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Removal of endosulfan residues from vegetables by household processing

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Effect of washing, peeling and cooking on endosulfan residue (ER) levels was determined in winter (spinach, cauliflower, potato) and summer vegetables (brinjal, tomato, okra) grown under controlled supervised field trials. Highest ER was found at raw stage in brinjal (2.43 mg/kg) followed by okra (1.83 mg/kg) and spinach (1.25 mg/kg) and lowest in potato (0.177 mg/kg). ER in vegetables was found to be reduced as follows: washing, 15-30; peeling, 60-67; and cooking, 13-35%. High levels of ERs exceeding maximum residue limits (MRLs) were observed in brinjal and tomato samples at raw stage. However, vegetable household processing reduced ER below the MRLs.

Keywords: Cooking, Endosulfan, Supervised field trial, Vegetables, Washing

Introduction

Vegetables absorb applied insecticides and often create health hazards to human while consuming fresh either raw or without much processing leading to adverse effect on human health, in addition to disturbance in ecosystem[1]. Pesticides use is increasing in Asia, Africa, Central and South America because of high demand for good quality products and urgent need for self-sufficiency in food production[2]. Pakistan is a large consumer of pesticides in the world after India and the 2[nd] highest among the South Asian countries. About 27 % of the total pesticide consumption is used on fruit and vegetable crops[3]. In Pakistan, annual consumption of endosulfan for vegetable crop production is higher than that of other pesticides. Endosulfan, an organochlorine insecticide containing sulphite group and marketed as a mixture of two isomers having the same insecticidal activity, is highly persistent, practically insoluble in water, non-volatile and stable[4]. Acute oral LD_{50} for rat is 80-110 mg/kg. Chronic poisoning resulting from food residues causes kidney and liver damage[5]. Several food-processing operations (peeling, washing and heat processing) can reduce pesticide residues in various food crops[6-9].

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Present study determines residue level and dissipation behavior of endosulfan during washing, peeling and cooking in vegetables grown at supervised field trials.

Materials and Methods

Field Trials for Endosulfan

Endosulfan residue (ER) was determined in winter [spinach (*Spinacia oleracea* L.), cauliflower (*Brassica oleracea* L.), potato (*Solanum tuberosum* L.)] and summer vegetables [brinjal (*Solanum melongena* L.), tomato (*Lycopersicon esculentum* Mill.), okra (*Abelmoschus esculentus* L.)] grown at the Institute of Horticultural Sciences, University of Agriculture, Faisalabad, Pakistan in separate plots (150' x 30' each) in 3 replicates. Vegetables were sprayed with a endosulfan-based product (Thiodan 35 EC 9 ml/l water). As per the pest population, winter vegetables were sprayed twice, and summer vegetables thrice during growth and harvested after 7 days of last spray, with a Knap-sack hand sprayer in the absence of precipitation and wind.

Processing of Vegetables

Potato and brinjal samples, after washing, were hand peeled into slices with a stainless steel peeling knife and cooked. Spinach, cauliflower, tomato and okra were washed, sliced into a suitable size and cooked. Vegetable samples (raw) were dry cleaned to remove soil contamination with a disposable paper towel and blended in a Warring Blender (WB) to make a homogenous sample for subsequent endosulfan analysis.

Washing

Vegetables were washed by placing in a plastic colander and rinsed under normal tap water (25-30°C) for 30 sec[9] with gentle rotation by hands and blotted dry with a paper towel. These samples were divided into two portions, of which one was analyzed as such after homogenizing in a WB and other was further peeled and cooked.

Peeling

After washing, potato and brinjal were peeled into appropriate size with peeling knife and categorized into two portions, of which one was subjected to endosulfan analysis, and \other was further subjected to cooking. Remaining vegetables (spinach, cauliflower, okra and tomato) were reduced into suitable size and further processed.

Cooking

Sliced vegetables were cooked[10] by placing ¾ cup of water in the saucepan and adding ½ teaspoon of salt. Vegetable (50 g) was added immediately in boiled water. Saucepan was covered with a lid and the vegetable boiled for 10-12 min were subjected to endosulfan analysis.

Extraction of Endosulfan Residue (ER)

ERs were extracted[11] from all the samples. Vegetables (50 g) were homogenized with ethyl acetate (50 ml) in the presence of anhydrous sodium sulphate (20 g) and sodium chloride (10 g) in a WB at high speed for 3 min. Homogenate was filtered through Whatman No. 40, transferred directly to a column of activated charcoal, and left to pass through the column at an approx rate of 1 ml/min. Filtrate was taken for drying in a rotary evaporator at 35°C and redissolved in an ethyl acetate-cyclohexane (1:1, v/v, 10 ml) solution and filtered through a 0.45-μm filter membrane. Then sample was cleaned by gel permeation chromatography using a column with Bio-Beads SX-3 (mesh size 200-400) and ethyl acetate-cyclohexane (1:1, v/v) as eluting solvent. The collected elute was reduced to a small volume under a gentle stream of nitrogen gas and analyzed for endosulfan by HPLC.

HPLC Analysis of Endosulfan

ERs were redissolved in mobile phase (methanol; 300-μl) and analyzed[12] by HPLC, Perkin Elmer Series 200. Analyses were performed in isocratic system using a reverse phase C18 column (25 cm x 4.6 mm id). For separation of endosulfan, a 70:30 mixture of methanol and water was passed through the column at a flow rate of 1.0 ml/min and UV wavelength at 214 nm. Column temperature was ambient and injection was made with a 50-μl SGE syringe and a manual loop injector (20-μl). Sample filtration was carried out with solvent compatible Gelman acrodisc syringe filter of 0.20-μm GHP membrane. The eluent was degassed by purging with helium. The concentration was calculated to compare peak area of standard endosulfan as external standard. Mean values and standard deviations were calculated and analysed by Minitab Software Package Version 14.0 (Minitab Inc. State College, PA, USA).

