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A Peer Reviewed & Refereed, International Open Access Journal

Vol.4.Issue.1.2017 (Jan-Mar) ISSN:2455-0221(P), 2394-2606(O)

RESIDUAL ANALYSIS OF PESTICIDES / PERSISTENT ORGANIC POLLUTANTS IN SOIL AND WATER BY USING GAS CHROMATOGRAPHY – MASS SPECTROSCOPY

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ABSTRACT

A simple accurate and precised GC-MS method for the determination of Dimethoate, 4,4'- DDD, 4,4' – DDE, α-Endosulfan pesticide was developed. By the analysis, retention time was found to be 17.235min for Dimethoate, 21.739min for 4,4'-DDE, 22.518min for 4,4'-DDD and 21.316min for α-Endosulfan with a correlation coefficient (r2) of 0.9967. The limit of detection (LOD) was calculated and found to be 0.11 µg and limit of quantification (LOQ) was found to be 0.33 µg. In water samples precision values % RSD values were found to be 0.170 and in soil samples precision values were found to be 0.205 respectively. The peaks are clearly indicated that the compounds are fairly enriched after Solid Phase Extraction which is the main point of extraction.

Keywords: Pesticides, Solid Phase Extraction, GC-MS Equipment, Water and Soil.

INTRODUCTION

Chemicals are used in the production of many items that we depend on every day. They have been widely used all over the world because they have enabled the development of agricultural and farming production by controlling a wide range of pest and disease. Evidence demonstrates that certain organic chemicals are persistent, bioaccumulative, toxic and that these chemicals cause long-term harm to the health of human beings and the planet's environment. However, it is well known that the application of these substances can leads to damage for human health as well as the environment [1][2].

Throughout the world, people and their environments are exposed on a daily basis to persistent organic pollutants (POPs). POPs and their effects on human health and the environment is a global issue of concern. They accumulate in animals and humans, predominantly in fatty tissues. As these chemicals move up the food chain they concentrate to the levels that are harmful to fish, humans, and wildlife. As chemicals plays a key role in our daily lives, while essential to our economic development and our current standard of living, the production, uses and release of thousands of chemicals into the market place are also associated with problems including those related to unintentionally produced by-products, waste generation and disposal, environmental degradation and wild life and human exposure [3].

Study area:

The water and soil samples were collected from Bapatla, Nizampatnam, Repalle, Bhattiprolu and Kolluru areas of Guntur district, Andhra Pradesh for the analysis of Dimethoate, 4,4'- DDD, 4,4' – DDE and α-Endosulfan pesticides. Guntur district has coastline of approximately 100km and is situated on the right bank of Krishna

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River which separates it from Krishna River and extends till it empties into the Bay of Bengal. It has total area 11,391 km^2. The geographical coordinates of Guntur district are 16^{0}18′N 80^{0}27′E. The district is a major centre for agriculture. It exports large quantities of chillies and tobacco. The water and soil samples were collected from the selected areas which were represented in the Figure 1.

Figure 1: A Map indicating the sampling sites in Guntur district

Materials and Methods

Chemicals: Reference standards of 4,4'-DDD, α-Endosulfan, Dimethoate,4,4'-DDE (purity >99%), Methanol, Dichloromethane, Deionised water, Ethyl Acetate, anhydrous sodium sulphate of pesticide residue analysis grade were purchased from Sigma Aldrich.

Equipments: Agilent 6890 N GC equipped with 5973 inert mass selective detector (Agilent Technologies, USA), Agilent 7683 Series auto sampler, 3ml SPE C18 Cartridge, Centrifuge Model: RC -8C, Disposal Syringe Filters – pre-size 0.45µm, Micro Spatula, 15ml Centrifuge Tubes, 2ml Vials were used.

Preparation of working standards and stock solutions: A 1000 mg/L standard solution of α-Endosulfan was prepared in methanol by dissolving 10 mg of α-Endosulfan in 10 mL of methanol. This solution was diluted with methanol to obtain the necessary concentrations (10 µg/mL to 100 ng/mL) to draw a calibration curve for quantification of α-Endosulfan in the samples. Similarly, the standard solutions of 4,4'-DDD, 4,4'-DDE and Dimethoate was prepared. Individual standard stock solutions were prepared at 1mg/mL by dissolving 10mg of each pesticide in 10ml of ethyl acetate and were stored at −20 ^{0}C until use.

