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MULTIRESIDUE METHOD FOR DETERMINATION OF PESTICIDES IN CAULIFLOWER SAMPLES WITH GAS CHROMATOGRAPHY

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ABSTRACT

India finds it difficult to suffice the needs of ever growing population and use of pesticides in vegetables has increased several folds in the last several years. In tropical conditions, fruits and vegetables are grown throughout the year and cauliflower is highly nutritious and fibrous, helps in scavenging free radicals from the body to prevent incurable diseases. Vegetable samples of cauliflower collected from Bangalore urban district and subjected to gas chromatography equipped with ECD and FTD. Recovery studies performed at fortification levels of 1.0, 0.5 and 0.01 mg/kg and the average recovery rates ranged from 77.3 to 94.7 %. Variation in acephate, chlorpyriphos, dichlorvos, monocrotophos, phorate, cyfluthrin-β, cyhalothrin-λ, cypermethrin, deltamethrin and fenvalerate residues in cauliflower samples is recorded. It is found that 12.5 % of samples from Bangalore urban were having phorate residue value above the MRL of 0.05mg/kg. Pesticides though present in cauliflower sample in Bangalore urban district and not exceeded the MRL.

KEYWORDS: Cauliflower, pesticides, gas chromatography.

INTRODUCTION

India has varied agro-climatic zones and factors responsible for growth and development of vegetables differ across the country. India being the largest producer of vegetables after China and accounts for 13.4 % of world production. Pesticides, the plant protection chemicals are widely used in agriculture to increase the yield, improve the quality and extend the storage life of food crops (Fernandez-Alba and Garca-Reyes, 2008). Pesticides are widely used during production and post-harvest treatment of agricultural commodities (Levitt and Wehr, 2001). In India, crops are considerably damaged by more than 200 pests and 100 plant diseases. Loss of food grains is estimated to be 23 % and 25 % respectively due to insects and diseases (Mauskar, 2007). Based on the chemical compositions, pesticides are classified into Organochlorines, Organophosphates, synthetic pyrethroids, carbamates and Biopesticides. Pesticides are found to affect the pest metabolism, interfere with the hormones and disrupt plant growth and development. Agriculture is the backbone of 1.25 billion population with diminishing cultivable land. To policy makers, the increased use of inputs like fertilizers and chemical pesticides often seems to be most effective ways to increase production and food supply, since a good part of produce is lost through diseases, pest and weeds in the field and storage.

Pesticides remain the main stay in the agri-horticultural production in both developed and developing countries. Organophosphates (36 %) dominated the insecticide market followed by Pyrethroids (25 %), carbamates (21 %), organochlorines (8 %) and other (10 %) (Dhaliwal and Pathak, 1993).

MATERIALS AND METHODS

Sampling: Eight samples of cauliflower collected from the growing areas of Bangalore urban district during January to March 2018. The samples were kept in a refrigerator (5 ºC) till analysis. All the samples were extracted fresh and information regarding pesticide applied to vegetable crops was collected from farmers at the time of sampling. Composite samples consisted of 1kg was cut into small pieces and macerated in a grinder (Borgert *et al*. 2003 and Colborn *et al*. 1993).

Chemicals: The glasswares rinsed with acetone and dried in an oven at around 350 ºC prior to use. All solvents like n-hexane, acetonitrile, petroleum ether and diethyl ether (HPLC grade) were procured from Sigma

Aldrich co. and were glass distilled before use. Sodium chloride and anhydrous sodium sulfate (AR grade) procured from Himedia Pvt. Ltd, India. Before use, anhydrous sodium sulfate was purified with acetone and heated for 4h at 600^0 in muffle furnace to remove possible phthalate impurities. Florosil, 60-100 mesh, Merck India limited was activated at 350 ºC for 5 h before use. The pesticide standards were procured from All India Network Project on pesticide residues, Division of Agriculture Research Institute (IARI), Delhi, India (Obano and Hori, 1996 and Tekel and Hatrik, 1996).

Preparation of standard solution: An accurately weighed 10 mg of an individual analytical grade pesticide was dissolved in 10 ml volumetric flask using n-hexane to prepare the standard stock solution to 1000 mg kg^{-1}. Standard stock solution of each pesticide was serially diluted to obtain immediate lower concentration of 100 mg kg^{-1}. A mixture of standard stock solution was prepared by taking 0.1 ml solution of compatible (Acephate, aldrin, chlorphyriphos, cyfluthrinβ, cyhalothrin, cypermethrin, deltamethrin, dichlorvos, deldrin, α-endosulfan, β-endosulfan, endosulfan-sulfate, fenvalerate, α-HCH, β-HCH, γ-HCH, heptachlor, monocrotophos, phorate and prefenofos) pesticide in a 10 ml volumetric flask and making the volume up to the mark with n-hexane. Standard mixture contained 10 mg kg^{-1} of solution to determine the time of detection and stored in a refrigerator at (Abiodun Falodun *et al*. 2009 and Torres, 1995).

