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Evaluation of different household practice to decontaminate synthetic pyrethroid insecticide residues from *Amarnathus tricolor* L.

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Abstract
Amaranth (*Amaranthus tricolor* L) is a major leafy vegetable extensively consumed in Kerala. Farmers have been using pesticide in a wide range to protect crop from pests. This resulted in high residues of pesticides on amaranth. A preliminary survey was conducted to monitor pesticide residues on amaranth. Among the insecticides using on amaranth organo phosphates and synthetic pyrethroid insecticides are major insecticides group. Here to evaluate efficiency of different decontaminating methods in removal of synthetic pyrethroid pesticide residues from amaranth plants were sprayed with different SP insecticides *viz.,* bifenthrin 10% EC, cypermethrin 25% EC, fenvalerate 20% EC and Lambda cyhalothrin 5% EC were subjected for decontamination methods viz., washing, 2% common salt, 2% vinegar, 1% turmeric, 2% tamarind, 1% veggie wash (produced by Kerala Agricultural university, Vellayani) and cooking. The residues in control and processed plants were estimated by using Gas Chromatograph equipped with Electron Capture Detector. Dipping in 2% tamarind, 1% veggie wash for 10 minutes followed by three further washings + cooking and 2% common salt were found to be effective in removal of SP insecticide residues at the average of 67%, 64% and 63%. This study helped to standardize simple and effective method in removal of most commonly used synthetic pyrethroid insecticide's residues in amaranth.

Keywords: Amaranth, pesticide, residues, decontamination

Introduction
Amaranth is extensively cultivated as a green leafy vegetable and grain crop in many temperate and tropical regions. Amaranths have excellent nutritional value because of their high content of essential micronutrients such as β-carotene, iron, calcium, zinc, phosphorous and calcium (Schonfeldt and Pretorius, 2011) [11]. *A. tricolor* is rich in vitamin C and folic acid. In India, it is cultivated largely in the southern states. It is raised throughout the year in paddy lowlands, garden lands and homesteads. The crop cultivated throughout the year. Farmers use chemical pesticides in wide range. This tendency leads to high residues on amaranth crop. Nair *et al.,* (2013) [6, 7] reported that most of the agriculture commodities tested had multiple residues containing three to six pesticides. Department of Agriculture, Government of Kerala and Kerala Agricultural University through the Plan Scheme "Production and marketing of safe to eat (pesticide free) vegetables, fruits and food products for sale through government outlets" revealed that out of 37 red amaranthus samples analyzed during period of January – December 2013, 7 samples were found as unsafe with high level of residues, in these detected insecticides most of residues belongs to organophosphate group (PAMSTEV, 2014). However, these pesticides widespread use together with their unique physical, chemical and biological properties has raised serious concern among the public regarding their adverse effects on human health and environment. So there is need to remove pesticide residues from commodity at consumer level. Standardization of simple cost effective methods to remove residues on amaranth has been reported here.

Materials and methods
A preliminary survey was conducted to assess the pesticide residues in amaranth in Kalliyoor and Pappanchani of Thiruvananthapuram district during March 2014- June 2014. Out of 20 samples tested, 17 samples were contaminated with pesticide residues. In 17 contaminated samples the detected insecticides were four organo phosphates and four synthetic pyrethroids (Muralikrishna *et al.,* 2016) [5]. The red amaranth plants were grown organically in polythene grow bags in premises of PRRAL, Vellayani were used for the standardisation of household

practices to decontaminate residues. An insecticide mixture emulsion (100 ppm) was prepared by using required quantities of each insecticide mixed in one litre water. The insecticide used were bifenthrin 0.4 ml L^{-1} (MARKER), lambda cyhalothrin 0.5 ml L^{-1} (KARATE), cypermethrin 1.0 ml L^{-1} (CYMBUSH 25 EC) and fenvalerate 0.2 ml L^{-1} (FENVAL 20 EC). The amaranth plants were sprayed with this insecticide mixture using a hand sprayer (1 L). Sprayed plants were kept under covered conditions to protect from rain. Treated plants were harvested at one day after spraying. Individual plants were subjected to different decontamination practices and some plants were kept as un processed control for comparison. Preparation of working standards and analysis of each sample processed according to specific procedure for vegetables and samples were analysed under specific working parameters of Gas Chromatography (Nair *et al*., 2013) [6, 7]. The residues in processed and unprocessed (control) samples were extracted in acetonitrile and clean-up was done with QuEchERS method. The different decontamination treatments used in this experiment are mentioned in Table 2.

