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Determination of Pesticide Residues in Spinach

KEYWORDS: spinach , QuEChERS method, chlorpyrifos,profenofos, triazophos, and LC-MS/MS.

Geetha G	Sreenivas C	Shashi Vemuri
Department of Entomology, College of Agriculture, PJTSAU, Rajendranagar, Hyderabad- 500 030, Telangana, India	Department of Entomology, College of Agriculture, PJTSAU, Rajendranagar, Hyderabad- 500 030, Telangana, India	Department of Entomology, College of Agriculture, PJTSAU, Rajendranagar, Hyderabad- 500 030, Telangana, India

ABSTRACT *Studies were conducted for determining the residues of commonly used pesticides in spinach leaf samples collected from three different local vegetable markets viz., Shamshabad, Mehadipatnam and Gudimalkapur in and around Hyderabad during 2014. A total of 30 samples were collected from three markets of Hyderabad and analysed using QuEChERS method on LC-MS/MS. The most commonly detected pesticide residues were chlorpyrifos, profenofos, triazophos, deltamethrin + triazophos, cypermethrin, acephate, dimethoate, imidacloprid, spinosad A, acetamiprid and indoxacarb (Table.1.).However chlorpyrifos, triazophos,cypermethrin, deltamethrin+ triazophos and profenophos were frequently observed indicating that leaf samples contained detectable level of the pesticides residues for which Maximum Residue Limits (MRL) are not fixed. As there are no MRLs for spinach leaves, it should be considered as most important to fix MRLs to ensure food safety and consumer health and to create awareness among the farmers about the application dose, method of application and Pre Harvest Intervals. The mismanagement or non-availability of proper information about the pesticide application can lead to contamination of pesticide residues in spinach. The findings of this study provided important data about contamination of pesticide residues in spinach sold in the local markets of Hyderabad and hence, it is essential to conduct monitoring studies in other spinach growing districts and states , which may serve as basis for future policy about the standards and quality control of pesticides.*

Introduction :Spinach (*Spinacia oleracea*) belonging to the family Chenopodiaceae, originated in ancient Persia (modern Iran and neighbouring countries) and subsequently introduced to ancient China is a widely grown leafy vegetable often recognized as one of the functional foods for its nutritional, antioxidants and anti-cancer constituents, is a super-cold-hardy vegetable that can be planted in winter and tolerates very low temperatures to some extent.It is higher in iron, calcium and one of the best sources of vitamins A, B, and C than most cultivated greens. rich in mineral Vitamin A and C are present in abundant quantities. Beside this, soft fibrous matter is specially present in providing necessary roughage in diet. Spinach is one of the most common leafy vegetables of tropical and sub tropical regions. The popular Spinach (palak) growing states include Uttar Pradesh,West Bengal, Maharashtra and Gujarat. Though it is not very popular in South India, it is extensively cultivated in Telangana region (Hyderabad, Rangareddy,Medak,Nalgonda districts). This crop is infested by caterpillars (Lepidopterous Pests),green peach aphid, potato aphid, leafminers, spinach crown mite and wireworms resulting in ragged appearance that diminishes the beauty of the garden to wilted or dead plants. Though pesticides are the important tool in integrated pest management, their indiscriminate use causes severe ecological consequences like destruction of natural enemy fauna, effect on non-target organisms, secondary pest outbreaks. Besides, unguided use of pesticides leads to pesticide residues in leafy vegetables and also contaminates the environment which may lead to deleterious impacts not only on human health but also on other biota (Sreelatha and Diwakar, 1997). As consumption of leafy vegetables has been increasing in recent times and peri urban cultivation of leafy vegetables is on the rise, it is essential to know the extent of usage of different pesticides and also evaluate their residues. Keeping all these important issues of concern, the present study of evaluating the spinach samples from different markets for determination of pesticide residues was taken up

Materials and methods

Market study

The spinach samples were collected from three different local vegetable markets viz., Shamshabad, Mehadipatnam and Gudimalkapur in and around Hyderabad and analyzed for residues following the validated QuEChERS method. For the evaluation of pesticide residues, a total of 30 spinach leaf samples were collected from three local markets of Hyderabad, India . Each sample was processed and analyzed for determination of pesticides. Samples were analyzed within 24 hrs.