Extraction and cleanup: Fresh vegetable samples were thoroughly shredded and homogenized. Approximately 20 gm of the sample was macerated with 40 ml of ethyl acetate. Sodium hydrogen carbonate (5 g) and anhydrous sodium sulfate (20 gm) was added to remove moisture and further macerated for 3 minutes in ultra-turax macerator. Samples were centrifuged for 5 min at 3000 rpm to obtain two phases. Extraction process was followed by clean-up step using solid-phase extraction with florosil. Florosil column (500 mg/8ml) cartridge was conditioned with 10 ml ethyl acetate. Pesticide in sample extract (5 ml) was eluted with 10 ml of ethyl acetate. Concentrated to 1 ml using rotary evaporator and dried by a gentle nitrogen gas and was dissolved in 1 ml of ethyl acetate. Pesticide was then quantified by gas chromatograph and Electron Capture Detector (GC-ECD) and the analysis was carried out in pesticide analysis laboratory, Department of Agriculture, Government of Karnataka, Bangalore (Abhilash, 2009).

RESULTS AND DISCUSSION

Cauliflower samples analyzed for pesticides like acephate, chlorphyriphos, Dichlorvos, Monocrotophos, Phorate, cyfluthrin-β, cyhalothrin-λ, cypermethrin, deltamethrin and fenvalerate. 37.5 % of cauliflower samples showed contamination with acephate ranging from 0.143 to 0.221 mg/kg (Mean=0.072 mg/kg). Kousik and Balwinder (2010) reported residue of acephate in the range of 0.05-0.37 mg/kg in the farmgate samples of cauliflower from Punjab while Hjorth *et al*. (2011) showed the residue concentration of acephate in fruits and vegetable samples from South America as 0.06-0.028 mg/kg. 25 % of cauliflower is contaminated with dichlorvos and none of the samples crossed the MRL of 0.15 mg/kg (Table 1). Beenakumari *et al*. (2003) showed dichlorvos residue concentration ranging from 0.004-0.022 mg/kg in Cabbage, cauliflower, tomato, potato and green chilly samples collected from wholesale markets of Hisar, Haryana. Phorate values ranged from 0.011 to 0.023 mg/kg (Mean=0.007) and are well within the stipulated MRL values (0.05 mg/kg). Liang Wang *et al*. (2008) revealed the presence of phorate in Shanghai green (0.0257 μg/g) from Nanjing, China.

Table 1: Pesticide residues (mg/kg) in cauliflower samples.

Sl. No.	Chemicals	a(b)	Min	Max	Mean
1	Acephate	3(37.5)	0.143	0.221	0.072
2	Chlorpyriphos	BDL	BDL	BDL	BDL
3	Dichlorvos (DDVP)	2(25)	0.014	0.016	0.004
4	Monocrotophos	BDL	BDL	BDL	BDL
5	Phorate	3(37.5)	0.011	0.023	0.007
6	Cyfluthrin-β	BDL	BDL	BDL	BDL
7	Cyhalothrin-λ	1(12.5)	BDL	0.322	0.04
8	Cypermethrin	BDL	BDL	BDL	BDL
9	Deltamethrin	BDL	BDL	BDL	BDL
10	Fenvalerate	2(25)	0.027	0.052	0.01

BDL: Below detection level, a: Number contaminated, b: % contaminated.

Chen *et al*. (2011) reported phorate residues in fruits and vegetables vary from BDL to 0.405mg/kg from Xianen, China. Cyhalothrin-λ (Maximum=0.322 mg/kg) and fenvalerate though present in the cauliflower samples but none crossed the MRL values. Kousik and Balwinder (2010) reported cyhalothrin-λ (0.14 mg/kg) farmgate samples of cauliflower from Punjab and Hjorth *et al*. (2011) analyzed cyhalothrin-λ residue value of 0.057-0.125 mg/kg in fruits and vegetables from South America. Maria *et al*. (2011) reported the presence of fenvalerate in the range of 0.061-0.07 mg/kg in vegetables like green pepper, white onion, tomato, potato

produced in Sonara, Mexico. Chlorphyriphos, cypermethrin, monocrotophos, Deltamethrin and fenvalerate was not detected in any of the samples analysed.

CONCLUSION

Bangalore enjoys tropical climatic condition throughout the year and demand for the vegetables is enormous in the city due to large populace living in the city. Following are the recommendations made keeping in mind sample contamination rates and type of pesticides being used in the study area. It is necessary that these recommendations are addressed from time to time for improvement.

- Regular monitoring of the vicinity should be encouraged to avoid possible consumption of contaminated foodstuff.
- For accurate and rapid analysis of pesticide residues, standard methods must be developed.
- Farmers should be educated and encouraged not to use higher dosage of pesticides. In order to avoid residue problems, rotation of pesticides to combat the pests and diseases are recommended.
- A regular training/workshop on the use and safety measures to be followed should be imparted to farmers, retailers, distributors, consumers policy makers and other stake holders.
- Multimedia awareness activities in local language should be massively conducted on the dangers posed by pesticides contamination in the food.
- Proper legislations on handling of pesticide should be introduced and practiced.

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