Preparation of Blank sample: 5ml of stock solution was added to the purified water and it was used as blank for water analysis. Similarly, 5ml of stock solution was added to the soil samples and it is extracted by using SPE and analysed by GC-MS.

Sampling: The water samples were collected from appropriate 0.4m depth below the water surface. Approximately 0.5-2L water sample were collected in a screw cap glass bottles which was already cleaned with water followed by an acetone rinsed and dried. The P^H of the water was measured, if it is found to be above 8, the container was sealed with Teflon to avoid chemical volatilization of Endosulfan isomers if present. But we were adjusted the P^H below 7 by adding the phosphate buffer whose P^H is 6 [4][5]. The water samples were immediately shifted to the laboratory in an ice chest with ice and stored at 4 ^{0}C.

The sediment samples were taken from the positions where a fine texture substrate deposition takes place. Approximately the upper 2cm of the bed sediment were collected with a Teflon coated spoon and stored it in a aluminium containers at -20^{0}C in the laboratory until analysis [6].

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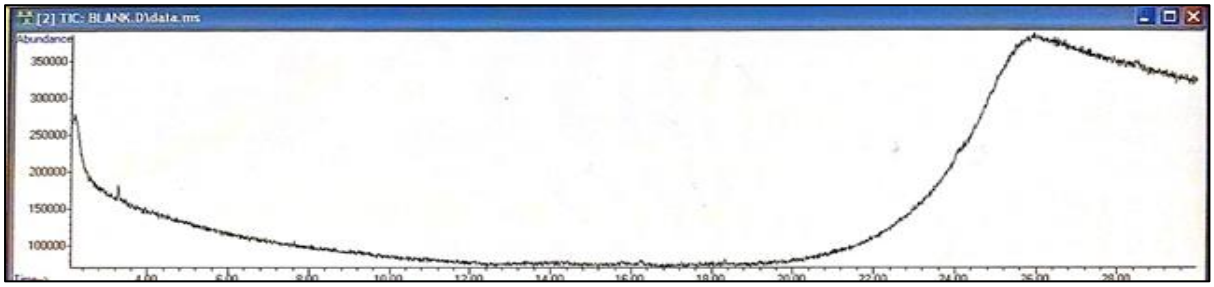
Extraction: To the 3ml C18 solid phase extraction column, 5ml of methanol is added. This is known as conditioning the cartridge. Conditioned step is very important to active the material in SPE cartridge because the cartridge is packed with dry material. Generally, the cartridge is packed with polymers/silica materials. It is critical to place silica materials and easy to place polymer SPE materials. By conditioning the polymer SPE material improves the flow. So, it must be active and effective by using organic solvents. After conditioning, equilibrated by 5ml aqueous solution i.e., water. Organic solvents will have dispersed by aqueous solution. By these particles will interact with the sample. Loaded 5ml of water sample which is already containing spiked 10µl/1ml of pesticide mixture stock solutions. Washed the cartridge with 1ml deionized water for 3times. The importance of washing by using a solvent/ solution is to elute the interferences from the SPE material without removing the analytes of interest. After washing the column, the cartridge is dried, and then eluted the analytes with 5ml of ethyl acetate. Collected the eluate in a 15ml centrifuge tube and added 500mg of sodium sulphate to it. Centrifuged the collected eluate for 10mins at 5000rpm. Collected the supernatant and evaporated the organic layer under reduced pressure passing nitrogen gas. Reconstituted it with in 200µl ethyl acetate solvent. Finally, the 1µl of reconstituted sample was injected into GC-MS for analysis in both SCAN and SIM modes.

5g of soil is taken into 5ml vial and spiked with 5ml of 10µl/1ml of pesticide solutions to it. From the above mixture, weighted 1g of soil from it into another 2ml vial. Transferred the mixture into 15ml centrifuge tube. Extracted the soil sample by adding 5ml Dichloromethane. After adding the dichloromethane, centrifuged the sample at 5000rpm speed in a centrifuge machine for 10mins. Collected the supernatant and filtered it with syringe filter which is having 0.44mm particle size. After filtering the supernatant, evaporated the organic layer under reduced pressure passing nitrogen gas. It is reconstituted the evaporated soil sample by adding 200µl dichloromethane solvent. Finally, 1µl of reconstituted sample was injected into the GC-MS. The sample is analysed in SCAN and SIM mode.

