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***Research Paper***

# Effect of household processing treatments on malathion residues in cucumber

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**ABSTRACT :** Pesticides are widely used in vegetable production despite the fact that they have been associated with several health adversities. Malathion is an organophosphate which is used as a recommended pesticide on cucumbers. However, it is a parasympathomimetic which binds irreversibly to cholinesterase and impose neurotoxic effects on human body. Decontamination of vegetables at household level is the best strategy that can be adopted to reduce the intake of pesticide residues. The study was conducted to analyse the effect of common household methods on the reduction of malathion residues in cucumber. Recoveries were checked at 0.1 and 0.05 fortification levels and were found to be within the acceptable range *i.e.* 85- 115 per cent. Cucumber samples were spiked with recommended doses of malathion and were subjected different household processing treatments *viz.*, washing, dipping in chemical solutions and peeling. Analysis was done through QUEChERS and the residue content was analysed through Gas- Liquid chromatography. Least reduction was observed in simple washing with tap water. Peeling was found to be the most effective treatment with a reduction of 91.64 per cent. Reductions from dipping in chemical solutions ranged from 55.87 per cent by 10 per cent sodium chloride solution to 77.98 by 5 per cent sodium bicarbonate. Hence, it can be concluded that household processing can effectively reduce malathion residues in cucumber.

**KEY WORDS :** Pesticides, Cucumber, Malathion, Household processing

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Cucumber is a salad vegetable and is usually consumed in raw or pickled state. Cucumbers are now known to contain lariciresinol, pinoresinol, and secoisolariciresinol-three lignans that have a strong history of research in connection with reduced risk of cardio-vascular disease as well as several cancer types, including breast, uterine, ovarian, and prostate cancers. Fresh extracts from cucumbers have recently been show to have both antioxidant and anti-inflammatory properties. One cup (104 g) of cucumber provides 21 per cent of the daily value of vitamin K and is also rich in minerals like molybdenum, potassium, manganese and magnesium (Milder *et al.,* 2005; Abiodun 2010; Lee *et al.,* 2010).

Cucumbers, during their growth cycle are infested by various pests. To combat the insect pest problem several pesticides are applied which may leave certain amount of residues on them and in turn reach the human body through food chain. The widespread use of pesticides not only contaminates water, soil, and air, but also causes them to accumulate in crops. These residues if consumed in even small quantities may keep on accumulating in the body for years and can impose several health hazards leading to many chronic diseases. Cucumbers have been ranked tenth most contaminated fruit according to the Environmental Working Group's annual "Dirty Dozen" report and are found contaminated with organophosphate insecticides. (Csomor, 2012).

Malathion is widely used as organophosphorus pesticide (OPP) due to its low persistence in the environment, low mammalian toxicity and high insecticidal activity. It has moderate toxicity but its crude and formulation contain impurities which are far more toxic to mammals.

**Table A: The operating parameters of the instrument**

| Column | | | | Injector | | Detector | |
|---|---|---|---|---|---|---|---|
| Rate | 0 | 10 | 5 | Temperature | 280 °C | Temperature | 300 °C |
| Temperature | 150 °C | 220 °C | 240 °C | Total flow | 12.6 ml/min | Make up flow | 27.5 ml/min |
| Hold time | 5 min | 3 min | 13 min | Column flow | 0.87 ml/min | Hydrogen flow | 1.5 ml/min |
| Length of column = 30 m | | | | Split ratio | 10 | Air flow | 145 ml/min |

OPPs usually have an ester structure, decomposing fairly easily on the surfaces and interiors of plants, and in the soil (Fenik *et al.,* 2011). Organophosphates are neurotoxic and are associated with several neurological deformities. Its toxicity depends on inhibiting the activity of enzymes, controlling the functions of the nervous system, mainly acetylcholinesterase. They permanently bind the group hydroxylating the enzyme, which prevents acetylcholinesterase from decomposing, and act through contact or systemically. Blockage of cholinesterase activity causes the amount of acetylcholine at the synapses to increase, leading to a state of hyperarousal and paralysis of the muscles and the main respiratory centre. (Singh, 2000; Margni *et al.,* 2001; Kaushik *et al.,* 2009).

Unfortunately, the debate on pesticide residues is restricted only to quality standards and norms. The basic problem of contamination of all natural resources with chemical pesticide residues and further in the food chain is of serious concern and is often ignored. Present study was conducted to see the effect of common household methods applied on malathion residues in cucumber.

