

**ANALYSIS OF PESTICIDE RESIDUES IN VEGETABLES IN  
VIJAYAPUR, KARNATAKA INDIA****U.S.Pujeri, A.S.Pujar, S.C.Hiremath, K.G.Pujari and M.S.Yadawe\***

S.B. Arts and K.C.P.Science College, Vijayapur, Karnataka India.

Article Received on  
16 May 2015,Revised on 07 June 2015,  
Accepted on 29 June 2015**\*Correspondence for  
Author****Dr. M.S.Yadawe**S.B.Arts and  
K.C.P.Science  
College,Vijayapur,  
Karnataka India.**ABSTRACT**

The aim of this study was to investigate pesticide residues in vegetables in Vijayapur market. A study conducted to analyze 40 residues in locally sold vegetables like cauliflower and cabbage. The vegetable samples of cabbage and cauliflower were collected from local market, Vijayapur, Karnataka. For this purpose, a total of 25 samples of each were analyzed by GCMS/MS. Three (12%) out of 25 samples, contained residues of Chlorpyrifos ethyl (MRL value = 0.05, 0.068, 0.093 and 0.11mg kg<sup>-1</sup>) in cauliflower and two (8%) out of 25 samples, contained residues of Confidor (MRL value = 0.046 and 0.079mg kg<sup>-1</sup>) in cabbage above MRL values. In view of their

potential toxic and persistent nature, there is a pressing need for their control and monitoring in the environment.

**KEYWORDS:** GCMS/MS, Vegetables, MRL, Vegetables, Vijayapur etc.**INTRODUCTION**

Fruits and vegetables are important components of the human diet since they provide essential nutrients that are required for most of the reactions occurring in the body. Like other crops, fruits and vegetables are attacked by pests and diseases during production and storage leading to damages that reduce the quality and the yield. In order to reduce the loss and maintain the quality of fruits and vegetables harvest, pesticides are used together with other pest management techniques during cropping to destroy pests and prevent diseases. The use of pesticides have increased because they have rapid action, decrease toxins produced by food infecting organisms and are less labour intensive than other pest control methods. However, the use of pesticides during production often leads to the presence of pesticide residues in fruits and vegetables after harvest. The presence of pesticide residues is a concern

for consumers because pesticides are known to have potential harmful effects to other non-targeted organisms than pests and diseases. The major concerns are their toxic effects such as interfering with the reproductive systems and fetal development as well as their capacity to cause cancer and asthma.<sup>[1]</sup> Some of the pesticides are persistent and therefore remain in the body causing long term exposure. The concern has led to governments setting up monitoring systems in order to assess the safety situation and make informed decisions when passing legislation.

Pesticides are used worldwide to protect crops before and after harvest in agriculture, gardening, homes and soil treatment. Variety of pesticides is used in current agricultural practice to manage pests and infections that spoil crops.<sup>[2]</sup> A wide range of pesticides (13–14%) are used for the production of fruits and vegetables in India due to heavy pest infestation throughout the cropping season of horticultural crops whereas cropped area is only 3%.<sup>[3]</sup> Pesticides have potentially adverse effects on vegetables, fruits, animal resources and human health.<sup>[4]</sup> Pesticides are widely used in food production to increase food security despite the fact that they can have negative health effects on consumers.

India is an agrarian country. In the year 2000, the vegetable production in India was 92.8 million tones, grown over an area of 6 million hectares, which is about 3% of the gross cropped area of the country. Potato is the most important vegetable crop in India as it occupies 20% of vegetable area and contributes 27% to the total vegetable production. Nevertheless, vegetable production has been diversifying gradually.<sup>[5]</sup> In the world, India occupies first position in the production of cauliflower, brinjal and peas, second in onion and third in cabbage.<sup>[6]</sup> Vegetables form an important component of human diet. They are however, infested by various insect pests like aphids, jassids, diamond moths, caterpillars, etc. Among the vegetables, brinjal, cauliflower, tomato and okra etc. are some very common vegetables cultivated, throughout the country but all are badly affected by insect-pest and diseases. Brinjal (*Solanum melongena*L.) suffers heavily at fruiting stage due to attack of shoot and fruit borers causing 70% damage to the crop and making it totally unfit for human consumption.<sup>[7],[8]</sup> Cauliflower (*Brassica oleracea*) also an important vegetable crop with an annual production of 3.39 million tones is heavily attacked by various insects, resulting in severe loss of quality and production.<sup>[9]</sup> The application of pesticides to agriculture has greatly improved the food production worldwide. India is the second largest producer of vegetables after China, and accounts for 13.4% of world production. Surveys carried out by

institutions spread throughout the country indicate that 50-70% of vegetables are contaminated with insecticide residues. India has a wide variety of climate and soils on which a range of vegetable crops can be grown.