GC-MS Analysis: The GC–MS analyses were carried out on Agilent 6890 N GC equipped with 5973 inert mass selective detector (Agilent Technologies, USA), Agilent 7683 Series auto sampler and a SPB-624 (Supelco, USA) capillary column of length 30 m, 0.25 mm internal diameter and 0.25µm film thickness. The column oven was programmed initially from 4 ^{0}C with 2 min hold up time to the final temperature of 240 ^{0}C with 10 ^{0}C/min ramp. The final temperature hold-up time was 8 min. Helium was used as carrier gas in constant flow mode at a flow rate of 1.2 mL/min. The inlet and GC–MS interface temperatures were kept at 280 ^{0}C. The temperatures of the EI source and quadrupole analyzer were kept at 230 ^{0}C and 150 ^{0}C, respectively. The Mass Selective Detector scan range was 20–800 m/z. The quantification of targeted pesticides was done in the SIM mode and the dwell time was set to be 25 ms. The samples (1.0 µL) were injected in to GC–MS by using auto sampler in splitless injection mode with 0.2 min splitless time and each extract was analysed three times to obtain an average peak area.

GC-MS results for water and soil samples from the different areas of Guntur district is shown on the following chromatograms



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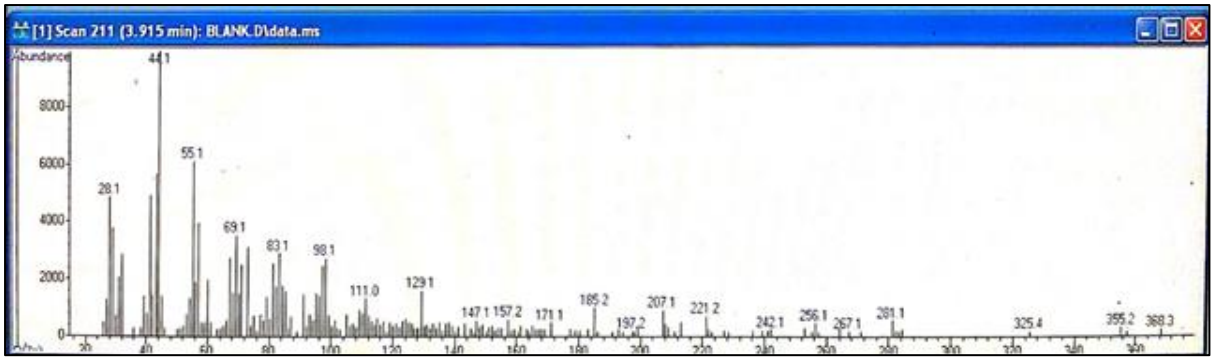

Figure 2: Chromatogram for blank sample (water and soil)

