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# **Research Article**

# **Effects of Household Processing on reduction of**

# Pesticide Residues in Brinjal and Okra

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## ABSTRACT

The experiments were conducted to investigate the effects of household processing on removal of organophosphate (chlorpyrifos and monocrotophos) and pyrethroid (cypermethrin) residues in brinjal and okra. The household processes included washing separately with water, 2.0% NaCl, 1.0 % NaHCO3, 0.5 % acetic acid and boiling in water. Samples of brinjal and okra were collected from experimental field which were sprayed with different concentration of pesticide on the vegetables. In the household processes of brinjal, residues reduced by 29.5-99.2%, 30.2-92.1% and 65.6-99.7%, chlorpyrifos, cypermethrin and monocrotophos respectively. Whereas household processes of okra, residues reduced by 24.5-98.9%, 27.2-92.2% and 62.4-99.5%, chlorpyrifos, cypermethrin and monocrotophos respectively. Maximum residues were reduced by boiling (99.7%) reduction of pesticides was observed in brinjal, and okra by household processing. Boiling was found comparatively more effective than washing in dislodging the residues. The samples were extracted with 1% acetic acid in acetonitrile mixture and cleaned up with primary secondary amine (PSA) and magnesium sulphate. Experiments on two fortification concentrations are carried out, and the limits of detection are 0.004, and 0.003 mg/kg for chlorpyriphos, monocrotophos respectively. The average recoveries of pesticide residues in brinjal and okra

#### Keywords: Brinjal, Okra, Household processing, GC-MS.

## **INTRODUCTION**

Vegetables are the inseparable components of Indian cuisine and are consumed throughout the country in different forms and preparations. They form the bulk and are the major source of vitamins and nutrients. hence fulfilling the requirements of our balanced diet. Among the vegetables, brinjal, cauliflower and okra are very common and give better return over investment to the farmers. Brinjal (Solanum melongena L.) is an important vegetable crop grown extensively in India. It suffers heavily at fruiting stage due to attack of shoot and fruit borer causing 70% damage to the crop making it totally unfit for human consumption<sup>1</sup>. Okra (Abelmoschus esculentus L.) belongs to family malvaceae is also an important vegetable crop grown extensively in India and for the control of numerous insect pests, different insecticides have been used<sup>2,3</sup>. Hence, in order to combat the insect pest problem, lot of pesticides is

used by the vegetable growers for better yield and quality; insecticides are repeatedly applied during the entire period of growth and sometimes even at the fruiting stage. It accounts for 13-14 percent of total pesticides consumption, as against 2.6 percent of cropped area<sup>4</sup>. Indiscriminate use of pesticides waiting period leads to accumulation of pesticide residues in consumable vegetables. Contamination of vegetables with pesticide residues has been reported by several researchers<sup>5-7</sup>. Vegetables need washing for three main reasons: 1) They need washing to remove dirt and dust so that they can be presented to the consumer in a visually appealing manner, 2) They need washing with sanitisers for postharvest treatment purposes so that postharvest plant diseases can be kept under control and thus increase vegetable shelf life and 3) They need washing with sanitisers for food safety purposes so that human pathogens

that may be present on the surface of vegetables are not passed on to consumers.

Washing of vegetables is the most traditional and the preliminary unit operation applied to remove debris and dirt from vegetables prior to consumption. In some studies it were also, observed the effects household methods for removal of pesticide residues from different vegetables by<sup>8-15</sup>. To estimate the potential pesticide exposure from contaminated food, it is important to estimate the level of exposure at the point of consumption after processing. It has already been reported that commercial and household processing such as washing, peeling, cooking, blanching and concentrating can reduce residue levels in food, which further reduces the impact on human health<sup>16-</sup>.

The extent of residue reduction by washing depends on the physiochemical properties of the pesticides, such as water solubility, hydrolytic rate constant, volatility and octanol-water partition coefficient (Pow), in conjunction with the actual physical location of the residues; washing processes lead to reduction of hydrophilic residues which are located on the surface of the crops.

The aim of this study was to evaluate the chlorpyriphos, cypermethrin and monocrotophos residue in brinjal and okra to assess the effect of different household processing to reduced pesticides residues to a safe level for human consumption. The resent study can be used as a reference point for future monitoring and taking preventive measures to minimize human health risks.

