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Full Length Research Paper

Evaluation of Pesticide Residues in Spice Crop on Ginger (*Zingiber officinale rosc*)

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Abstract

Evaluation of organic and inorganic farming on Ginger crop. Pesticides were determined by Gas chromatography with electron impact mass spectrometric detection in the selected ion monitoring mode. Spiked blank samples were used as standards to counteract the matrix effect observed in the chromatographic determination. Pesticides were confirmed by their retention times. Applying organic manure and chemical fertilizers, the study reveals that low pesticide residue was recorded in the organic treated plot when supplied with Vermicompost + Farm yard manure (FYM) and the high pesticide residue was recorded inorganic farming treated plot when supplied with FYM + RDF (Recommended Dose of Fertilizers) Nitrogen, phosphorus and potassium where as minimum residue was recorded in organic plot Dieldrin calculated amount in units (11719.162 PPB), Phosphamidon (1034.809 PPB). Thus it was concluded that organic treated plot shows low pesticide residue. The proposed method was used to determine pesticide levels in samples, taken from organic and inorganic plot, where several herbicides and insecticides were found.

Keywords: *Ginger, Pesticide residue, Gas chromatography, Vermicompost, NPK.*

Introduction

Pesticides are the chemicals that are used in agriculture for the control of pests, weeds or plant diseases. Pesticide is the general term for insecticides, fungicides and similarly active compounds. FAO defined a pesticide as any substance or mixture of substances intended for preventing, destroying or controlling any pest, including vectors of man and animals causing harm or otherwise interfering with the production, processing storage, transport or marketing of food, agricultural commodities. In today's era, heavy doses of chemical fertilizers and pesticides are being used by the farmers to get a better yield of various field crops. These chemical fertilizers and pesticides decreased soil fertility and caused health problems to the consumers. Due to adverse effects of chemical fertilizers, interest has been stimulated for the use of organic manures. Fertilizers play a major role in agricultural output. The huge amount of chemical fertilizers and pesticides has made depletion of soil microorganisms and create environmental pollution which affects human health and environment (Nath *et al*., 2009). In addition, the runoff of fertilizers into streams, lakes, and other surface waters, they can induce the growth of algae, leading to the death of other aquatic animals and eutrophication. Such events are recorded from many studies and revealed by several reviews (Relyea, 2005).

India is the leading country in the world for production, consumption and export of spices. Ginger is an important spice crop grown in India. It is herbaceous rhizomatous perennial plant belonging to the family *Zingiberaceae.* It is a tropical plant believed to have originated in South East Asia probably India or China. In India, ginger is cultivated in an area of 83,940 ha with annual production of 3.06 lakh tonnes (Anon., 2002). India is also largest exporter of ginger, accounting to 6580 tonnes of dried ginger valued at Rs. 22.95 crores (Anon., 2002). In Karnataka, it is grown in an area of about 8,421 ha with an annual production of 62610 tonnes (Anon., 2001). Organic Agriculture should sustain and enhance the health of soil, plant, animal, human and plant as one and indivisible. Organic Agriculture should be based on living ecological systems and cycles, work with them, emulate them and help sustain them. Organic Agriculture should build on relationships that ensure fairness with regard to the common environment and life opportunities. Organic Agriculture should be managed in a precautionary and responsible manner to protect the health and well-being of current and future generations and the environment.

Materials and Methods

Methods of Pesticide Residue Analysis

Sample Collection

Rhizome samples were collected and brought to the laboratory. Samples maintained in refrigerator for further process. Taking 20-25gms of samples crushed in mortar and pestle make it in to fine powder.

Diacid Method: (Tandon 1993): Taken 25gm of sample in 100ml of conical flask and adding 20ml of diacid mixture. Digested 10-15 minutes till the white ash colour up to the end product is obtained. Filter through the volumetric flask of 50 ml and make up to 100ml.

Stock solutions: (500 μg/mL) of each pesticide standard were prepared by dissolving 0.050 g of the pesticide in 100 mL of ethyl acetate and stored at 4 °C.

A pesticide intermediate standard solution (5 μg/mL) was prepared by transferring 1 mL from each pesticide solution to a 100 mLVolumetric flask and diluting to volume with ethyl acetate to obtain a concentration of 5 μg/mL. A set of calibration standard solutions of 5.0, 1.0, and 0.5 μg/mL was prepared by dilution. The solutions containing 5.0, 1.0, and 0.5 μg/mL of each pesticide were used to Ginger samples. The internal standards were prepared by dissolving lindane and hexazinone in ethyl acetate to make a 500 μg/mL solution.