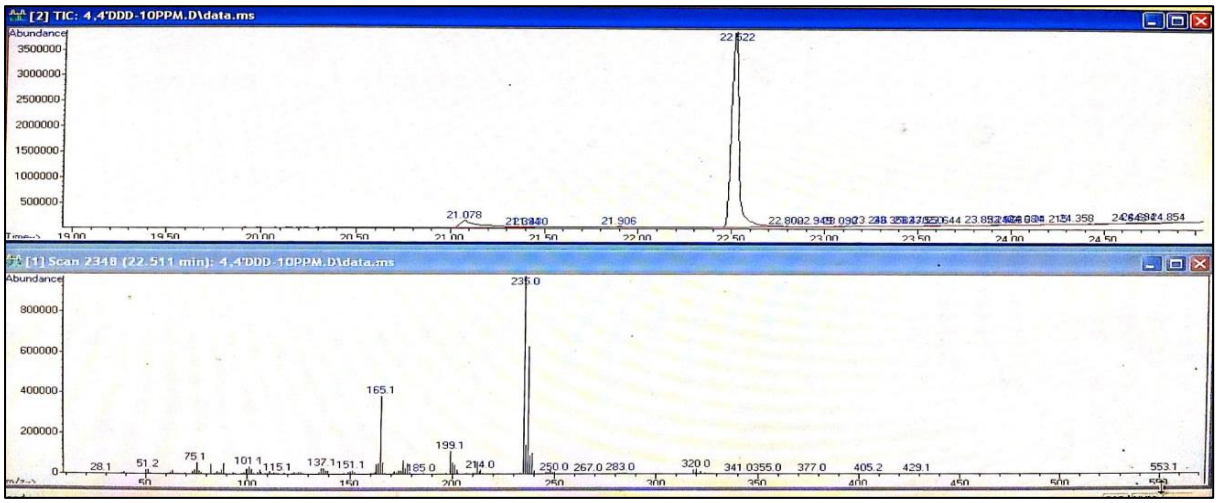

Figure 3: Chromatogram for 4,4'–DDD of 10ppm concentration.

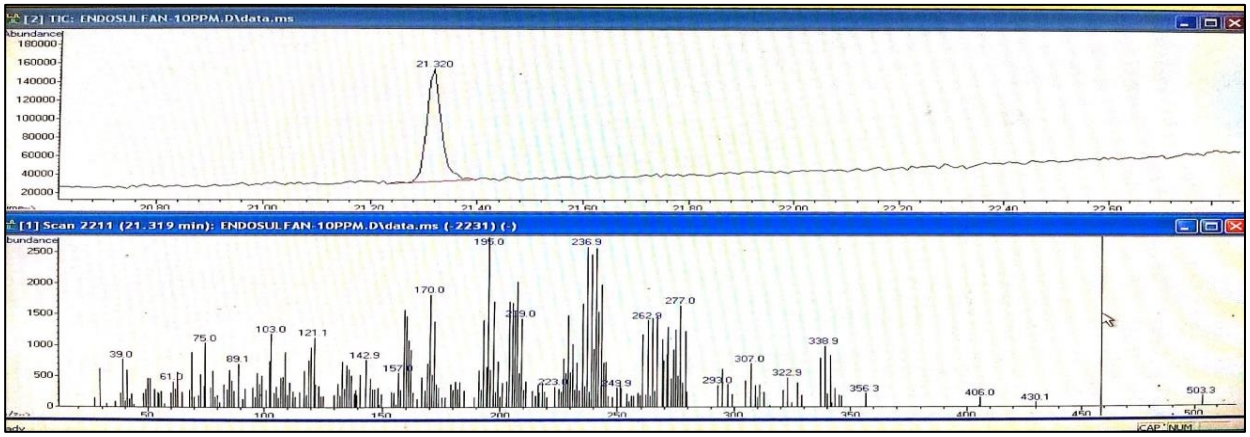

Figure 4: Chromatogram for α-Endosulfan of 10ppm concentration

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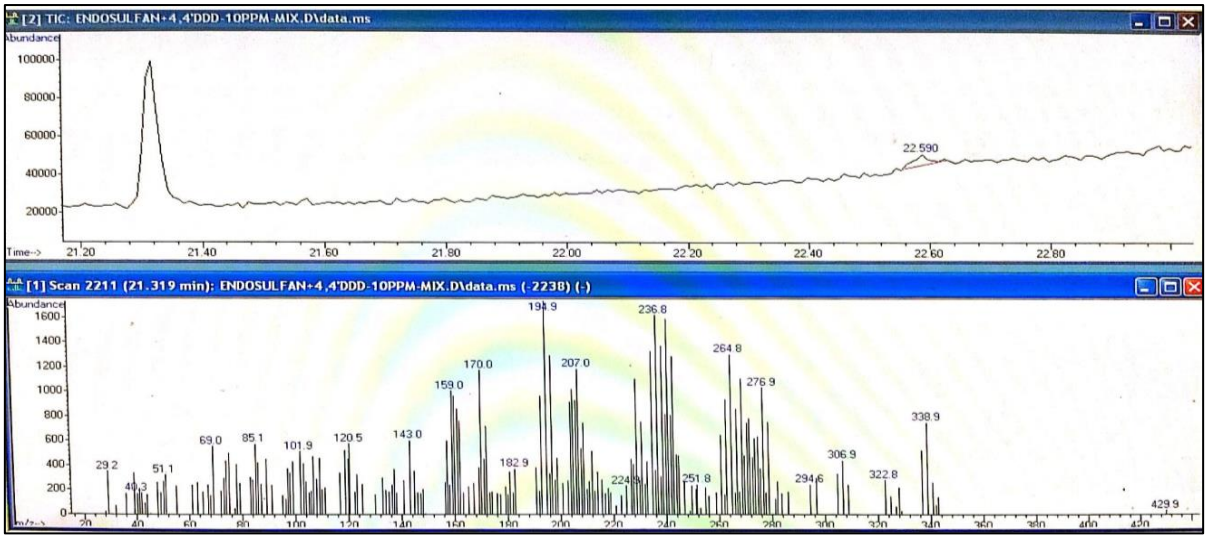