Pesticides are widely used to ensure high crop yields. They are used during production and post-harvest treatment of agricultural commodities.<sup>[10]</sup> However, increased use of chemical pesticides has resulted in contamination of the environment and also caused many associated long term effects on human health.<sup>[11]</sup> The presence of pesticide residues in food commodities has always been a matter of serious concern. The problem is especially serious when these commodities are consumed.<sup>[12]</sup> Pesticides have been associated with a wide spectrum of human health hazards, ranging from short-term impacts such as headaches and nausea to chronic impacts like cancer, reproductive harm and endocrine disruption.<sup>[13]</sup> The heavy use of pesticides may result in environmental problems like disturbance of the natural balance, widespread pest resistance, environmental pollution, hazards to non-target organisms and wildlife, and hazards to humans. Therefore, the objective of present research work was to assess the concentration of such deleterious agrochemicals in vegetables of Vijayapur market and to generate awareness about the lethal effects of these synthetic pesticides on human being.

## **MATERIALS AND METHODS**

### **Sampling**

A total of 25 samples of cauliflower and 25 samples of cabbage were collected from Vijayapur market, when they were ready to sell. After collection, these samples were kept in polythene bags and then transported on ice to the laboratory where they were analyzed immediately or stored at 4°C analysis within 24 hours.<sup>[14]</sup>

### **Sample preparation and clean up**

Samples consisted of 1–2 kg of each vegetable comprising cauliflower and cabbage were collected from Vijayapur market, Karnataka. In the laboratory, samples were packed in plastic bags and kept in refrigerated for analyses. The fresh vegetables samples cut into small pieces and homogenized with a household mill (equipped with stainless steel knives). A 15 g portion of the homogenized sample was weighed into a 50 ml polytetrafluoroethylene (PTFE) tube added 15 ml of acetonitrile containing 1% acetic acid (v/v). Then, 6 g MgSO<sub>4</sub> and 2.5 g sodium acetate trihydrate (equivalent to 1.5 g of anhydrous form) were added, and the sample was shaken forcefully for 4 min and kept in ice bath. The samples were then centrifuged at

4000 rpm for 5 min and 6 ml of the supernatant were transferred to a 15 ml PTFE tube to which 900 mg MgSO<sub>4</sub> and 300 mg PSA were added. The extract was shaken using a vortex mixer for 20 s and centrifuged at 4000 rpm again for 5 min, approximately 2ml of the supernatant were taken in a vials. This extracts were evaporated to dryness under a stream of nitrogen and reconstituted in n-hexane in auto sampler tube for the GC-MS analysis. Standard preparation For preparation of stock solution, standards were dissolved in ethyl acetate and four levels of intermediate standard solution of each pesticide were prepared maintaining the same matrix concentration for the preparation of calibration curve and stored at -4°C in the dark. Working solutions were prepared daily by appropriate dilution with ethyl acetate. Instrumentation GC-MS analysis was performed with a Varian 3800 gas chromatograph with electronic flow control (EFC) and fitted with a Saturn 2200 ion-trap mass spectrometer (Varian Instruments, Sunnyvale, CA, USA). Samples were injected into a Varian 8200 auto sampler SPI / 1079 split / splitless programmed-temperature injector using a 10µl syringe operated in the large volume injection technique. The glass liner was equipped with a plug of carbofrit (Resteck, Bellefonte, PA, USA). A fused-silica untreated capillary column 30m 0.2mm I.D. from Supelco (Bellefonte, PA, USA) was used as a guard column connected to a Rapid-MS [wall-coated open tubular (WCOT) fused-silica CP-Sil 8 CB low bleed of 10m 30.53 mm I.D., 0.25 mm film thickness] analytical column from Varian Instruments (Sunnyvale, CA, USA) for high speed analysis. The mass spectrometer was operated in electron impact (EI) ionization mode. The computer that controlled the system also held a GC-MS library specially created for the target analytes under our experimental conditions. The mass spectrometer was calibrated weekly with perfluoro-tributylamine. Helium (99.999%) at a flow-rate of 1 ml min<sup>-1</sup> was used as carrier and collision gas.