Apparatus Extraction Equipment : Polypropylene columns (20mL) of 10 cm - 20 mm Whatman no.1 filter paper circles of 2 cm diameter (Whatman filter paper were used. One-way stopcocks were employed to close the columns. An ultrasonic water bath was used in the extraction step. The generator of this ultrasonic bath has an output of 150 W and a frequency of 35 kHz. A vacuum manifold was employed to remove the extraction solvent.

GC-MS Analysis: GC-MS analysis was performed with a Gas chromatograph. A fused silica capillary column (ZB-5MS), 5% phenyl polysiloxane as nonpolar stationary phase (30 m - 0.25 mm) and 0.25μm film thickness, supplied by Phenomenex (Torrance, CA), was employed. Operating conditions were as follows: injector port temperature, 280C; helium as carrier gas at a flow rate of 1.0 mL/min; pulsed splitless mode (pulsed pressure 45 psi) 310 kPa for 1.5 min). The column temperature was maintained at 70C for 2 min and then programmed at 25C/min to 150C, increased to 200 C at a rate of 3C/min followed by a final ramp to 280C at a rate of 8C/min, and held for 10 min. The total analysis time was 41.87 min and the equilibration time 2 min. A 2 filter paper µL volume was injected splitless, with the split valve closed for 1 min. The mass spectrometric detector (MSD) was operated in electron impact ionization mode with an ionizing energy of 70 eV, scanning from *m/z* 60 to 500 at 3.62 s per scan. The ion source temperature was 230 C and the quadrupole temperature 150C. The electron multiplier voltage (EM voltage) was maintained 1000 V above autotune, and a solvent delay of 5 min was employed.

Analysis was performed with selected ion monitoring (SIM) using one target and one or two qualifier ions. The target and qualifier abundances were determined by injection of individual pesticide standards under the same chromatographic conditions using full scan with the mass/charge ratio ranging from *m/z* 60 to 500 Quantification was based on the peak area ratio of the target ion divided by the peak area of the internal standard in samples versus those found in the calibration standard. Standards were prepared in blank matrix extracts, to counteract the matrix effect. Blank matrix extracts were made following the procedure for sample preparation described below, using a blank soil sample without pesticide fortification.

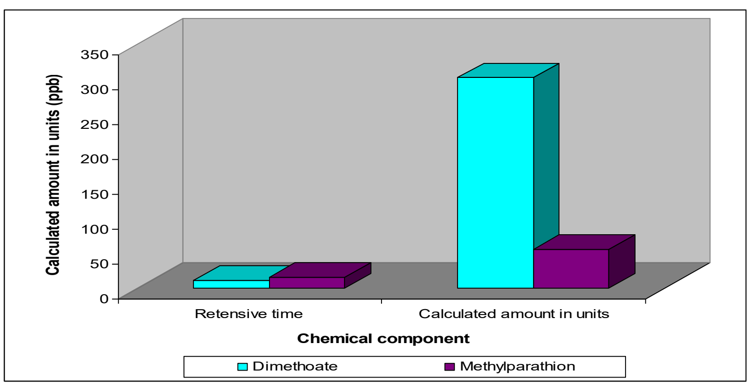
Procedure: Two filter paper circles were placed at the end of a plastic column, and anhydrous sodium sulfate (2 g) was added; sieved samples of Ginger (5g) was then placed in the column. In the recovery assays, samples were previously fortified with 0.5 ml of a mixture of the different pesticides to reach final concentrations of 0.05, 0.1, and 0.2 μg /g, allowing 20 min for solvent evaporation Ginger samples were extracted with 4 mL of ethyl acetate for 15 min in an ultrasonic water bath at room temperature. Ethyl acetate was selected as extraction solvent due to the good results obtained in previous works (*9-11*, *15*). The water level in the bath was adjusted to equal the extraction solvent level inside the columns, which were supported upright in a tube rack and closed with one-way stopcocks. After extraction, the columns were placed on the multiport vacuum manifold, where the solvent was filtered and collected in graduated tubes. Samples were extracted again with another 4 mL of ethyl acetate (15 min). The extracting solvent was filtered, and samples were washed with 1 mL of additional solvent. The total extracts collected in 10 mL graduated tubes were concentrated with a gentle stream of air to an appropriate volume (10 mL for the highest and intermediate levels and 5 mL for the lowest level and real samples) and stored at 4°C until analyzed by GC-MS. A 0.5 mL of the internal standard solution of 1 μg/mL (lindane and hexazinone) was added before GC analysis. Chromatographic standards were prepared using blank sample extracts. These blank extracts were fortified with 0.5 mL of the pesticide standard solution of 1 μg/mL and with 0.5 mL of the internal standard solution (lindane and hexazinone) of 1 μg/mL.