### 2. EXPERIMENTAL

#### **Chemical and reagents**

The organic solvent acetic acid, acetonitrile HPLC grade, magnesium sulphate, sodium acetate AR grade, sodium chloride (salt), sodium hydrogen carbonate purchased from E Merck and primary secondary amine purchased from Agilent Technologies. The technical grade pesticide standards were used for standardizations. Anhydrous magnesium sulphate used during residue extraction was maintained at 300°C overnight and kept in air tight container. Polyethylene or PFTE 15ml and 50 ml with screw cap tubes.

#### Sample collection

The fresh brinjal and okra 2 kg each samples were collected at regular interval as such from the supervised trial was conducted at the experimental field at Vasco-Da-Gama, Goa, during summer season. In supervised trials, chlorpyrifos, cypermethrin and monocrotophos were applied with different concentrations (100, 200 and 300g a.i.ha<sup>-1</sup>) on the brinjal and okra. One control plot (unsprayed)

was included for each crop for blank analysis and also recovery experiments.

#### Extraction

The fresh brinjal and okra 2 kg each samples were taken for the extraction of pesticide residues. The samples were macerated to make paste with Philips mixer (equipped with stainless steel knives), a 15 g portion of the homogenized sample was weighed into a 50 ml polytetrafluoroethylene (PTFE) tube added 15 ml of acetonitrile containing 1% acetic acid (v/v). Then, 6 g MgSO4 and 2.5 g sodium acetate trihydrate (equivalent to 1.5 g of anhydrous form) were added, and the sample was shaken forcefully for 4 min and kept in ice bath. The samples were then centrifuged at 4000 rpm for 5 min and 6 ml of the supernatant were transferred to a 15 ml PTFE tube to which 900 mg MgSO4 and 300 mg PSA were added. The extract was shaken using a vortex mixer for 20 s and centrifuged at 4000 rpm again for 5 min, approximately 2ml of the supernatant were taken in a vials. This extracts were evaporated to dryness under a stream of nitrogen and reconstituted in n-hexane in auto sampler tube for the GC-MS analysis.

#### Washing solution preparation

All washing solutions for treatment of vegetables were prepared from the standard reagents. These consisted of filtrated water generated from water purifier, 2.0% NaCl, 0.1 % NaHCO3, and 0.5 % acetic acid. The purified water was used as diluents for all the solution and treatments.

#### Recovery

Recovery studies were performed to examine the efficacy of extraction and clean up. Control samples of brinjal and okra were spiked with known concentration of the pure pesticides standard solution and extraction and clean-up were performed as described earlier. The concentration of each pesticide in the final extracts was calculated (table 1). The average recoveries of pesticide residues in brinjal and okra samples were 80.0 to 105.0 %.

### **RESULTS AND DISCUSSIONS**

Among the household processes using, water, 2.0% NaCl, 1.0% NaHCO3, 0.5% acetic acid and boiling in water chlorpyrifos residues reduced by 29.5-32.5%, 62.5-65.6%, 59.8-61.8%, 65.6-67.3% and 98.5-99.2%, cypermethrin residues reduced by 29.5-32.5%, 60.04-62.9%, 61.9-62.8%, 67.9-68.5% and 90.1-92.1%, monocrotophos residues reduced by 65.6-66.4%, 71.1-72.8%, 80.8-82.2%, 78.8-79.2% and 98.5-99.7% respectively in brinjal. The chlorpyrifos residues reduced by 24.5-29.1%, 68.0-69.4%, 62.0-64.8%, 65.6-69.8% and 97.9-98.9%, cypermethrin residues reduced by 27.2-29.3%, 64.9-67.4%, 62.8-65.8%, 65.2-69.4% and 89.2-92.2%, monocrotophos residues reduced by 62.4-65.5%, 75.6-76.9%, 80.1-84.3%, 74.6-78.0% and 98.8-99.5% respectively in okra (table 2 and 3).

