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STATUS OF PESTICIDE RESIDUE IN GRAPES OF BIJAPUR (KARNATAKA)

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Abstract

Grapes grown in Bijapur district (Karnataka) were analysed for pesticide residue content employing multiresidue analysis by gas liquid chromatography equipped with ECD and NPD detectors. All the fruit samples showed the presence of one or the other group of pesticides. Out of 8 samples analyzed only two samples showed pesticide residue content below the European MRL. The detected pesticides are Chlorothalonil, Chloropyriphos, Monocrotophos, Triazophos, Cypermethrin, Lamda Cyhalothrin, Matalaxyl, Flusilazole, Hexaconazole, Myclobutanil, Penconazole, Propiconazole, Triadimenol, Difenoconazole, Carbendazim, Azoxystrobin. On the bases of these studies it is suggested that pesticide residue monitoring should be extended to the grape grown for the supply in domestic market similar to the export quality, which may serve as basis for the future policy in chemical use.

Keywords: Grape, Gas Liquid chromatography (GLC), Maximum Residue Limit (MRL), Pesticides

Introduction

Grape is an important commercial horticulture crop cultivated in 9000 ha of Bijapur District of Karnataka (India). A wide range of pesticides are used for the better yield of grape due to pest infestation throughout the season of the crop. Pesticides have potential adverse effects on vegetables, fruits, animal resources and human health (1). Because of their wide spread use, their toxic residue have been reported in various environmental matrices (2, 3, 4, 5). Thus the determination of pesticide residue in foods and other environmental components/ commodities like water, soil, fruits vegetables and total diet has become an essential requirement for the consumers, producers and authorities for food quality control.

Various studies have been done towards the beneficial properties of fruit juice because they have several components such as phenols, vitamins and flavonoids with antioxidant effect. However, fruit juice can also contain residue of pesticide used as standard pest control method in crops. Many of the pesticides are degraded through oxidative mechanism and their persistence in juice can be enhanced by antioxidants (6). However, injudicious and indiscriminate use of these highly persistent and toxic chemicals in horticulture, caused serious contamination in fruits like grape.

The farmers, authorities and exporters are taking care to maintain pesticide residue in the grape below minimum residue limit (MRL) put by the receiving country by following the precised techniques, proper time period and applying optimum quantity of degradable pesticide etc. during the production of export quality grapes. But most of the farmers who are interested to sell the grapes in domestic market, bother only about yield of the fruit and economy of the farming and apply the pesticide injudiciously without bothering about MRL. Indiscriminate use of pesticides particularly in fruiting stage and non adoption of safe waiting period leads to accumulation of pesticide residue in consumable fruits and vegetables. Contamination of pesticide residue in vegetables has been reported by several researchers (4, 7). There is no intensive monitoring body to control the pesticide residue limit in the grape of every farm in India. Aim of the study was to evaluate the residue of different chemical group of pesticide: Organochlorines (OC) Synthetic Pyrethriods (SP), Organo-Phosphorous (OP) and Carbamate in grape of the Bijapur district. It is hoped that data will produce awareness among the grape farmers and consumers and establish a base line in determining changes in the residue level of different pesticides in different fruits in future.

Materials and Methods

Eight grape samples were collected from different grape farms of Bijapur district which were ready to harvest and sale, with recommended procedures. Homogenized 2kg samples from each farm were sealed in polythene bag and kept in refrigerator till analysis.

Sample extraction and clean up

The extraction of the samples and subsequent clean-up of the extracts were in accordance with the method used by Kumari et al (8) for vegetables, which consists of homogenization, extraction of homogenates, liquid-liquid partitioning with ethyl acetate and clean up

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by column chromatography with eluting solvent ethylacetate:hexane (3:7 v/v) for organo chlorines and synthetic pyrethroids and acetone: hexane (3:7 v/v) for organophosphates.

