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EFFECTS OF HOUSEHOLD PROCESSING ON REDUCTION OF PESTICIDE RESIDUES IN VEGETABLES

Beena Kumari
Department of Entomology, Chaudhary Charan Singh Haryana Agricultural University, Hisar, Haryana, India
E-mail: beena@hau.ernet.in

ABSTRACT

In a monitoring study residue levels of organochlorines (OC), synthetic pyrethroids (SP), organophosphates (OP) and carbamates were determined in unprocessed and processed three vegetables viz. brinjal, cauliflower and okra to know the residue levels and evaluate the effect of different household processes (washing and boiling/cooking) on reduction of residues. Samples were procured from local market at different interval of time. Residues were estimated by using multi residue analytical technique employing gas liquid chromatograph (GC), with electron capture detector (ECD) and nitrogen phosphorous detector (NPD) equipped with capillary columns. In all the three vegetables, washing reduced the residues by 20-77 percent and boiling by 32-100 percent. Maximum (77%) reduction of OP insecticides was observed in brinjal, followed by 74% in cauliflower and 50% in okra by washing. The same trend was observed by boiling process where maximum (100%) reduction of OP insecticides was observed in brinjal followed by 92% in cauliflower and 75% in okra. Boiling was found comparatively more effective than washing in dislodging the residues.

Keywords: vegetables, pesticide, processing, residues, washing, boiling, cooking.

INTRODUCTION

Vegetables are the fresh and edible portion of the herbaceous plants. They are important food and highly beneficial for health. They contain valuable food ingredients, which can be successfully utilized to build up and repair the body. In India, vegetables are major constituents of diet as majority of Indians are vegetarian, with a per capita consumption of 135 g per day as against the recommended 300 g per day. It is still very less than recommended diet level. However, several factors limit their productivity, mainly insect pests, and diseases. As several insect pests attack the vegetables, they are produced under very high input pressure. Among the vegetables, brinjal, cauliflower and okra are very common and give better return over investment to the farmers. But all the three are badly affected by insect-pest attack. 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 (Misra and Singh, 1996; Duara *et al*., 2003). Cauliflower (*Brassica oleracea*), an important vegetable crop grown in India with an annual production of 3.39 million tones, is heavily attacked by various insects, resulting in severe loss of quality and production (Regupathy *et al*., 1985; Patel *et al*., 1999). 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 (Singh *et al*., 2004; Sinha and Sharma, 2007). 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 (Sardana, 2001). Indiscriminate use of pesticides particularly at fruiting stage and non adoption of safe waiting period leads to accumulation of pesticide residues in consumable vegetables. Contamination of vegetables with pesticide residues has been reported by several researchers (Madan *et al*., 1996; Kumari *et al*., 2002 and 2003).

The aim of this study was to evaluate the pesticide residues of four different chemical groups i.e., organochlorines (OC), synthetic pyrethroids (SP) organoposphorous (OP), and carbamates in brinjal, cauliflower and okra and to assess the effect on residues of some household processes like washing and boiling/cooking.

MATERIALS AND METHODS

The composite samples consisted of 1-2kg of each vegetable i.e. brinjal, cauliflower and okra were collected from local market at weekly interval. Each sample was divided in to three parts and were refrigerated and analysed with in two days of collection. Only edible part was processed and analysed for the analysis of organochlorine (OC), synthetic pyrethroid (SP), organophosphate (OP) and carbamate group of pesticides.

In order to assess the effects of household processing like washing and boiling/cooking, one part of the sample of each vegetable was washed for one minute under tap water and dried the samples on filter paper. To the other part of each unwashed samples of three vegetables, 15ml water was added and boiled till softness. Washed and boiled samples were processed in a similar manner as of unprocessed samples.

Extraction

All the samples were extracted fresh. Each vegetable was chopped into small pieces and after quartering, a representative sample (25g) was macerated with 5-10g anhydrous sodium sulphate in Warring blender to make a fine paste. The macerated sample was extracted with 100ml acetone on mechanical shaker for 1 h by using the method of Kumari *et al.* (2001). Extract was filtered, concentrated up to 40ml and subjected to liquid-liquid partitioning with ethyl acetate (50, 30, 20 ml) after diluting 4-5 times with 10% aqueous NaCl solution. Concentrated the organic phase up to 10ml on rotary evaporator and divide it into two equal parts. One part was kept for OC and SP and second for OP and carbamates.