Sample extraction procedure

Spinach leaf samples were analyzed for pesticide residues following the AOAC official method 2007.01 (QuEChERS) after validation of the method in the laboratory. The samples were collected randomly from 5 locations of the market in polythene bags. Each sample was homogenized separately with robot coupe blixer and homogenized 15 ±0.1g sample was taken in 50 ml centrifuge tube and 30±0.1 ml acetonitrile was added to sample tube. The sample was homogenized at 14000-15000 rpm for 2-3 min using Heidolph silent crusher. 3±0.1 g sodium chloride was added to sample, mixed thoroughly by shaking gently followed by centrifugation for 3 min at 2500-3000 rpm to separate the organic layer. The top organic layer of about 16 ml was taken into the 50 ml centrifuge tube and added with 9±0.1 g anhydrous sodium sulphate to remove the moisture content. 8 ml of extract was taken in to 15 ml tube, containing 0.4±0.01 g PSA sorbent (for dispersive solid phase d-SPE cleanup),1.2±0.01 g anhydrous magnesium sulphate and 0.05 g of GCB(Graphatised Car-

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bon Black).The sample tube was vortexed for 30 sec then followed by centrifugation for 5 min at 2500-3000rpm. The extract of about 1 ml (0.5 g sample) was taken for analysis on LCMS/MS under standard operational conditions.(Table-1).

Results and Discussions:

A multi residue method was used to monitor pesticide residues by LC MS-MS. The targeted pesticides were detected (Table -3) and quantified based on calibration standard at 0.1mg kg^{-1} and of the 30 spinach leaf samples The results revealed that monitoring samples of spinach contained pesticide residues of namely, chlorpyrifos,profenofos, triazophos, deltamethrin+triazophos, cypermethrin, acephate, dimethoate ,imidacloprid ,spinosad A, acetamiprid and indoxacarb (Table.1.) However chlorpyrifos, triazophos, cypermethrin, deltamethrin + triazophos and profenophos were frequently observed in the 3 market samples. In the present study cypermethrin was found in the samples of spinach from local vegetable markets viz.,Shamshabad (3ppm),Mehadipatnam(0.05ppm),Gudimalkapur (0.1ppm) [Table.1] and it is in conformity with work of Sultan *et al*. (1997), Mattern *et al*.(1991), Srivastava *et al*.(2011),Mills *et al*.(2008),Kobayashi *et al*. (2011),Sheikh *et al*.(2013) and Oviedo *et al*.(2003).Chlorpyrifos also found in the spinach samples brought from local vegetable markets viz.,Shamshabad (0.05 ppm),Mehadipatnam (4 ppm) and Gudimalkapur (5 ppm) [Table 1] which is in line with work done by Sheikh *et al*.(2013), Xutianfing *et al*. (2014),Wang liang *et al*. (2008), Chen junjie *et al*. (2011), Srivastava *et al*. (2011), Fansufang *et al*.(2013),Takatoroi *et al*.(2013) and Yawar latif *et al*. (2011). Profenophos was found in the samples of spinach brought from local vegetable markets viz., Shamshabad (0.9ppm), Mehadipatnam (6 ppm) and Gudimalkapur (0.05 ppm) [Table 1] which is in compliance with work of Sheikh *et al*. (2013), Chen junjie *et al*. (2011), Srivastava *et al*.(2011), Tatakori *et al*. (2013), Kobayashi *et al*. (2011) and Zidan *et al*. (2000).Triazophos was found in the samples of spinach, brought from local vegetable markets viz., Shamshabad (6.3 ppm), Mehadipatnam (0.01 ppm) and Gudimalkapur (6.8 ppm) [Table.1] which was in conformity with work done by Chen junjie *et al*. (2011), Srivastava *et al*. (2011),Tatakori *et al*.(2013), Kpbayashi *et al*.(2011) and Srivastava *et al*.(2011).

Table-1 Pesticides detected in the samples of spinach from different markets

Market name	Pesticides detected in samples nach	ppm
Gudimalkapur	Chloropyriphos	5
	Cypermethrin	0.1
	Imidacloprid	0.2
	Profenophos	0.05
	Triazophos	6.8
	Deltamethrin+Triazophhos	0.5
	Acephate	1.2
Mehadipatnam	Deltamethrin+Triazophos	0.06
	Profenophos	6
	Spinosad A	0.4
	Chloropyrifos	4
	Triazophos	0.01
	Cypermethrin	0.05
	Acetamiprid	0.5
Shamsahabad	Triazophos	6.3
	Cypermethrin	3
	Profenophos	0.9
	Deltamethrin+Triazophos	5
	Chloropyrifos	0.05
	Dimethoate	0.9

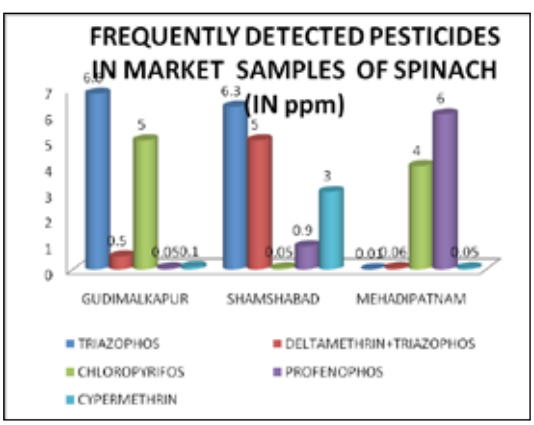

Fig: 1 Pesticides detected from different vegetable markets

References :