Beena Kumari (2008) observed that washing process reduced the OC residues by 27-44 percent in brinjal, 34-36 percent in cauliflower and 20-38 percent in okra. Whereas the residues of SP insecticides in brinjal, cauliflower and okra were reduced to 26, 29 and 31 percent, respectively. Maximum reduction of residues was observed in case of OP where the residues decreased to the extent of 77, 74 and 50 percent, in brinjal, cauliflower and okra, respectively. Boiling/cooking was observed to be more effective in reducing the residues. By this process, reduction of residues of OC insecticides was observed in the range of 39-55 per cent in brinjal, 57-61 percent in cauliflower and 32-47 percent in okra. Reduction to an extent of 37, 40 and 42 percent of SP insecticides was observed in brinjal, cauliflower and okra, respectively. Among OP insecticides, reduction was 100 percent in brinjal, 92 percent in cauliflower and 75 percent in okra<sup>11</sup>.

Washing of cauliflower treated with chlorpyriphos, quinalphos, endosulfan, fenvalerate and deltamethrin reduced 28.92 %–78.64 % residues of these insecticides8. Tomatoes contaminated at level of 1 mg kg-1 upon washing with 10 % NaCl solution gave 42.90, 46.10, 27.20, 90.80, 82.40 and 91.40 per cent loss in HCB, lindane, p,p-DDT, dimethoate, profenophos and pirimiphos-methyl, respectively<sup>21</sup>.

Liang and co-workers (2012) reported that 63.40, 60.00, 50.00, 31.10 and 66.70 per cent reduction in the residues of trichlorfon, dimethoate, dichlorvos, fenitothion and chlorpyriphos respectively, were observed in cucumber when dipped in 2 % sodium chloride solution for 20 min<sup>22</sup>. These results agree with those reported earlier that soaking of contaminated potatoes in neutral (NaCl) solution (5 and 10 %) for 10 min resulted in 100 percent removal of pirimiphos methyl residues<sup>17</sup>. The cause and effect of the reduction in 2 % NaCl washing solutions is still not known and needs further investigation.

The effects of household processing on removal of residues of malathion, fenitrothion, formothion, parathion, methyl parathion and chlorpyriphos in tomato, bean, okra, eggplant, cauliflower and capsicum were studied. The processes included washing water, 0.9 % NaCl, 0.1 % NaHCO3, and 0.1 % acetic acid, 0.001 % KMnO4, 0.1 % ascorbic acid, 0.1 % malic acid and 0.1 % oxalic acid and 2 % aqueous solution of raw Spondias pinnata (SP)) and boiling. In all of the vegetables, washing with different household chemicals reduced the residues by 20-89 % and boiling reduced the residues by 52-100 %. Boiling of vegetables was found to be more effective than washing in dislodging the residues (Gouri Satpathy et al., 2012). Residues of Monochrotophos, fenitrothion and fenvalerate were removed to an extent of 41.81%, 100% and 100% by dipping in lemon juice, dipping in 2% tamarind solution for 5 min followed by wash tap water and steam cooking for 10 min respectively $^{23}$ .

In the earlier studies it was found that washing effective in dislodging the residues as it depends on a number of factors like location of residues, age of residues, water solubility and temperature and type of washing. Current results are in consistent with some earlier reports where reduction (10-30%) of alphamethrin residues was found in tomato and brinjal and cauliflower <sup>24-25</sup>. Rinsing of various vegetable was found very effective<sup>8</sup>.

In present study it was observed that washing with water, 2.0% NaCl, 1.0 % NaHCO3, 0.5 % acetic acid was found comparatively less effective in reducing the pesticides residues while boiling was observed to be more effective in reducing the residues.

|         | Recovery of pesticides in the spiked control samples |   |                 |                                |  |  |  |
|---------|--|---|-----------------|--------------------------------|--|--|--|
| Sample  | Compound   | Concentration<br>(mg kg <sup>-1</sup> ) | Recovery<br>(%) | Coefficient of variation(n=5)% |  |  |  |
| Brinjal | Chlorpyrifos   | 1.0                                     | 104.70          | 4.58                           |  |  |  |
| Brinjal | Cypermethrin   | 1.0                                     | 99.80           | 4.78                           |  |  |  |
| Brinjal | Monocrotophos  | 1.0                                     | 98.60           | 4.80                           |  |  |  |
| Okra    | Chlorpyrifos   | 1.0                                     | 101.00          | 4.85                           |  |  |  |
| Okra    | Cypermethrin   | 1.0                                     | 90.50           | 4.86                           |  |  |  |
| Okra    | Monocrotophos  | 1.0                                     | 92.10           | 4.95                           |  |  |  |