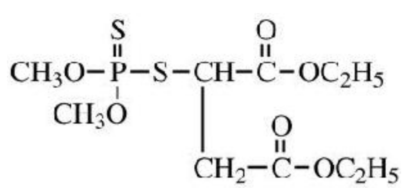

**Fig. A: Malathion structure**

## ■ RESEARCH METHODS

Cucumbers were collected from the local market and sprayed with 120 mg/kg of malathion as recommended in the 'Package of Practices' for farmers published by Punjab Agricultural University, Ludhiana and kept overnight after spiking before processing. The level of spiking was kept high to have a clear picture of the effect of processing treatment. After that different processing treatments were given to see the impact on reduction of pesticide residues.

### Extraction of residues:

The analysis of pesticides was carried out in Pesticide Residue Analysis Laboratory, Department of Entomology, Punjab Agricultural University, Ludhiana. Cucumber samples were extracted by QuEChERS (Quick easy cheap effective rugged safe) method with slight modifications (Anastassiades *et al.,* 2003). Vegetable samples were macerated in a blender (Blixer 6 V.V. by robot coupe, France) and a representative 15 g (±0.1 g) of the sample was transferred to 50 ml centrifuge tube. Added 30 ml of acetonitrile to each tube with the help of dispenser and homogenized @ 15,000 rpm for 2 - 3 min using a high speed homogenizer (High Speed Silent Crusher-Heidolph) to ensure that the sol-vent interacted well with entire sample. Mixed 5 - 10 g NaCl to each tube and shook the tubes on rotaspin for 5 minfollowed by centrifugation for 3 min @ 2500 rpm. The acetonitrile layer was collected in another centrifuge tube containing 5 g activated anhydrous sodium sulphate and again shaken for 5 minute at 50 rpm on the rotaspin to remove the moisture completely. The samples were cleaned up by dispersive solid phase extraction method. An aliquot of 6 ml was dispensed into 15 ml centrifuge tube containing 0.15 ± 0.01 g (PSA) primary secondary amine sorbent ( to remove fatty acid among other components) and 0.9 ± 0.01 g anhydrous $MgSO_4$ (to reduce the remaining water in the extract) and 0.05 ± 0.01 g graphitised carbon. The tubes were vortexed for 3 minfollowed by centrifugation for 1 min @ 2500 rpm. From the upper layer of the prepared samples, 4mL of the extract were transferred into another 15 ml tube and were rotary evaporated at <35° to remove the acetonitrile completely. Finally, the volume was reconstituted to 2 - 5 ml with distilled acetone.

### Estimation of residues:

Estimation of residues was carried out by gas liquid chromatography (GLC) equipped with Flame Thermoionic Detector (FTD). Injection volume was 1 µl.

## ■ RESEARCH FINDINGS AND DISCUSSION

In order to evaluate the efficiency of extraction, cleanup and determinative steps the analytical method was standardized by processing spiked samples, before taking up the analysis of the test samples. The experiment was performed by spiking vegetables samples with Malathion insecticides. The recoveries were found to be consistent and more than 80 per cent at different concentrations (0.01, 0.05, 0.1mg $kg^{-1}$) and an RSD below 15 per cent as given in Table 1. The area of peak so obtained was compared with the area of standard and recoveries were calculated in terms of percentage.

**Table 1 : Per cent recovery of malathion at different fortification levels in cucumber**

| Fortification level (mg/kg) | Recovery (%) | RSD (%) |
|---|---|---|
| 0.1 | 92.67 | 2.20 |
| 0.05 | 89.29 | 3.25 |
| 0.01 | 87.25 | 3.21 |

The impact of different processing treatments on malathion content in cucumber is given in Table 2.

The cucumber samples were spiked with 120 mg/kg of malathion, however, when analysed on gas chromatograph, the residues were detected up to the level of 118.21 mg/kg. Table 2 shows that all treatments reduced malathion contents significantly. Washing in tap water reduced the residue content to 85.06 mg/kg with a reduction of 28.03 per cent. Malathion is a non-systemic insecticide and has high water solubility (145 mg/L) relative to many of the OP insecticides. In a study, washing removed 21 per cent of malathion in cucumbers (Gehad *et al.* 2012) and the results in the present study were slightly higher. Krol *et al.* (2000) reported that malathion residues were significantly reduced during washing. Washing with 2 per cent NaOH and $KMnO_4$ reduced residues by 68.31 and 73.73 per cent and these two treatments were found to be at par as analysed by Post-Hoc Tukey's test. Significant reduction was observed by dipping in 5 per cent sodium bicarbonate solution. This solution neutralizes malathion into harmless by-products and is also recommended for use in case of malathion toxicity. If the pesticide has been ingested unless the patient is vomiting, rapid gastric lavage should be performed using 5 per cent sodium bicarbonate (WHO/FAO, 2013).