1. Chen, J. Y., Lin, Y. J and Kuo, W. C. 2013. Pesticide residue removal from vegetables by ozonation *Journal of Food Engineering*. 114: 404-411.
2. Fan SuFang, Zhang FengZu, Deng KaiLin, Yu ChuanShan, Liu ShaoWen, Zhao PengYue and Pan CanPing. 2013. Spinach or amaranth contains highest residue of metalaxyl, fluazifop-Pbutyl, chlorpyrifos and lambda-cyhalothrin on six leaf vegetables upon open field application. *Journal of Agricultural and Food Chemistry*. 61(9): 2039-2044.
3. Kobayashi, M., Ohtsuka, K., Tamura, Y., Tomizawa, S., Kamijo, K., Iwakoshi, K.,Kageyama, Y.,Nagayama, T and Takano, I. 2011. Survey of pesticide residues in imported frozen vegetables and fruits .*Journal of the Food Hygienic Society of Japan*. 52(2): 121-129. 11.
4. Mattern, G. C.,Liu, C. H., Louis, J. B and Rosen, J. D. 1991. GC/MS and LC/MS determination of 20 pesticide residues for which dietary oncogenic risk has been estimated. *Journal of Agricultural and Food Chem-*

Volume : 6 | Issue : 11 | November 2016 | ISSN - 2249-555X | IF : 3.919 | IC Value : 74.50

istry. 39(4): 700-704.

5. Mills. G.A., Dean, J. R and Pawliszyn, J. 2008. Determination of 23 pesticide residues in leafy vegetables using gas chromatography-ion trap mass spectrometry and analyte protectants. *Journal of Chromatography*, A; 1196/1197: 100-109.
6. Oviedo, M. T. P., Toledo, M. C. de F and Vicente, E. 2003. Pyrethroid pesticide residues in vegetables. [Portuguese]. *Pesticidas - Revista de Ecotoxicologia e MeioAmbiente*. 13:9-18.
7. Sheikh, S. A., Nizamani, S. M., Panhwar, A. A and Mirani, B. N. 2013.Monitoring of pesticide residues in vegetables collected from markets of Sindh, Pakistan. Food Science and Technology Letters. 4(1): 41-45.
8. Sreelatha, N. and Diwakar, B.J. (1997).Impact of imidacloprid seed treatment on insect pest incidence in okra. *Indian Journal of plant Protection, 25* : 52-55.
9. Srivastava, A. K., PurushottamTrivedi, Srivastava, M. K., Lohani, M and Srivastava, L. P. 2011. Monitoring of pesticide residues in market basket samples of vegetable fromLucknow City, India: QuEChERS method. Environmental Monitoring and Assessment. 176(1/4): 465-472.32.
10. Sultan, M. A., Reddy, D. J., Babu, T. R., Reddy and K. N. 1997. Monitoring of pesticide residues from Vegetables in Hyderabad markets. Advances in IPM for horticultural crops. Proceedings of the First National Symposium on Pest Management in Horticultural Crops: environmental-implications and thrusts, Bangalore, India. 250-251.
11. Takatori, S., Yamamoto, H., Fukui, N., Yamaguchi, S., Kitagawa, Y., Kakimoto,Y., Osakada, M.,Okihashi, M., Kajimura, K and Obana, H. 2013.Validation study on a rapid multi-residue method for determination of pesticide residues in vegetables and fruits by LC-MS/MS. Foo Hygiene and Safety Science. 54(3): 237-249.
12. Wang LiGang, Liang YongChao and Jiang Xin. 2008. Analysis of eight organophosphorus pesticide residues in fresh vegetables retailed in agricultural product markets of Nanjing,China. *Bulletin of Environmental Contamination and Toxicology.* 81(4): 377-382.
13. XuTianFeng, PengYanKun, Li YongYu, DhakalSagar and Zhai Chen. 2014. Detection method of chlorpyrifos pesticide residue on spinach surface based on Raman spectroscopy. Journal of Food Safety and Quality. 5(3):707-711.
14. YawarLatif, Sherazi, S. T. H and Bhanger, M. I. 2011. Assessment of pesticide residues in commonly used vegetables in Hyderabad, Pakistan. Ecotoxicology and Environmental Safety, 74(8): 2299-2303.
15. Zidan,Z. H., Mohamed, K. A and Bayoumi, A. E. 2000. Market basket monitoring of pesticides residues on vegetables and fruits from Kalubia governorate, Egypt.. Annals of Agricultural Science (Cairo). 45(2): 743-756.