Results and discussion

The recovery and repeatability of four SP insecticides are presented in table 1. The effect of different household practices in removal of SP pesticide residues from amaranth are summarized in Table 2. The treatments varied significantly on their effect of remove bifenthrin residues. Tamarind was found to be superior over all other treatments (0.86 ppm) and it was on par with KAU veggie wash plus cooking (0.92), common salt (1.00), cooking (1.02). Whereas vinegar (1.10), water (1.12) and turmeric (1.17) were found to be next best treatments and KAU veggie wash (1.49) found to be less effective.

Table 1: Recovery and repeatability of synthetic pyrethroid insecticides in amaranth at different fortification levels

S. No	Insecticides	Level of fortification					
		LOQ (0.05 mg kg $^{-1}$)		5 x LOQ (0.25 mg kg $^{-1}$)		10 x LOQ (0.5 mg kg $^{-1}$)	
		Mean recovery (%) ± SD	RSD (%)	Mean recovery (%) ± SD	RSD (%)	Mean recovery (%) ± SD	RSD (%)
1	Bifenthrin	90.80 ± 7.40	8.15	94.69 ± 1.94	2.05	92.72 ± 5.62	6.063608
2	Lambda cyhalothrin	89.85 ± 2.87	3.20	97.84 ± 1.61	1.64	94.58 ± 3.32	3.51238
3	Cypermethrin	93.01 ± 4.49	4.83	96.56 ± 0.86	0.89	92.46 ± 3.45	3.736445
4	Fenvalerate	74.12 ± 3.13	4.22	90.68 ± 3.29	3.63	92.54 ± 2.98	3.220281

Table 2: Extent of removal synthetic pyrethroid insecticides residues from amaranth

Treatments	Bifenthrin		Lambda-cyhalothrin		Cypermethrin		Fenvalerate	
	Residues (ppm)	% removal	Residues (ppm)	% removal	Residues (ppm)	% removal	Residues (ppm)	% removal
T1- Water*	1.12	54.59	1.16	61.23	0.99	65.12	1.38	63.06
T2-2%Tamarind*	0.86	65.33	1.07	65.54	0.83	71.17	1.28	66.73
T3- 2% Common salt*	1.00	60.15	1.28	58.64	0.97	65.87	1.11	69.88
T4-1%Turmeric*	1.17	52.61	1.38	55.36	1.14	60.14	1.50	60.45
T5-2% Vinegar*	1.10	55.24	1.31	58.25	1.06	61.89	1.74	54.41
T6-1% KAU Veggie wash*	1.49	40.28	1.45	42.34	1.53	48.39	2.12	43.90
T7-1% KAU Veggie wash* + cooking	0.92	61.03	1.10	66.05	1.04	62.72	1.30	65.07
T8- Washing* + cooking	1.02	58.83	1.14	63.13	1.13	60.12	1.57	59.75
Untreated	2.54	--	3.17	--	2.95	--	3.96	--
CD (0.05)	0.244	12.841	0.339	12.559	0.354	16.707	0.518	16.342
MRL (EU)	0.05		1.00		0.70		0.02	

In removal of lambda cyhalothrin residues KAU veggies wash plus cooking (1.07) was found as superior. More or less similar amount of residues were removed when subjected to tamarind (1.10), cooking (1.14), water (1.16), common salt (1.28), vinegar (1.31) and turmeric (1.38). All the above treatments were statistically not different. In removal of lambda cyhalothrin KAU veggie wash alone (1.45) found as inferior among all the treatments. In case of cypermethrin highest reduction of residues removal was observed when subjected to tamarind (0.83) and this was on par with common salt (0.97), water (0.99) and KAU veggie wash plus cooking (1.04). These treatments were followed by cooking (1.13) and turmeric (1.14) and these two treatments did not show any significant variation. However, KAU veggie wash alone was the least effective treatment.

Among the all treatments, the superior treatment for removing fenvalerate residues was common salt (1.11) and this was followed by tamarind (1.28), KAU veggie wash plus cooking (1.30), water (1.38), turmeric (1.50) and cooking (1.57). All the above treatments were statistically on par. Next best removal observed in vinegar treated samples (1.74). Whereas least amount of fenvalerate residue removal observed in KAU veggie wash.