 Table 1

 Recovery of pesticides in the spiked control samples

| of pesticide | e residues (m  | igkg ) on b  | orinjal when was  | hing with diff   | erent washing  | solutions.   |
|--------------|--|--|---|--|--|--|
| Sample       | Initial<br>residue<br>(mgkg <sup>-1</sup> )  | Water  | 1%NaHCO3  | 2% NaCl  | 0.5% Acetic<br>Acid  | Boiling in<br>water  |
| 1            | 0.362  | 29.5±2   | 62.5±2  | 59.8±1   | 65.6±3   | 98.5±1   |
| 2            | 0.679  | 31.2±2   | 62.4±2  | 61.8±1   | 68.5±3   | 98.9±1   |
| 3            | 0.876  | 32.5±2   | 65.6±2  | 61.2±1   | 67.3±3   | 99.2±1   |
| 1            | 0.340  | 0.2±2  | 61.9±2  | 62.2±1   | 68.5±3   | 90.1±1   |
| 2            | 0.661  | 32.0±2   | 60.4±2  | 61.9±1   | 67.9±3   | 90.4±1   |
| 3            | 0.858  | 31.4±2   | 62.9±2  | 62.8±1   | 68.1±3   | 92.1±1   |
| 1            | 0.388  | 65.6±2   | 71.1±2  | 81.2±1   | 78.9±3   | 98.5±1   |
| 2            | 0.690  | 66.4±2   | 72.2±2  | 82.2±1   | 78.8±3   | 99.7±1   |
| 3            | 0.891  | 65.8±2   | 72.8±2  | 80.8±1   | 79.2±3   | 99.1±1   |
|              | Sample<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>2<br>2<br>2<br>2<br>2<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>3<br>1<br>2<br>2<br>3<br>1<br>2<br>2<br>3<br>1<br>2<br>2<br>3<br>1<br>2<br>2<br>2<br>3<br>1<br>2<br>2<br>2<br>3<br>1<br>2<br>2<br>2<br>3<br>2<br>2<br>3<br>1<br>2<br>2<br>2<br>3<br>1<br>2<br>2<br>2<br>2<br>2<br>3<br>2<br>2<br>3<br>2<br>2 | Sample         Initial residue (mgkg <sup>-1</sup> )           1         0.362           2         0.679           3         0.876           1         0.340           2         0.661           3         0.858           1         0.388           2         0.690 | Sample         Initial<br>residue<br>(mgkg <sup>-1</sup> )         Water           1         0.362         29.5 $\pm 2$ 2         0.679         31.2 $\pm 2$ 3         0.876         32.5 $\pm 2$ 1         0.340         0.2 $\pm 2$ 2         0.661         32.0 $\pm 2$ 3         0.858         31.4 $\pm 2$ 1         0.388         65.6 $\pm 2$ 2         0.690         66.4 $\pm 2$ | Sample         Initial<br>residue<br>(mgkg <sup>-1</sup> )         Water         1%NaHCO3           1         0.362         29.5 $\pm 2$ 62.5 $\pm 2$ 2         0.679         31.2 $\pm 2$ 62.4 $\pm 2$ 3         0.876         32.5 $\pm 2$ 65.6 $\pm 2$ 1         0.340         0.2 $\pm 2$ 61.9 $\pm 2$ 2         0.661         32.0 $\pm 2$ 60.4 $\pm 2$ 3         0.858         31.4 $\pm 2$ 62.9 $\pm 2$ 1         0.388         65.6 $\pm 2$ 71.1 $\pm 2$ 2         0.690         66.4 $\pm 2$ 72.2 $\pm 2$ | Sample         Initial<br>residue<br>(mgkg <sup>-1</sup> )         Water         1%NaHCO3         2% NaCl           1         0.362         29.5 $\pm$ 2         62.5 $\pm$ 2         59.8 $\pm$ 1           2         0.679         31.2 $\pm$ 2         62.4 $\pm$ 2         61.8 $\pm$ 1           3         0.876         32.5 $\pm$ 2         65.6 $\pm$ 2         61.2 $\pm$ 1           1         0.340         0.2 $\pm$ 2         61.9 $\pm$ 2         62.2 $\pm$ 1           2         0.661         32.0 $\pm$ 2         60.4 $\pm$ 2         61.9 $\pm$ 1           3         0.858         31.4 $\pm$ 2         62.9 $\pm$ 2         62.8 $\pm$ 1           1         0.388         65.6 $\pm$ 2         71.1 $\pm$ 2         81.2 $\pm$ 1           2         0.690         66.4 $\pm$ 2         72.2 $\pm$ 2         82.2 $\pm$ 1 | residue<br>(mgkg <sup>-1</sup> )residue<br>(mgkg <sup>-1</sup> )Acid1 $0.362$ $29.5\pm 2$ $62.5\pm 2$ $59.8\pm 1$ $65.6\pm 3$ 2 $0.679$ $31.2\pm 2$ $62.4\pm 2$ $61.8\pm 1$ $68.5\pm 3$ 3 $0.876$ $32.5\pm 2$ $65.6\pm 2$ $61.2\pm 1$ $67.3\pm 3$ 1 $0.340$ $0.2\pm 2$ $61.9\pm 2$ $62.2\pm 1$ $68.5\pm 3$ 2 $0.661$ $32.0\pm 2$ $60.4\pm 2$ $61.9\pm 1$ $67.9\pm 3$ 3 $0.858$ $31.4\pm 2$ $62.9\pm 2$ $62.8\pm 1$ $68.1\pm 3$ 1 $0.388$ $65.6\pm 2$ $71.1\pm 2$ $81.2\pm 1$ $78.9\pm 3$ 2 $0.690$ $66.4\pm 2$ $72.2\pm 2$ $82.2\pm 1$ $78.8\pm 3$ |

