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Multiresidue method for determination of pesticides in Cauliflower samples with gas chromatography.

KEYWORDS

Cauliflower, pesticides, 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 0.01mg/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.*

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 was collected from the growing areas of Bangalore rural district during January to March 2016.The samples were kept in a refrigerator (5^0) 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 glassware were rinsed with acetone and dried in an oven at around 350^0c 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 Himediapvt.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, 60-100mesh, Merck India limited was activated at 350^0c for 5h before use. The pesticide standards was procured from All India Network Project on pesticide residues, Division of Agriculture Research Institute (IARI), Delhi, India (Obano, H and Hori, S. 1996 and Tekel, J and Hatrik, S. 1996).

Preparation of standard solution:

An accurately weighed 10mg of an individual analytical grade pesticide was dissolved in 10ml volumetric flask using n-hexane to prepare the standard stock solution to 1000mg kg^{-1}. Standard stock solution of each pesticide was serially diluted to obtain immediate lower concentration of 100mg kg^{-1}. A mixture of standard stock solution was prepared by taking 0.1ml solution of compatible (Acetate, 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 10mg kg^{-1} of solution to determine the time of detection of the instrument and stored in a refrigerator at (AbiodunFalodun, et al., 2009 and Torres, CM.,1995).

Extraction and cleanup:

Fresh vegetable samples were thoroughly shredded and homogenized. Approximately 20gm of the sample was macerated with 40 ml of ethyl acetate. Sodium hydrogen carbonate (5g) and anhydrous sodium sulfate (20gm) was added to remove moisture and further macerated for 3 minutes in ultra-turax macerator. Samples were centrifuged for 5 min at 3000rpm to obtain two phases. Extraction process was followed by clean-up step using solid-phase extraction with florosil. Florosil column (500mg/8ml) cartridge was conditioned with 10 ml ethyl acetate. Pesticide in sample extract (5ml) was eluted with 10 ml of ethyl acetate.Concentrated to 1ml using rotary evaporator and dried by a gentle nitrogen stream 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, PC.,2009).

Results and discussions:

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.,

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(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 0.15mg/kg. Beenakumari et al., (2003) showed dichlorvos residue concentration ranging from 0.004-0.022mg/kg in Cabbage, cauliflower, tomato, potato and green chilly samples collected from wholesale markets of Hisar, Haryana.Phorate values ranged from 0.011 to o.o23mg/kg (Mean=0.007) and are well within the stipulated MRL values (0.05mg/kg). Liang Wang et al., (2008) revealed the presence of phorate in Shanghai green (0.0257µg/g) from Nanjing, China. Chen et al., (2011) reported phorate residues in fruits and vegetables vary from BDL to 0.405mg/kg from Xianen, China. Cyhalothrin-λ (Maximum=0.322mg/kg) and fenvalerate though present in the cauliflower samples but none crossed the MRL values. Kousik and Balwinder (2010) reported cyhalothrin-λ (0.14mg/kg) farmgate samples of cauliflower from Punjab and Hjorth et al., (2011) analyzed cyhalothrin-λ residue value of 0.057-0.125mg/kg in fruits and vegetables from South America. Maria et al., (2011) reported the presence of fenvalerate in the range of 0.061-0.07mg/kg in vegetables like green pepper, white onion, tomato, potato produced in Sonara, Mexico. Chlorphyriphos, monocrotophos, cypermethrin, 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. Bangalore due to

		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

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