In removal of synthetic pyrethroid pesticide residues from amaranth all treatments including water, tamarind (2%), common salt (2%), turmeric (1%), vinegar (2%) and KAU veggie wash (1%) and cooking process such as washing plus cooking and cooking after washing with KAU veggie wash (1%) showed significant effect when compared with untreated samples. Among all treatments cooking treatments removed significantly more amount of residues when compared to dipping in water and washing with different treatments. Maximum removal of pesticide residues was noticed in amaranth plant dipped in KAU veggie wash (1%) for 10 minutes plus washing and cooking. These results agree with observations of Vemuri *et al.* (2015) [16] who reported synergic effect of ionisation of residues in acidic solution and thermal effect which together dislodged 99 to 100 per cent residues. Whereas the combination of washing with water (dipping of amaranth plants in water for 10 minutes) and cooking for 10 minutes had given satisfactory removal. These results may be influenced by the physico-chemical properties of the pesticides. The loss of pesticide residues during heat

processing may be due to evaporation, co-distillation, thermal degradation which vary with the chemical nature of the individual pesticides (Sharma *et al.* 2005 and Balinova *et al.*, 2006) [12, 1].

Up to 71.17 per cent of residues of cypermethrin were removed when amaranth plants were subjected to dipping in tamarind (2%) for 10 minutes followed by three normal washings with tap water. Kumar (1997) [3] reported the effectiveness of tamarind solution in removing residues of phosphamidon and monocrotophos from bittergourd and cowpea pods. Singh *et al*. (2007) [13] reported that tamarind pulp had significant amount of organic acids, of which tartaric acid (98%) the major one is having a pH of 2.7. In removal of bifenthrin, lambda cyhalothrin and fenvalerate also tamarind (2%) was more or less superior to other treatments. Varghese and Mathew (2013) [14] reported that two per cent tamarind solution was the best decontaminating solution in removing residues of spiromesifen (90.03%) and propargite (96.69%) from green chilli fruits.

When amaranth plants were dipped in two per cent common salt solution for 10 minutes followed by three normal washing in water maximum reduction observed fenvalerate among all insecticides and it was followed by cypermethrin, bifenthrin and lambda cyhalothrin. Lalah and Wandiga (2002) [4] reported that dipping of beans in two per cent salt solution for five minutes removed 59 per cent of malathion residues. These results agree with those of Nair *et al.*, (2014) [8] who reported that up to 68 per cent of organophosphate and 50 per cent of synthetic pyrethroid insecticide residues were dislodged in okra by subjecting to two per cent common salt for 15 minutes. The cause and effect of the reduction in two per cent common salt washing solutions is still not known and needs further investigation.

In case of vinegar (2%) 50%-60% of synthetic pesticide residue removal observed. Varghese (2011) [15] reported that the polar nature of insecticides is the deciding factor in removal by vinegar. These results with those obtained by Nair (2013) [6, 7] who reported that dipping of curry leaf in two per cent vinegar for 15 minutes resulted in up to 93 per cent of organo phosphate residues and up to 66 per cent residues removal at one day after spraying. Where as in case of turmeric also more or less same amount of residues removed when compared to vinegar (2%). When amaranth plants were dipped in 1 per cent KAU veggie wash least amount of residues removal observed. These results were not in agree with findings of Muralikrishna *et al.*, (2016) [5] who reported 1% KAU veggie wash was effective in removal of organo phosphate insecticide residues from amaranth plants. The chemical nature of synthetic pyrethroid insecticides may be the reason for less removal when compared to organo phosphate insecticides. More removal noticed in organophosphate insecticide residues case than in synthetic pyrethroid insecticides it may be because of higher solubility for organophosphate insecticides than synthetic pyrethroids and also because of polarity of compound (PPDB, 2015) [10].

The efficiency of treatments differed with respect to different insecticidal chemistries. The effect of processing depends upon many factors such as water-octanol partition coefficients, water solubility, heat stability, vapour pressure. For removal of SP pesticide residues washing with acidic solutions or with different treatments followed by cooking was very effective when compared to washing alone with different treatments. This was supported by results of Dikshit (2001) [2]. From the above results it is clear that dipping in two percent tamarind, dipping one per cent KAU veggie wash for 10 minutes followed by cooking and washing with water followed by cooking are recommendable treatments to remove pesticide residues from amaranth.

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