

Analysis of Pesticide Residues in Winter Fruits

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ABSTRACT

Fruit samples of winter fruits (apple, grapes, banana cheeku, papaya, lemon) for pesticide residues employing a multiresidue analysis by gas liquid chromatography. All the fruit samples showed the presence of residues with one or other group of pesticides. Some samples exceeded the quantification limit. The increasing interest in the pesticides in fruit samples is justified from the ecological point of view. In this paper pesticide mobility on fruit samples was studied. Out of nine pesticides tested for most of the sample show very high levels of malathion, while other pesticides residues are within the established tolerance, BHC, endosulphan, dieldrin are within limits. Thus consumer intake of pesticides from fruit samples studied in this work should be reduced by washing fruits with water. In this paper multiresidue determination of pesticides are discussed using GLC, HNMR, IR.

Key words: Pesticides, GLC, HNMR, IR, fruits, residues.

INTRODUCTION

A pesticide is a chemical substance used for preventing, destroying, repelling or mitigating a pest, which can be an insect; rodent, bird, weed or fungus, as well as micro organism like bacteria and viruses. Pesticides can be broadly classified as insecticides, herbicides, fungicides, rodenticides, and antimicrobials, with many subclasses. The major insecticide groups are the organochlorines, organophosphates, carbonates and pyrethroids. Pesticides are considered hazardous chemicals and improved the regulation of pesticides particularly in developed countries, a health risk remains. Both the potency of primary factor affecting the level of risk.

The use of pesticides provided an important socioeconomic benefit to the areas of agriculture and food productions. Pesticide production is market driven with high investment in industrialized countries. In the U.S, 77% of all pesticides are used. In developing countries, public health programs represent an important use of pesticides in the control of vector borne diseases like malaria. Countries in Africa, Asia and central

South America are highly dependent on pesticides. Other areas in which pesticides are used include forestry, gardening and lawn care horticulture and livestock and to a large extent domestic use in home. In the U.S pesticides are used in around 70 million homes. Usage of OCP's have been prohibited in most of countries, but 70% of banned pesticides low cost, in India DDT was banned for use in agriculture in 1985, but still 7500 metric tons per year is used here.

The problems of pesticides residues in crops has been attracting growing attention the use of organic insecticides for the control of insect on crops has become common during the past few years.

The detection and identification of pesticides in our environment is a problem of increasing public interest.

Pesticides residues in food has become a consumer safety issue. The consumer has a right to know how much pesticide gets incorporated in the food he eats. At many laboratories expanded research programmes have been instituted to

understand and control more fully the varied effects of pesticides like:-

- 1) The appraisal of the potential carcinogenicity of ingested substances.
- 2) Palatability and organoleptre evaluation of fruits, meats and vegetables.
- 3) Assimilation of detailed data on acute and chronic toxicity for all compounds.
- 4) Nature of plant surfaces and the chemical modes of penetration subsequent

translocation, distribution and metabolic fate in plants and exudation of regulating compounds into the soil.

- 5) Establishment of safety threshold levels within a human being without immediate or future harm. The short as well as long term impact of the use of pesticides on biological systems is being evaluated continuously in an effort to minimize.

Table 1

Pesticide	Chemical Name	Molwt.	Trale Name	Chemical Class	ADI mg/Kg/Day
BHC $\alpha, \beta, \gamma, \delta$	1,2,3,4,5,6-	290.85	HCH, Grammexane	Organochlorine	0.008
DDT	Hexachlorocy clohexane 1,1-(2,2,2-trichloro ethylidene) bis [4-chlorobenzene]	354.41	Anofex, Cesarex, Digmar, Gezarex	Organochlorine	0.02
Methyl Parathion	0,0-dimethye 0-4-Nitrophenyl Phasphorothioate	263.21	Matafos, metacide, dalf, Gearphas	Organophosphate	0.02
Malathion	Diethyl (Dimethoxy Thiophas Phosphorylthio succolnate	330.36	Carbophos, Mercaptothion	Organophosphate meldison	0.02
Dimethoate	0,0 dimethye S-methylcarbo mouylemethyl phosphoridithiote	229-28	Cygon400, Demos, Dicap, rogor	Organophosphate	0.01
Ethion	0,0,0, ¹ 0 ¹ -tetraethyl, s-s ¹ methyloe bis (phosphorodithioate)	384.48	Acithion, Ethanox, Hylmox	Organophosphate	.002
Endsulfan	6,7,8,9,10,10-hexachloro 1,5a,6,9,9a-hexahydro 6,9-methano-2,4,3 benzadioxathiepin 3-oxide	406.96	Hexasulfan, Afidan, Cyclodan Beosit	Organochlorine	.006
Dieldrin	1,2,3,4,10.10-Hexachloro 6,7 epoxy 1,4,-4a,5,6,7,8,8a, octahydro-1,4,5,8-dimetanophthalene	380.9	Dieldriti, Octalox Panoram D-31	Organochlorine Dieldrex,	

Potential or latent hazards while maximizing the benefits derived by marking form increased agricultural production and communicable disease eradication.