Reductions were also found to be at par in samples treated with sodium hydroxide and acetic acid with the latter giving 73 per cent reduction. The results obtained were similar to a study conducted in 2010 in which dipping of cucumbers in 10 per cent acetic acid solution for thirty minreduced profenofos (another pesticides belonging to the organophosphate group) residues by 67.8 per cent (Chai and Huat, 2010). As shown in Table 2, no significant difference was observed between samples treated with potassium permanganate and sodium hydroxide; the latter giving the reduction percentage of 77.98 per cent and a processing factor of 0.22 (Fig. 1). All chemical solutions used may have released different reactive/oxidant species in solution state like the hydroxyl ion from sodium hydroxide, permanganate ion from potassium permanganate, chloride ion from sodium chloride etc., which may have been responsible for the oxidation and degradation of pesticide (Cengiz and Certel, 2012).

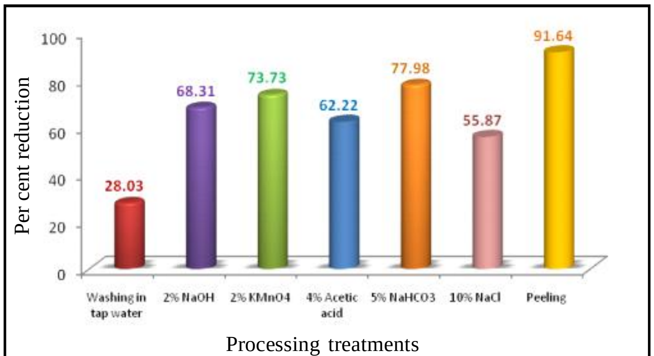


**Fig. 1: Effect of processing treatments on reduction of malathion residues in cucumber**

Maximum reductions were observed on peeling with a reduction percentage of 91.64 and processing factor of 0.08. This may be attributed to the non-systemic nature of malathion due to which the residues get accumulated on the outer surfaces of the fruit and hence, can be easily removed by peeling. Gehad *et al.* (2012) observed comparatively less reduction (66 %) of malathion residues on cucumber.

## Conclusion:

Hence, it can be concluded that peeling was found to be the most effective household processing treatment in reducing malathion residues in cucumber and can be attributed to the non-systemic nature of organophosphates

**Table 2: Effect of different household treatments on malathion residues in cucumber**

| Treatment | Mean* ±SE | Per cent reduction | Processing factor | F-value |
|---|---|---|---|---|
| Before treatments | $118.21^{a}\pm2.57$ | | | |
| Washing in tap water | $85.06^{b}\pm2.64$ | 28.03 | 0.72 | |
| 2% NaOH | $37.46^{c}\pm2.12$ | 68.31 | 0.32 | |
| 2% $KMnO_4$ | $31.06^{dc}\pm0.69$ | 73.73 | 0.26 | 356.63** |
| 4% Acetic acid | $44.66^{ec}\pm0.97$ | 62.22 | 0.38 | |
| 5% $NaHCO_3$ | $26.03^{fd}\pm2.41$ | 77.98 | 0.22 | |
| 10% NaCl | $52.16^{ge}\pm1.24$ | 55.87 | 0.44 | |
| Peeling | $9.88^{h}\pm0.89$ | 91.64 | 0.08 | |

* Mean values are in terms of mg/kg, a-h Means in the same column without common superscripts differ (P=0.05),** indicate significance of value at P=0.01

(malathion being an organophosphate shows this property). Least reduction was observed in simple washing with tap water and can be attributed less time given for washing in running tap water (2 min) as compared to other treatments (30 mindipping followed by rinsing in running water). Significant reduction in malathion residues through different detergent washings can also be associated with their ability to oxidise. All solutions used may have released different reactive/oxidant species in solution state like the hydroxyl ion from sodium hydroxide, permanganate ion from potassium permanganate, chloride ion from sodium chloride etc., which may have been responsible for the oxidation and degradation of pesticide. It is the need of the hour to optimize household processing techniques for reduction of pesticide residues in vegetables to reduce exposure and develop strategies that can be applied at household level.

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