Figure 5: Chromatogram for water sample of 4,4'-DDD & α-Endosulfan

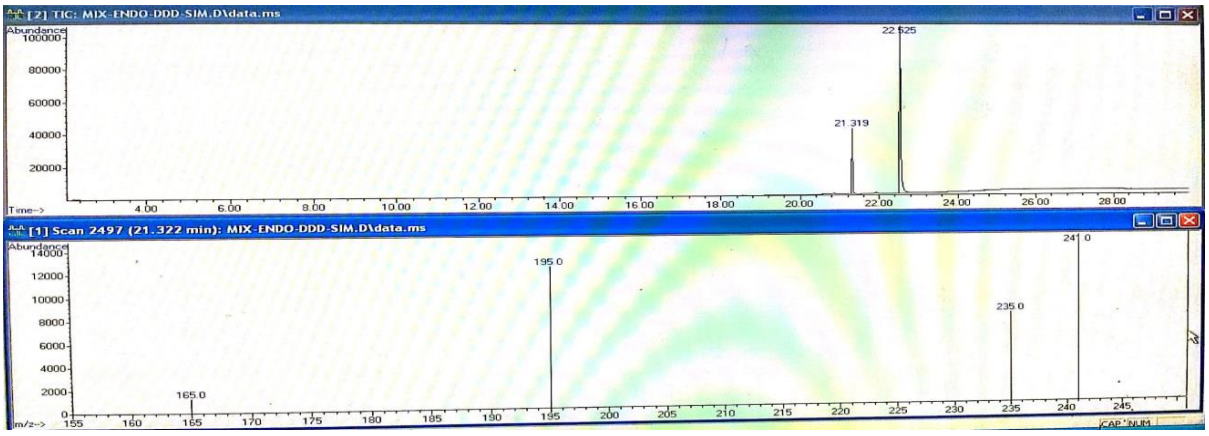

Figure 6: Chromatogram for soil sample of 4,4'-DDD & α-Endosulfan

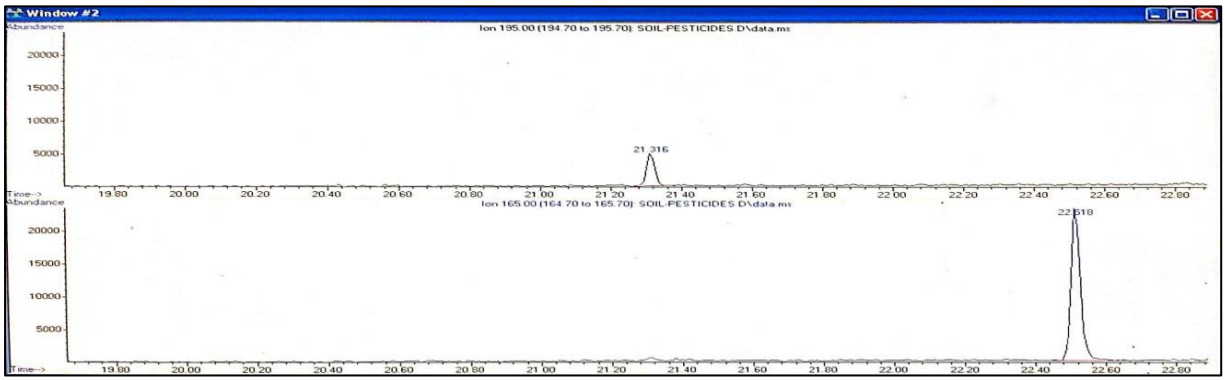

Figure 7: Chromatogram for Soil sample indicating R_t 21.316min for α-Endosulfan and R_t 22.518 min for 4,4'-DDD.