Clean-up

For OC and SP insecticides, clean-up was carried out by using column chromatography. Column (60cm × 22mm) was packed with, Florisil and activated charcoal (5:1 w/w) in between the two layers of anhydrous sodium sulphate. Extract was eluted with 125ml mixture of ethyl acetate: hexane (3:7 v/v). Eluate was concentrated to 2ml for residue analysis. Residues of OP and carbamates were also cleaned by adopting column chromatographic technique. Column was packed with silica gel and activated charcoal (5:1 w/w) in between the layers of anhydrous sodium sulphate. Extract was eluted with 125ml mixture of acetone: hexane (3:7 v/v). After concentrating the eluate on rotary evaporator, final volume was made to 2ml for analysis by gas liquid chromatography (GC).

Estimation

The cleaned extracts were analysed on Hewlett Packard 5890A GC equipped with capillary columns using ^{63}Ni electron capture detector (ECD) and nitrogen-phosphorous detector (NPD). Operating conditions were as per details: For OC and SP insecticides: Detector : ECD (^{63}Ni), column: SPB-5 of 5% diphenyl/ 95% dimethyl fused silica capillary column (30 m×0.32 mm ID, 0.25 μm film thickness) with split system.

Temperatures (^{0}C):150 (5 min) → 8 ^{0}min^{-1} → 190 (2 min) → 15 ^{0}min^{-1} 280° (10 min); injection port: 280; detector: 300; carrier gas: (N_2), flow rate 60 ml min^{-1}, 2 ml through column and split ratio 1:10. Carrier gas, N_2, flow rate 60 ml min^{-1}, 2 ml through column.

For OP and carbamates: Detector: NPD, megabore column: HP-1 of methyl silicone (10 m×0.53 mm ID, 2.65 μm film thickness). Temperatures(^{0}C): Oven: 100 (1 min) → 10^{0}min^{-1} → 200 (0 min) → 20^{0}min^{-1} → 260^{0} (3 min);injector port, 250 , detector, 275 , carrier gas N_2 18 ml min^{-1}, H_2, 1.5 ml min^{-1} and zero air 130 ml min^{-1}.

RESULTS AND DISCUSSIONS

The average percent recoveries at the spiking levels of 0.5 μgg^{-1} of each pesticide were in the range of 80–111, 73–95, 83–125 and 82– 104 for OC, SP, OP and carbamate insecticides, respectively. The data collected during this study is presented in Tables 1 and 2. In the analysed samples, the detected pesticides comprised of OC (HCH isomers [α, β, γ, δ-HCH], DDT analogues, [,o,p-DDT, p, p'-DDT, p, p'-DDE and p, p'-DDD], α-endosulfan, β-endosulfan, endosulfan sulfate), SP (cypermethrin, permethrin, fenvalerate, λ-cyhalothrin and β-cyfluthrin), OP (monocrotophos, dimethoate, malathion, chlorpyriphos and quinalphos) and among carbamates (carbofuran) only. The study revealed that contamination of all the three vegetables were found contaminated with HCH, DDT and endosulfan among the OC group. ∑-HCH was detected in the range of 0.010-0.044 μgg^{-1} in brinjal, 0.034-0.52 μgg^{-1} in cauliflower and 0.027-0.268 μgg^{-1} in okra. Level of contamination was maximum in okra and minimum in brinjal whereas ∑-DDT concentration was maximum (0.056-0.178 μgg^{-1}) in brinjal and minimum (0.018-0.025 μgg^{-1}) in cauliflower. Residues of endosulfan were detected in the range of 0.042-0.057 μgg^{-1} in brinjal, 0.017-0.042 μgg^{-1} in cauliflower and 0.143-0.409 μgg^{-1} in okra showing thereby maximum concentration in okra. Thus in all the three vegetables, ∑-HCH was detected in the range of 0.010-0.268, ∑-DDT, 0.019-0.178 and ∑-endosulfan, 0.029-0.263 μgg^{-1}. Although all the samples were found contaminated with OC insecticides but none of the samples contained residues of any of these insecticides above maximum residue limits (MRL) fixed by Prevention of Food Adulteration Act (PFA) 1954 and FAO/WHO (1996). As many organohalogen pesticides like BHC and DDT have been banned with effect from April 1993, In India, but they have remained in the environment where they continue to be incorporated into plant biomass. In India, practically, DDT has not been phased out completely because it is still used to control the mosquito in public health programmes from where it could enter the agricultural soils and water systems and possibly find its way into crops. Presence of endosulfan in the present study is due to use of endosulfan in almost every crop in Haryana, India among the OC pesticides after banning of use of DDT and HCH in 1993. Residues of cypermethrin (0.003-0.012 μgg^{-1}), permethrin (ND-0.024 μgg^{-1}), λ-cyhalothrin (ND-0.004μgg^{-1}) and β-cyfluthrin(0.047-0.087 μgg^{-1}) in brinjal, permethrin (0.633-0.725 μgg^{-1}) and fenvalerate (0.007-0.017 μgg^{-1}) in cauliflower and only cypermethrin (0.010-0.034 μgg^{-1}) in okra were detected among the SP insecticides. Among OP, chlorpyriphos (0.018-0.031μgg^{-1}) was detected in all the samples of brinjal and cauliflower. This major contaminant was detected in the range of 0.018-0.022 μgg^{-1} in brinjal and 0.024-0.031 μgg^{-1} in cauliflower whereas no sample of okra showed presence of chlorpyriphos residues in detectable amounts. Some other insecticides like monocrotophos, dimethoate, quinalphos and malathion were also detected in detectable amounts in few samples. Only carbofuran, among carbamate insecticides was detected in the range of 0.009-0.020 μgg^{-1} in brinjal. Residues of none of the pesticide exceeded the MRL value. The results obtained from the present study are consistent with an earlier study that show residues of these pesticides are present in different vegetables (Madan *et al.*, 1996; Kumari *et al.*, 2002 and 2003; Deka *et al.*, 2005).