Table 1: Pesticide residue values detected in organic rhizome plot at harvest.

S.NO	Chemical component Name	Retentive time	Calculated amount in units	Obtained results
1	a-Lindane	N/A	N/A	Non Detected
2	Dieldrin	25.30	11719.162 PPB	Detected
3	Alachlor	N/A	N/A PPB	Non Detected
4	Endosulfansulfate	N/A	N/A PPB	Non Detected
5	Dimethoate	N/A	N/A PPB	Non Detected
6	Op-DDT	N/A	N/A PPB	Non Detected
7	Chlorpyriphos	N/A	N/A PPB	Non Detected
8	op-DDD	N/A	N/A PPB	Non Detected
9	Metalaxyl	N/A	N/A PPB	Non Detected
10	Op-DDE	N/A	N/A PPB	Non Detected
11	Atrazine	N/A	N/A PPB	Non Detected
12	a-Endosulfan	N/A	N/A PPB	Non Detected
13	Malathion	N/A	N/A PPB	Non Detected
14	Moncrotophos	N/A	N/A PPB	Non Detected
15	Phosphamidon	15.00	1034.809	Detected
16	Aldrin	N/A	N/A PPB	Non Detected
17	Butachlor	N/A	N/A PPB	Non Detected
18	Phorate	N/A	N/A PPB	Non Detected
19	Methylparathion	N/A	N/A PPB	Non Detected
20	b-Endosulfan	N/A	N/A PPB	Non Detected
21	Ethion	N/A	N/A PPB	Non Detected
22	g-Lindane	N/A	N/A PPB	Non Detected
23	b-Lindane	N/A	N/A PPB	Non Detected
24	Edifenphos	N/A	N/A PPB	Non Detected

* N/A – Not available, * PPB – Parts per billion

S.NO	Name of Chemical component	Retensive time	Calculated amount in units
1	Dimethoate	10.99	301.822
2	Methylparathion	15.39	55.599



Results and Discussion

The data presented in **Table1** lists the pesticides along with their retention times, In the organically grown crops pesticide residue was analysed out of twenty four chemical components was analyzed. Deldrin 25.3% with the calculated amount of 11719.16 PPB was found which shows lower concentration and Phosphamidon Retention time was 15% with the calculated amount of 1034.81 PPB was record. This indicates clearly the organically grown crops were nearly less pesticide residues which resulted in good quality produce in organic Ginger crop plot.

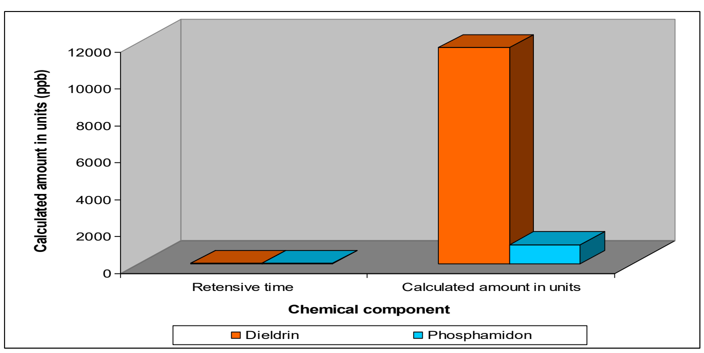
The data presented in **Table2** Pesticides were confirmed by their retention times. Inorganic plot showed a maximum number of chemicals pesticide.

Table 2: Pesticide residue values detected in Inorganic rhizome plot at harvest.