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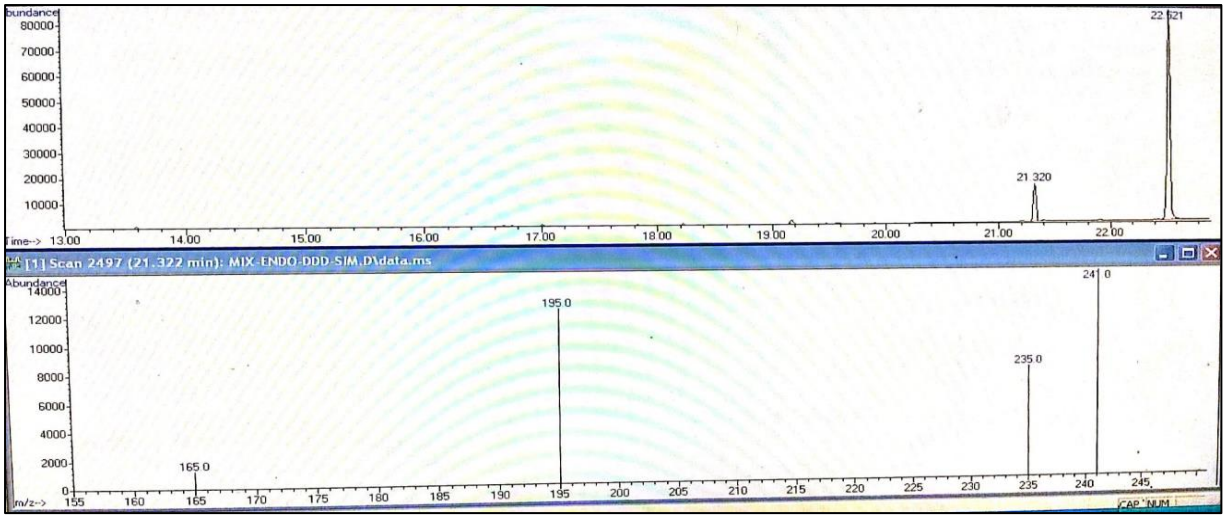

Figure 8: Chromatogram for water sample in SIM and SCAN mode indicating both minor and major fragments in α-Endosulfan and 4, 4'-DDD.

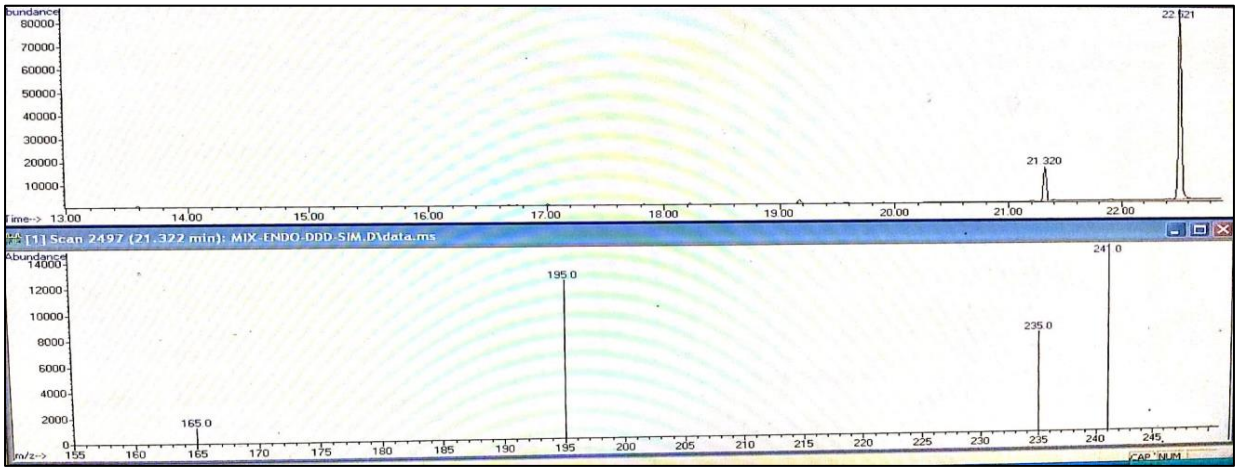

Figure 9: Chromatogram for soil sample in SIM and SCAN mode indicating both minor and major fragments in α-Endosulfan and 4, 4'-DDD.