Effects of washing and boiling

Among the household processes, 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

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OP where the residues decreased to the extent of 77, 74 and 50 percent, in brinjal, cauliflower and okra, respectively. Among the carbamate insecticides, carbofuran residues, which was detected in brinjal only, reduced by 21 percent by washing. In the present study, washing was found 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. In earlier studies also, effects of these factors were observed in different vegetables by various researchers (Sarode *et al.*, 1982; Dikshit *et al.*, 1986; Geisman, 1975; Gunther *et al.*, 1963). Farrow *et al.*, 1969; Sarode and Lal, 1982; Elkins, 1989 reported 20-89 percent reduction of DDT in potatoes and tomatoes, fenitrothion in okra, parathion in cauliflower and malathion in okra by washing. In present study, washing was found comparatively less effective in reducing the residues of SP insecticides than that of OC and OP insecticides. Current results are in consistent with some earlier reports where reduction (10-30%) of alphamethrin residues in tomato and brinjal and cauliflower by Gill *et al.*, (2001) and Malik *et al.*, (1999) was found. Reduction of fenvalerate residues on tomatoes to the level of 62 percent was reported by Jain *et al.*, (1979) and 38 percent by Sances *et al.*, (1992). Rinsing of various vegetable was found very effective (Krol *et al.*, 2000).

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. Carbofuran residues detected in brinjal only, reduced to the level of 50 percent by boiling. Thus, great variation in reduction of residues by boiling was observed which may be attributed to the rates of degradation and volatilization of residues as the concentration of residues increases by heat involved in boiling. Hotellier (1982) reported that deltamethrin residues reduced appreciably on cooking. Reduction of fenvalerate residues to an extent of 27-56 percent in brinjal was reported by Sharma and Kumar (1993). Reduction of alphamethrin in the range of 25-32 percent in brinjal and tomatoes and 12-17 percent in cauliflower was reported by Gill *et al.*, (2001) and Malik *et al.*, (1999). Holland *et al.*, (1994) reported appreciably reduction in pesticide residues in different commodities by using different processing methods. Hence, the present results are in consistent with the earlier results.

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Table-1. Pesticide residues* (µg g^{-1}) in Brinjal, Cauliflower and Okra.

Insecticides detected	Brinjal			Cauliflower			Okra		
	1	2	3	1	2	3	1	2	3
α- HCH	0.007	0.003	0.009	0.002	0.005	0.007	0.003	0.025	0.005
β-HCH	0.003	0.001	0.003	ND	ND	ND	0.004	0.024	0.003
γ-HCH	0.010	0.005	0.025	0.008	0.006	0.031	0.018	0.213	0.024
δ-HCH	0.008	0.001	0.007	0.042	0.019	0.007	0.002	0.006	0.006
∑-HCH	0.028	0.010	0.044	0.052	0.030	0.045	0.027	0.268	0.038
o,p'- DDT	0.012	ND	0.001	ND	ND	ND	ND	ND	ND
p,p'- DDT	0.029	0.160	0.038	0.018	0.019	0.025	ND	ND	ND
o,p'- DDE	ND	ND	ND	ND	ND	ND	ND	ND	ND
p,p'- DDE	0.008	0.018	0.012	ND	ND	ND	0.041	0.042	0.053
p,p'- DDD	0.009	ND	0.005	ND	ND	ND	0.018	ND	ND
∑-DDT	0.058	0.178	0.056	0.018	0.019	0.025	0.059	0.042	0.053
α-Endosulphan	ND	ND	0.007	0.006	0.010	0.017	0.013	0.011	0.009
β-Endosulphan	0.017	0.048	0.027	0.001	0.005	0.008	0.084	0.077	0.078
Endo. Sulphate	0.025	0.009	0.013	0.022	0.029	0.035	0.312	0.055	0.176
∑- Endosulfan	0.042	0.057	0.047	0.029	0.044	0.060	0.409	0.143	0.263
Cypermethrin	0.012	0.003	0.008	ND	ND	ND	0.034	0.010	0.014
Permethrin	0.022	ND	0.024	0.673	0.725	0.633	ND	ND	ND
Fenvalerate	ND	ND	ND	0.007	0.011	0.017	ND	ND	ND
λ-Cyhalothrin	0.004	ND	0.002	ND	ND	ND	ND	ND	ND
β -Cyfluthrin	0.087	0.047	0.075	ND	ND	ND	ND	ND	ND
Monocrotophos	ND	ND	ND	ND	ND	ND	0.002	0.005	0.008
Dimethoate	0.001	ND	0.002	ND	ND	ND	0.002	0.002	0.006
Malathion	ND	0.008	0.004	ND	ND	ND	ND	ND	ND
Chlorpyriphos	0.022	0.021	0.018	0.024	0.027	0.031	ND	ND	ND
Quinalphos	0.007	0.002	0.009	ND	ND	ND	0.006	0.002	0.007
Carbofuran	0.020	0.009	ND	ND	ND	ND	ND	ND	ND

