective, removing residues up to 80%.

Adding reagents such as CH_3COOH and NaCl to washing water can significantly decrease pesticide residues by affecting the chemical bonds between the pesticide and the plant surface (Srivastava *et al.* 2022). It is therefore likely that cations (H^+ from acetic acid or Na^+ from sodium chloride) present in decontaminants might bind with charged moieties on cuticular surface of chili fruits and expel the pesticides sorbed on its surface, reducing the firmness with which the organic pesticides are bound to cuticular surface of the fruits and vegetables and hence effect their removal during washing (Srivastava *et al.* 2021). Acidic reagents are particularly effective in reducing organophosphates and pyrethroids from spinach (Amir *et al.,* 2019).

The effectiveness of commercially available decontaminants such as Veggie Clean, Nimwash, and Arka Herbiwash varied slightly, likely due to differences in their chemical compositions. Though all three were effective in removing profenofos residues, but Nimwash emerged as the most effective commercial decontaminant for all tested pesticides, while Arka Herbiwash was the least effective among the treatments employed. The efficacy of pesticide decontamination depends on the interactions between the pesticide and the physicochemical properties of the applied decontaminants.

CONCLUSION

Thus it is concluded that thorough washing and dipping chili in saline solutions can effectively reduce high concentrations of pesticides applied on the crop. Since pesticide residues are typically higher in market-sold vegetables, it is crucial to monitor and decontaminate them. Household methods like washing, cooking, washing followed by cooking, and washing with salt water help in lowering pesticide residues. These decontamination methods are cost-effective as most solutions are household items and inexpensive. However, it is important to note that the treatment solutions should be used only once, as reusing them can reduce their effectiveness in removing pesticides. Commercial decontaminants can also be used for reduction of profenofos residues in chili but they are not so cost-effective.

Table 2: Effect of different decontamination methods in the removal of profenofos residues from chili

Decontamination Treatments	Profenofos (50%EC) Residues (mg / Kg)	Reduction (%)
T-0 (Without any decontamination)	30.87	0
T-1 (Washing with Running Tap water for 1min.)	5.68	81.60
T-2 (Soaking in water for 10 min.)	7.56	75.51
T-3 (Soaking in Lukewarm water for 10 min.)	3.08	90.03
T-4 (1% NaCl for 10 min.)	5.88	90.95
T-5 (5%NaHCO3 for 10 min.)	7.99	74.11
T-6 (2% Acetic acid for 10 min.)	5.87	80.98
T-7 (0.01% KMnO4 for 10 min.)	6.56	78.75
T-8 (Veggie clean for 10 min.)	6.82	77.91
T-9 (Nimwash for 10 min.)	6.51	78.91
T-10 (Arka Herbiwash for 10 min.)	8.62	72.05

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