## RESULTS AND DISCUSSION

Pesticides are widely used in agricultural production to prevent or control pests, diseases, weeds, and other plant pathogens in an effort to reduce or eliminate yield losses and maintain high product quality. Cauliflower is widely cultivated in almost all the parts of the world. In India, it is cultivated in about 90 thousand hectares area. There has been a substantial increase in the area of cauliflower (about 20%) during the last two decades. The important cauliflower growing states are Uttar Pradesh, Karnataka, Maharashtra, Bihar, West Bengal, Punjab and Haryana. In the analyzed samples, the detected pesticides comprised of diclorvos, monocrotophos, phorate, parathion, pendamethalin, endosulphan-II, captafol, permethrin and cypermethrin. Current results are in consistent with some earlier reports where reduction (10-

30%) of alphasmethrin residues in tomato and brinjal and cauliflower by Gill *et al.*<sup>[15]</sup> It has been reported that the farmers repeatedly spray different pesticides on cauliflower crop to protect their crop from various pests without any awareness about their hazardous effect on health. It is also routine practice of farmers to send their produce to market on the next day after the application of spray. These results are further confirmed by Khuhro and Nizamani<sup>[16]</sup> who reported that farmers frequently sprayed pesticides 2-3 times in a week and it is their common practice to supply the vegetables to the market only a day after spraying with contamination of residues beyond MRLs. Similar findings were reported by Pujeri *et al.*<sup>[17, 18]</sup> in grapes and pomegranates from our laboratory. Three (12%) out of 25 samples, contained residues of Chlorpyrifos ethyl (MRL value = 0.05, 0.068, 0.093 and 0.11 mg kg<sup>-1</sup>) in cauliflower and two (8%) out of 25 samples, contained residues of Confidor (MRL value = 0.046 and 0.079 mg kg<sup>-1</sup>) in cabbage above MRL values. (Table 1).

**Table.1: Test report of Cauliflower and Cabbage.**

Sr. No.	Name of Pesticide	Limit of Quatification (mg/kg)	Equipment used	Cauliflower Residue Content (mg/Kg)	Cabbage Residue Content (mg/Kg)
1	Aldrin	0.01	GCMS/MS	BLQ	BLQ
2	Cis Chlordane	0.01	GCMS/MS	BLQ	BLQ
3	Chlorothalonil	0.01	GCMS/MS	BLQ	BLQ
4	p p DDE (4 4 DDE)	0.01	GCMS/MS	BLQ	BLQ
5	o p DDD (2 4 DDD)	0.01	GCMS/MS	BLQ	BLQ
6	p p DDD (4 4 DDD)	0.01	GCMS/MS	BLQ	BLQ
7	Dicofol / Kelthane	0.01	GCMS/MS	BLQ	BLQ
8	Dieldrin	0.01	GCMS/MS	BLQ	BLQ
9	Endosulphan Alpha	0.01	GCMS/MS	BLQ	BLQ
10	Endosulphan Beta	0.01	GCMS/MS	BLQ	BLQ
11	Endosulphan Sulphate	0.01	GCMS/MS	BLQ	BLQ
12	Endrin	0.01	GCMS/MS	BLQ	BLQ
13	$\alpha$ - HCH	0.01	GCMS/MS	BLQ	BLQ
14	$\beta$ - HCH	0.01	GCMS/MS	BLQ	BLQ
15	Delta HCH	0.01	GCMS/MS	BLQ	BLQ
16	Heptachlor	0.01	GCMS/MS	BLQ	BLQ
17	Hept- endoepoxide	0.01	GCMS/MS	BLQ	BLQ
18	Hept- exoepoxide	0.01	GCMS/MS	BLQ	BLQ
19	Lindane	0.01	GCMS/MS	BLQ	BLQ

20	Confidor	0.01	GCMS/MS	BLQ	0.046 and 0.079
21	Chlorpyrifos ethyl	0.01	GCMS/MS	0.068,0.093and 0.11	BLQ
22	Dichlorovos	0.01	GCMS/MS	BLQ	BLQ
23	Fenitrothion	0.01	GCMS/MS	BLQ	BLQ
24	Parathion ethyl	0.01	GCMS/MS	BLQ	BLQ
25	Parathion methyl	0.01	GCMS/MS	BLQ	BLQ
26	Propetamphos	0.01	GCMS/MS	BLQ	BLQ
27	Allethrin	0.01	GCMS/MS	BLQ	BLQ
28	Bifenthrin	0.01	GCMS/MS	BLQ	BLQ
29	Etofenprox	0.01	GCMS/MS	BLQ	BLQ
30	Fenpropathrin	0.01	GCMS/MS	BLQ	BLQ
31	Fenvalerate	0.01	GCMS/MS	BLQ	BLQ
32	Esfenvalerate	0.01	GCMS/MS	BLQ	BLQ
33	Lambda Cyhalothrin	0.01	GCMS/MS	BLQ	BLQ
34	Permethrin I	0.01	GCMS/MS	BLQ	BLQ
35	Permethrin II	0.01	GCMS/MS	BLQ	BLQ
36	Tau fluvalinate	0.01	GCMS/MS	BLQ	BLQ
37	Transfluthrin	0.01	GCMS/MS	BLQ	BLQ
38	Iprodione	0.01	GCMS/MS	BLQ	BLQ
39	Trifluralin	0.01	GCMS/MS	BLQ	BLQ
40	Benfluralin	0.01	GCMS/MS	BLQ	BLQ