The use of pesticides has not permitted the control of diseases transmitted by insects but also has led to increased food production and better health.

EXPERIMENTAL

Selection of fruit samples were based on their availability in winter. The samples were purchased in Jhansi. The fruits sold here are bought from the near bus stand in Jhansi. The fruit samples was analyzed in the form, that is offered to the consumer. For example apple, Cheeku, Papaya and grapes were analyzed with peels whereas banana, pomegranate and coconut were analyzed without peels. Lemon was analyzed with peels as it is used in making pickles in the form each sample size taken 1 kg out of which a representative substance weighting 20gram was randomly taken and the pesticides were extracted for 8-10 hr at the rate (4-5) cycles per hr, in hexane in a soxhelt extractors. The rotary evaporators. The concentrate contained aqueous as well as organic residue.

The organic part was extracted in hexane with the help of a separating funnel and a pinch of

sodium sulphate was added to it. The solution thus obtained was filtered and concentrated again. To this 5 ml of hexane was added and the sample thus prepared was analyzed for the presence of 9 pesticides by gas chromatograph (Perkin Elmer-Auto system XL) with the selective electron-capture detector (ECD). This detector allows the detection of contaminants at trace level concentration in the lower ppm range in the presence of multitude of compounds extracted from the matrix to which these detectors do not resend. The column used was PE-17, length 30m. ID 0.25 film 0.25 mm with a 2 ml/min flow. The carrier gas and the make up gas was nitrogen employing the splitting mode. The oven temperature was kept at 190-280°C with a ramp of 5°C/min. The lam plies were calibrated (retention time, are a count) against a 10 ppm standard mixed solution of all 9 pesticides. Each peak is characterized by its retention time and the response factor in ECD. Sample results were quantitated in ppm automatically by the GC software.

One GC injection (30 min) was required in order to cover all 9 pesticides included in a analysis. Hamilton micro syringes injection of the pesticide dissolved in hexane as solvent were made directly onto the coated silanized column solid support, there by eliminating the possibility of catalytic degradation by metallic surfaces. Pesticides were identified according to their retention time. For accurate result the concentration

Table 2: Pesticide residues, in water fruits mg/Kg.

Sample	α BHC	β and γ BHC	dimetha noate	δ BHC	Methyl Parathion	Malathion	Endsuffan	DDT	Dieldrin
Apple	-	0.05	-	-	-	2.46	-	-	-
Pomegranate	-	0.01	-	-	-	1.70	-	-	-
-									
Black grapes	0.02	0.03	-	0.02	-	2.37		0.02	
Green Grapes	-	-	0.01	-	-	3.05	0.10	-	
0.01									
Banana	-	-	-	-	-	1.03			
Papaya	0.01	-	0.02	-	-	4.19			
0.01									
Cheeku	-	-	0.01		-	4.34		0.01	
Coconut			0.03		-	3.25			
Lemon					-	5.66		0.31	0.01

of the standard was kept same. The multiresidue method which can detect all 9 pesticides in one analytical run was preferred. This method is characterized by a broad scope of application good recoveries and sensitivity and low solvent consumption, coupled with good analytical quality control. The presence of malathion, DDE in the respective sample were further confirmed by HNMR (Joel, 400 MHz) and IR (Bruker) Spectral studies. HNMR and IR spectra of the standard pesticide was taken separately and compared with that of the sample containing those particular pesticides.

The study including the following parameters

Fruit sample, place of origin, Methods used and Pesticides tested for Table 1 and concentrations found for each pesticides table 2.

Where the No. of Pesticides-

- 1) a BHC
- 2) g and b BHC
- 3) Dimethanoate
- 4) Methyl Parathion
- 5) Malathion
- 6) Endosulphama
- 7) DDE
- 8) Dieldrin

- 9) Ethion
- 10) DDT

CONCLUSION

The high levels of malathion is alarming. We have analyzed for only 9 pesticides where as the presence of many others can not be ignored. Out of necessity the field of residue analytical chemistry has emerged as a devoted specifically to the determination of sub microgram concentration levels of pesticides to confirm the tolerance established by law for pesticides in or on agricultural forage and food crops and animal products. The area associated with the nature, persistence and concentration level of pesticides residues on produce need to be critically examined by academic, industrial and government agencies to ensure man's future well being.

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