*Average of two replicates
ND = Non detected
MRL (mg kg^{-1}) From PFA: HCH (γ-HCH): 3.0; DDT: 3.5; Endosufan: 2.0; Chlorpyriphos: 0.01; Malathion: 0.5; Fenvalerate: 2.0; Cypermethrin: 0.2and 2.0 in Cabbage
MRL (mg kg^{-1}) From FAO/WHO: DDT : 1.0; Endosulfan: 2.0; Cypermethrin: 0.5;Fenvalerate: 0.2; Monocrotophos: 0.2; Carbofuran: 0.1

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Table-2. Effect of processing on pesticide residues (µg g^{-1}) in Brinjal, Cauliflower and Okra.

Insecticides detected	**Range of residues** (Mean) **Brinjal** [% Reduction]			**Range of residues** (Mean) **Cauliflower** [% Reduction]			**Range of residues** (Mean) **Okra** [% Reduction]		
	UW	**W**	**B**	**UW**	**W**	**B**	**UW**	**W**	**B**
∑-HCH	0.010-0.044 (0.027)*	0.007-0.028 (0.015) [44]	0.005-0.023 (0.013) [52]	0.034-0.052 (0.042)	0.022-0.030 (0.027) [36]	0.005-0.019 (0.017) [59]	0.027-0. 268 (0.111)	0.023-0.158 (0.069) [38]	0.021-0.131 (0.059) [47]
∑-DDT	0.056-0.178 (0.097)	0.035-0.107 (0.061) [37]	0.015-0.079 (0.044) [55]	0.018-0.025 (0.021)	0.005-0.021 (0.014) [34]	0.003-0.013 (0.008) [61]	0.042-0.059 (0.051)	0.034-0.051 (0.041) [20]	0.020-0.047 (0.033) [35]
∑-Endosufan	0.042-0.057 (0.048)	0.029-0.042 (0.035) [27]	0.022-0.034 (0.029) [39]	0.029-0.060 (0.044)	0.017-0.042 (0.029) [34]	0.012-0.029 (0.019) [57]	0.143-0.409 (0.280)	0.139-0.263 (0.178) [36]	0.123-0.224 (0.172) [38]
SP	0.002-0.013 (0.027)	0.016-0.026 (0.020) [26]	0.015-0.022 (0.017) [37]	0.650-0.736 (0.688)	0.451-0.648 (0.490) [29]	0.394-0.434 (0.412) [40]	0.010-0.034 (0.019)	0.009-0.020 (0.013) [31]	0.006-0.020 (0.011) [42]
OP	0.008-0.010 (0.009)	0.001-0.004 (0.002) [77]	0.005-0.012 (0.009) [100]	0.024-0.031 (0.027)	0.005-0.008 (0.007) [74]	0.002-0.003 (0.002) [92]	0.003-0.007 (0.004)	0.001-0.003 (0.002) [50]	0.001 (0.001) [75]
Carbamates	0.009-0.020 (0.014)	0.004-0.018 (0.011) [21]	0.001-0.014 (0.007) [50]	-	-		-	-	-

Mean; UW: Unwashed; W: Washed; B: Boiled, Numbers in parenthesis [ ] is % reduction of residues.

CONCLUSIONS

It can be concluded that residues of none of the pesticides exceeded their respective maximum residue limits. Processing substantially lowers the residues of pesticides in vegetables. The percentage reductions in the present study are supported by both early and most recent publications. 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. The present study showed that boiling was found more effective than washing, which further support the fact that at least the vegetables under study are not consumed in raw form.

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