Estimation

Analysis were carried out using gas liquid chromatography (GLC) on model 5890A Hewlett-Packard (HP) equipped with ^{17}Ni electron capture detector, capillary column SPB-5(30mx0.32mm i.d.x0.25µm film thickness) of diphenyl 59% dimethyl siloxane for OC and SP. Column temperature: 150°C initially for 5 min. then programmed at 8°C/min upto 190°C for 2 min. and then at 15°C/min upto 280°C for 10 min.; Injector temperature 280°C and detector temperature 300°C, nitrogen flow rate : 2ml/min through column and make up 60ml/min with split ratio of 1:10.

For the analysis of OP insecticide, HP gas chromatography equipped with nitrogen phosphorous detector with mega bore column HP-1 (10m x 0.53mm i.d. x 2.65µm film thickness) of polysiloxane was used. Oven-Column temperature:100°C initially for 1 min then increased at a rate of 10°C/min to 200°C and was finally increased at the rate of 20°C/min to 260°C; gas flows H_2 1.5ml, N_2 : 18ml and O_2:135ml/min.

In order to ensure the quality assurance information, before taking up analysis of test sample, the analytical method was standardized by processing spiked samples in triplicate of each fruits separately at different fortification levels ranging from 0.01 to 0.50µg/g. Control samples were processed along the spiked ones. GC-MS spectra of standard and sample-1 only are shown in Fig.1 and Fig.2 respectively.