Among the winter vegetables, cabbage *Brassica oleracea* var. *capitata* Linn. is a popular and extensively cultivated crop because of its nutritional and economical values. It is grown for its edible enlarged terminal buds, which is a rich source of Ca, P, Na, K, S Vitamin A, Vitamin C and dietary fibre. Frequent detection of all pesticides in the super cabbage samples is worrisome as this may have health implications through synergistic tendencies.<sup>[19]</sup> The contamination level of pesticide residues could be considered as a possible public health problem. The results also emphasize the need for regular monitoring of a greater number of samples for pesticide residues, especially sample which has to be exported.

## CONCLUSIONS

Intensive cultivation technologies produce high infestation of crops by some pests and diseases, trigger off major losses of quality crops and initiate the use of more pesticides. It can be concluded that residues of none of the pesticides exceeded their respective maximum residue limits. It can be concluded that most of the samples of the vegetables having pesticide

residue their respective maximum residue limits. Processing (normal and hot water washing) substantially lowers the residues of pesticides in vegetables. The objective of this study was to create awareness among the vegetables consumers who were consuming contaminated vegetables. The present research will not only serve as reference document but also helpful in taking necessary and timely preventive measure to mitigate such problems.

## REFERENCES

1. Gilden, R. C.; Huffling, K. & Sattler B. (2010). Pesticides and Health Risks. JOGNN, 2010; 39: 103–110.
2. H. B. S. Conacher and J. Mes. Assessment of human exposure to chemical contaminants in foods Food Addit. Contam. 1993; 10: 5-15.
3. Agnihotri, N. P. Pesticide Safety Evaluation and Monitoring. Division of Agriculture Chemicals, Indian Agricultural Research Institute, New Delhi., 1999; 14.
4. Perez Bendito, D. and Rubio, S. In: Environmental Analytical Chemistry. (Volume XXXII Comprehensive Analytical Chemistry): Elsevier: Amsterdam., 1999.
5. Kumar, S., Pal, S., Joshi, P. K. Impact of Vegetable Research in India. NCAP Publication., 2004; 9-33.
6. Pandey, U. B. Development programmes and policies for accelerated production of vegetables in India. In Food Security and Vegetables – A Global Perspective. Eds: Premnath, P., 2004.
7. Misra, P. N., Singh, M. P. Chemical control of okra in the Terai region Uttar Pradesh. Indian J. Ent, 1996; 45(2): 152-158.
8. Duara, B., Baruah, A., Deka, S. C., Burman, N. Residues of cypermethrin and fenlinate on brinjal. Pesticide Res. J., 2003; 15(1): 43-46.
9. Regupathy, A., Habeebullah, B., Balasubramaniam, M. Dissipation of insecticides applied to control *Plutella maculipennis* Curtis and *Spodoptera litura* in cauliflower. Pesticide Res. J., 1985; 19(9): 53-56.
10. Levitt JA, Wehr HM. Food and Drug Law Journal., 2001; 56: 1-9.
11. Bhanti M, Taneja A. Chemosphere., 2007; 4: 69-70.
12. Solecki R, Davies L, Dellarco V, et al. Food and Chemical Toxicology., 2005; 43: 1569-1593.
13. Berrada H, Fernández M, Ruiz MJ, et al. Food Control., 2010; 21: 36-44.

14. Islam, S., M.S. Hossain, N. Nahar, M. Mosihuzzaman M.I.R. Mamun, Application of high Performance Liquid Chromatography to the analysis of pesticide residues in egg plants. *J. Applied Sci.*, 2009; 9(5): 973-977.
15. Gill, Kanta, Kumari, Beena and Kathpal T.S. Dissipation of alphasmethrin residues in/on brinjal and tomato during storage and processing conditions. *J. Food Sci. Technol.*, 2001; (38)1: 43-46.
16. Khuhro, R.D. and Nizamani, S.M. Monitoring pesticide residues in fruits and vegetables. Report submitted to EPA, Karachi, 2000; 51pp.
17. U.S.Pujeri,A.S.Pujar,S.C.Hiremath and M.S.Yadawe,Multiresidue analysis of pesticides in grapes in Bijapur district. *IJABPT*, 2010; 1(2).
18. U.S.Pujeri,A.S.Pujar,S.C.Hiremath and M.S.Yadawe, Monitoring of Pesticide Residues in Pomegranate of Bijapur District Karnataka, 2010; 2(11).
19. Farag, AT., El Okazy, AM., El Aswed AF. Developmental toxicity study of chlorpyrifos in rats. *Reprod. Toxicol.*, 2003; 17(2): 203-208.