S.NO	Chemical component Name	Retentive time	Calculated amount in units	Residue results
1	a-Lindane	N/A	N/A PPB	Non Detected
2	Dieldrin	25.18	2279.809 PPB	Detected
3	Alachlor	N/A	N/A PPB	Non Detected
4	Endosulfansulfate	31.92	6.852 PPB	Detected
5	Dimethoate	10.71	17.410 PPB	Detected
6	Op-DDT	29.32	12.247 PPB	Detected
7	Chlorpyrifos	18.73	2.636 PPB	Detected
8	op-DDD	26.09	1.408 PPB	Detected
9	Metalaxyl	N/A	N/A PPB	Non Detected
10	Op-DDE	22.97	3.406 PPB	Detected
11	Atrazine	11.21	49.688 PPB	Detected
12	a-Endosulfan	N/A	N/A PPB	Non Detected
13	Malathion	17.99	63.581 PPB	Detected
14	Moncrotophos	N/A	N/A PPB	Non Detected
15	Phosphamidon	N/A	N/A PPB	Non Detected
16	Aldrin	17.84	4.608 PPB	Detected
17	Butachlor	N/A	N/A PPB	Non Detected
18	Phorate	N/A	N/A PPB	Non Detected
19	Methylparathion	15.39	55.599 PPB	Detected
20	b-Endosulfan	N/A	N/A PPB	Non Detected
21	Ethion	29.96	4.301 PPB	Detected
22	a-BHC	N/A	N/A PPB	Non Detected
23	b-BHC	N/A	N/A PPB	Non Detected
24	Edifenphos	N/A	N/A PPB	Non Detected

** N/A – Not available, * PPB – Parts per billion*

S.NO	Chemical component Name	Retensive time	Calculated amount in units
1	Dieldrin	25.30	11719.162
2	Phosphamidon	15.00	1034.809



In inorganically grown Ginger crop the retention time which was tested for 24 toxic chemicals only BHC, Alachlor, Metalaxyl, Monocrotophos, Butachlor, Phorate, Endosulfan, Phosphomidon and Edifenphos were not available however rest of the chemicals were present in the produce Dieldrin 25.18 % calculated amount of 2279.809 PPB was recorded. Endosulfansulfate 31.92 % calculated amount of 6.852 PPB, Dimethoate 10.71 % calculated amount of 17.410 PPB, Op-DDT 29.32 % 12.247 PPB, Chlorpyrifos 18.73 % 2.636 PPB, Op-DDE 22.97 % 3.406 PPB, Atrazine 11.21 % 49.688 PPB, Malathion 17.99 % 63.581 PPB, Aldrin 17.84 % 4.608 PPB, Methylparathion 15.39 % 55.599 PPB and Ethion 29.96 % 4.301 PPB similarly in inorganic Okra crop Alachlor 16.00 %

calculated amount of 3398.590 PPB, Dimethoate 11.00 % 910.462 PPB, op-DDD 26.36 % 18.414 PPB, Metalaxyl 16.38 5 18.463 PPB, Op-DDE 23.12% 9.911 PPB, Malathion 18.15 % 328.863 PPB, Phosphomidon 15.06 2677.786 PPB, Phorate 9.98 % 113.271 PPB, b-Endosulfan 28.07 % 240.011 PPB, g-Lindane 11.80 % 158.151 PPB and b-Lindane 11.62 % calculated amount of 5511.597 PPB. It is in the large quantity this indicated that application of chemicals on the crops during different crop growth stages is unhealthy. This may result in too many consequences to the human beings as well as animals.

Conclusion

Ginger is the leading horticultural crops in Shivamooga district and also in the state of Karnataka since many decades use of chemical fertilizers has led to decrease the soil fertility this resulted in the low productivity. Indiscriminate use of chemical fertilizers and pesticides has led to several problems such as environmental pollution, decreases in crop yields and loss in soil fertility and increase in pesticide resistance in pests. In this situation alternative method of augmenting fertilizers and pesticides assume importance. The ill effects on chemical farming have generated interest in organic farming by using farm and urban wastes through vermiculturing. Use of pesticides is a necessary evil of the modern agro-technology and perhaps man would never stop their use. The use of pesticides is directed against economic pests, but they also affect a wide range of no target organisms, including desirable animals, plants and non harmful beneficial insects.

Organic farming is the form of agriculture that relies on techniques such as crop rotation, green manure, compost and biological pest control to maintain soil productivity and pest control on a farm. Organic farming excludes or strictly limits the use of manufactured fertilizers and pesticides, plant growth regulators such as hormones, livestock, antibiotics, food additive and genetically modified organisms. Organic farming is gaining ground in India and farmers are being encouraged to grow organic produces. Due to high prices of inorganic inputs, organic has become the main motivating factor for farmers and to produce organic inputs for successful organic crop production is still seems to be a big challenge before farmers. Thus it was concluded that organic treated plot shows that low pesticide residue.

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