Fig. 1: GC-MS spectra of pesticides Pesticide of standard

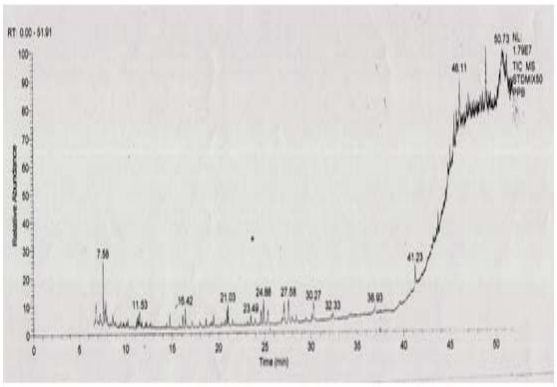

Fig.2: GC-MS Spectra of Pesticides Pesticide of Sample-1

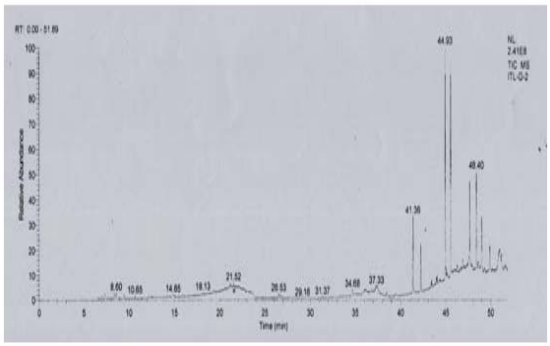

Table-1 Pesticide Residue in Grape of Bijapur District of Karnataka

	Sample No.→ Pesticide↓	LOQ	EU MRL	1	2	3	4	5	6	7	8
1	Chlorothalonil	0.01	0.01	*BLQ*	0.01	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*
2	Chloropyriphos	0.01	0.05	0.02	*BLQ*	0.02	*BLQ*	*BLQ*	*BLQ*	*BLQ*	0.02
3	Monocrotophos	0.01	0.01	0.04	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	0.03
4	Triazophos	0.01	0.01	0.06	*BLQ*	0.01	*BLQ*	*BLQ*	*BLQ*	*BLQ*	0.06
5	Cypermethrin	0.01	0.05	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*
6	λ-Cyhalothrin	0.01	0.02	*BLQ*	*BLQ*	0.02	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*
7	Matalaxyl	0.01	0.05	0.14	*BLQ*	0.07	*BLQ*	*BLQ*	*BLQ*	*BLQ*	0.10
8	Flusilazole	0.01	0.02	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*
9	Hexaconazole	0.01	0.02	0.05	*BLQ*	*BLQ*	*BLQ*	*BLQ*	0.01	0.02	0.05
10	Myclobutanil	0.01	0.02	0.12	0.02	*BLQ*	0.01	0.01	0.02	0.07	0.10
11	Penconazole	0.01	0.05	0.02	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	0.03
12	Propiconazole	0.01	0.05	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	*BLQ*	0.01	*BLQ*
13	Triadimenol	0.01	0.10	*BLQ*	*BLQ*	*BLQ*	0.01	0.03	*BLQ*	*BLQ*	*BLQ*
14	Difenoconazole	0.01	0.10	*BLQ*	*BLQ*	*BLQ*	*BLQ*	0.04	0.03	0.05	*BLQ*
15	Carbendazim	0.01	0.10	0.10	0.05	0.25	0.01	*BLQ*	*BLQ*	*BLQ*	0.10
16	Azoxystrobin	0.01	0.05	*BLQ*	*BLQ*	*BLQ*	*BLQ*	0.02	0.05	0.11	*BLQ*

LOQ: Limit of Quantification in mg/kg
BLQ: Below the Limit of Quantification in mg/kg
EU-MRL: European Maximum Residue Limit in mg/kg

Results and Discussion

Analysis of 8 grape sample for the residue of 94 pesticides, showed the presence of 16 pesticides and the results are shown in the Table-1. It reveals that all the samples contain one or the other pesticide. Organochlorines (OC): *Chlorothalonil*; Organo-phosphorous(OP): *Chloropyriphos, Monocrotophos, Triazophos;* Synhtetic pyrethroids: *Cypermethrin, Lamda cyhalothrin*; Triazoles: *Flusilazole, Hexaconazole, Myclobutanil, Penconazole, Propiconazole, Triadimenol, Difenoconazole*, Benzimidazole: *Carbendazim*; and others: *Azoxystrobi, Matalaxyl* were detected and rest of the pesticides were found to be below the limit quantification (BLQ).

Out of 8 samples analysed, Sample-1 & 8 contain *Chloropyriphos, Myclobutanil, Carbendazim and Penconazole* less than EU-MRL and *Monocrotophos, Triazophos, Matalaxyl and Hexaconazole* more than EU-MRL. Sample-2 and 4 contain *Myclobutanil, Triadimenol and Carbendazim* below EU-MRL. Sample-3 contains *Chloropyriphos, Triazophos and Lamda cyhalothrin below European Minimum Residue Limit (EU-MRL)* and *Carbendazim Matalaxyl above EU-MRL*. Sample-5 Showed the presence of *Myclobutanil and Azoxystrobin* below EU-MRL and *Difenoconazole* and *Azoxystrobin* above EU-MRL. Sample-6 contains *Hexaconazole, Azoxystrobin and Myclobutanil* below EU-MRL and *Difenoconazole* above EU-MRL. Sample-7 contains *Hexaconazole Propiconazole and Difenoconazole below EU-MRL while Myclobutanil and Azoxystrobin* above EU-MRL.

Sample-2 & 4 were found to be safe for consumption while rest are not according to European standards. The OC detected in one of the samples although their use has been banned or restricted during last decade, may be attributed to carry over of residue from previous use which dominated in use, in Indian agriculture during eighties and nineties (9). Presence of OP and SP residue in the samples is an indicative of change in usage of pattern of pesticide in India where shift has been taken place from OC to easily degradable group of these insecticide in last decade.

Conclusion

Most of the grapes available in Karnataka market contain one or the other pesticide. Epidemiology studies on pesticide have found association with hematological cancer, neurotoxin effects, neurobehavioral disorders, reproductive problems including birth defects and infertility, newborn deaths etc (10). On the bases of these studies it is suggested that pesticide residue monitoring should be extended to the grape grown for the domestic market supply also similar to the export quality, which may serve as basis for the future policy in chemical use.

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