 Table 2

 esidues (mgkg<sup>-1</sup>) on brinjal when washing with different was

| Table | 3 |
|-------|---|
|-------|---|

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| Reduction (%) of pesticide residues (mgkg ) on okra when washing with different washing solutions. |        |  |        |          |         |                     |                     |
|--|--------|--|--------|----------|---------|---------------------|---------------------|
| Pesticide  | Sample | Initial residue<br>(mgkg <sup>-1</sup> ) | Water  | 1%NaHCO3 | 2% NaCl | 0.5% Acetic<br>Acid | Boiling in<br>water |
| Chlorpyrifos   | 1      | 0.389                                    | 24.5±2 | 68.0±2   | 62.0±1  | 65.6±3              | 98.2±1              |
|  | 2      | 0.696                                    | 28.2±2 | 68.2±2   | 64.8±1  | 69.6±3              | 97.9±1              |
|  | 3      | 0.874                                    | 29.1±2 | 69.4±2   | 64.2±1  | 69.8±3              | 98.9±1              |
| Cypermethrin   | 1      | 0.378                                    | 27.2±2 | 64.9±2   | 62.8±1  | 65.2±3              | 90.1±1              |
|  | 2      | 0.685                                    | 28.3±2 | 67.4±2   | 65.8±1  | 68.4±3              | 89.9±1              |
|  | 3      | 0.862                                    | 29.3±2 | 67.2±2   | 65.6±1  | 69.4±3              | 92.2±1              |
| Monocrotophos  | 1      | 0.391                                    | 65.5±2 | 75.7±2   | 80.1±1  | 74.6±3              | 98.8±1              |
|  | 2      | 0.698                                    | 62.4±2 | 76.9±2   | 84.3±1  | 78.0±3              | 99.1±1              |
|  | 3      | 0.898                                    | 63.7±2 | 75.6±2   | 81.4±1  | 77.6±3              | 99.5±1              |

#### CONCLUSION

Use of pesticides on vegetables is an inevitable part of agriculture but their unscientific usage can cause significant health adversities. From the extensive review collected on the extent of dissipation of pesticide residues and mechanisms involved during household processing techniques, it is concluded that these can serve as an effective tool for reduction of residues within safe limits. There is a need to regulate pesticide intake for leading a healthy life. Processing substantially reduces the residues of pesticides in vegetables. These reductions are extremely important in evaluating the risk associated with ingestion of pesticide residues, especially in vegetables, which are eaten by almost all income group people. However, there is dearth of knowledge involving exact mechanism of action by which different processing treatments ought to reduce pesticide residues in different food items.

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