Results and Discussion

Vegetables are short duration crops and in subtropical climate prevailing during growth and maturity of vegetables, it is necessary to spray a pesticide on crop plants repeatedly during the entire period of growth and sometimes even at the harvesting stage[1].

Recovery of endosulfan (mean of 5 replicates) in vegetables was as follows: spinach, 103 ± 2.5; cauliflower, 95 ± 1.2; potato, 98 ± 0.68; brinjal, 92 ± 4.5; tomato, 101 ± 2.8; and okra, 100 ± 0.25%. Highest ER level in raw vegetable sample was detected in brinjal (2.43 mg/kg) followed by okra (1.83 mg/kg) and spinach (1.25 mg/kg) and lowest in potato (0.18 mg/kg). Same trend of relative ER levels was found in the same samples after washing (Table 1). Retention of ER after cooking was found as follows: spinach, 0.73; cauliflower, 0.26; potato, 0.04; brinjal, 0.76; tomato, 0.30; and okra, 0.83 mg/kg. In brinjal and tomato samples at raw stage, ER was above maximum residue limits[13] (MRLs). Many countries have restricted the use of organochlorine pesticides mainly in food applications because of their long persistence and high stability[14].

Washing decreased ER (Table 1) in okra (30%), followed by tomato (25%), spinach (22.2%) and brinjal (10.3%). Although solubility of pesticide in water is an important factor during washing operation, correlation between solubility of pesticide and removal percentage has not been reported[9]. In the present study, water solubility was not found to be the important factor during washing operation. Residues of six pesticides on olives decreased after washing with no correlation to water solubility of the pesticides[15].

Table 1 — Endosulfan residue in vegetables of supervised field trial

Vegetables	MRL mg/kg	Raw, mg/kg Mean*	CV	Washed, mg/kg Mean*	CV	Peeled, mg/kg Mean*	CV	Cooked, mg/kg Mean*	CV
Spinach	2.0	1.25 ± 0.044	3.49	0.972 ± 0.077	7.87	NA	NA	0.733 ± 0.058	7.85
Cauliflower	0.5	0.408 ± 0.066	6.07	0.347 ± 0.026	7.49	NA	NA	0.260 ± 0.017	6.47
Potato	0.2	0.177 ± 0.003	1.49	0.141 ± 0.013	9.27	0.046 ± 0.004	9.48	0.038 ± 0.003	6.96
Brinjal	2.0	2.43 ± 0.216	8.90	2.180 ± 0.217	9.93	0.872 ± 0.019	2.20	0.758 ± .049	6.46
Tomato	0.5	0.556 ± 0.020	3.59	0.417 ± 0.023	5.41	NA	NA	0.3 ± 0.020	6.56
Okra	2.0	1.83 ± 0.115	6.30	1.28 ± 0.149	11.67	NA	NA	0.832 ± 0.018	2.19

*Mean are replicate of 5 observations; NA, No peeling; MRL, Maximum residue limits; CV, Coefficient of variation

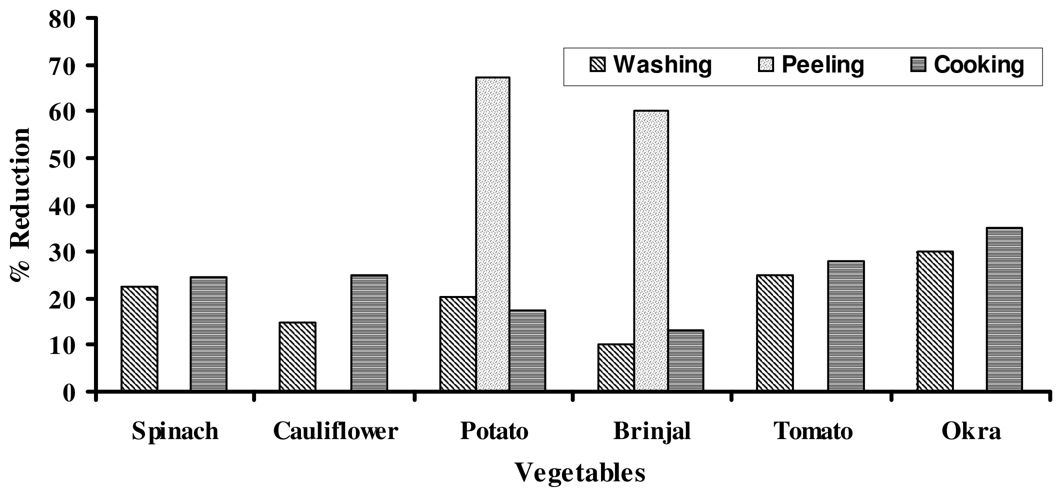

Fig. 1 — Percent reduction of endosulfan residues in vegetables of supervised field

Peeling had a significant effect on ER removal in the skin of potato (67%)and brinjal (60%). Heat treatment during cooking had a significant effect on the reduction (13-35%) of ER in all the tested vegetable samples. Effect was more pronounced in okra and tomato while least effect was observed in potato and brinjal (Fig. 1), which may be due to the nature of different matrices. However, present results agree fairly with previous findings[8,16,17].

Conclusions

Washing, peeling and cooking of vegetables contribute significantly for the reduction of pesticide residues to below MRL values. Pesticide applications may be avoided particularly at crop harvesting.

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