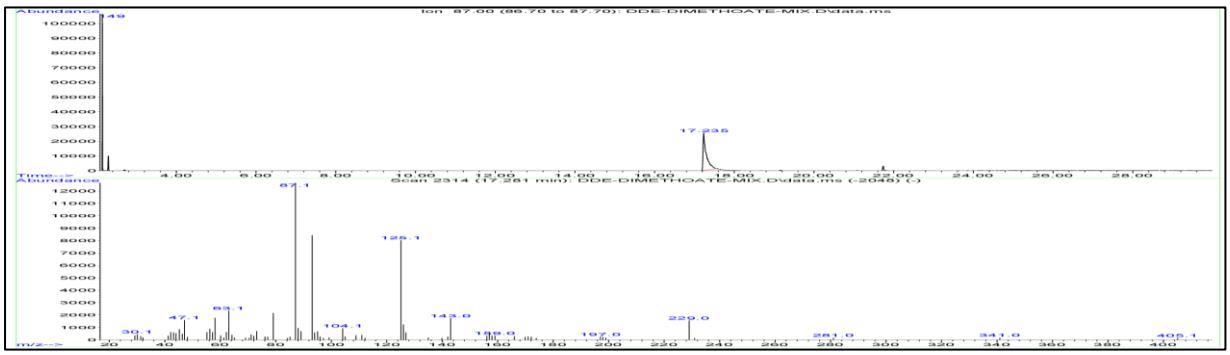

Figure 10: Chromatogram for Dimethoate showing the R_t value of 17.235 min and major fragment of 87 m/z and minor with 125 m/z values

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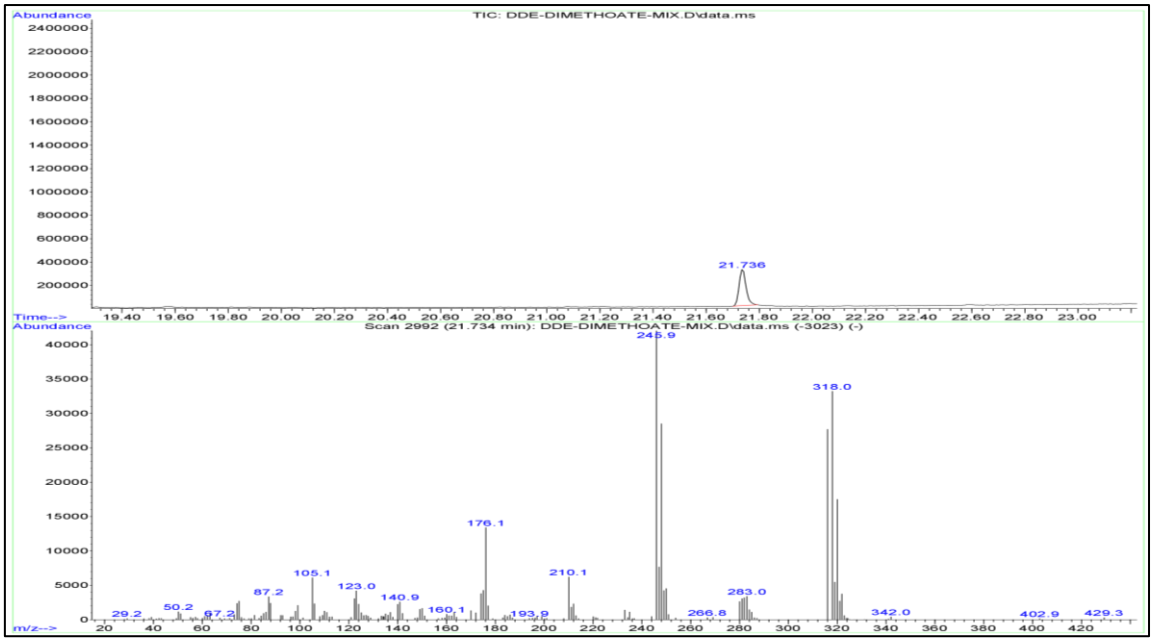

Figure 11: Chromatogram of 4,4'-DDE indicating the R_t value 21.739 min and major fragment 318 m/z and minor with 246 m/z values.

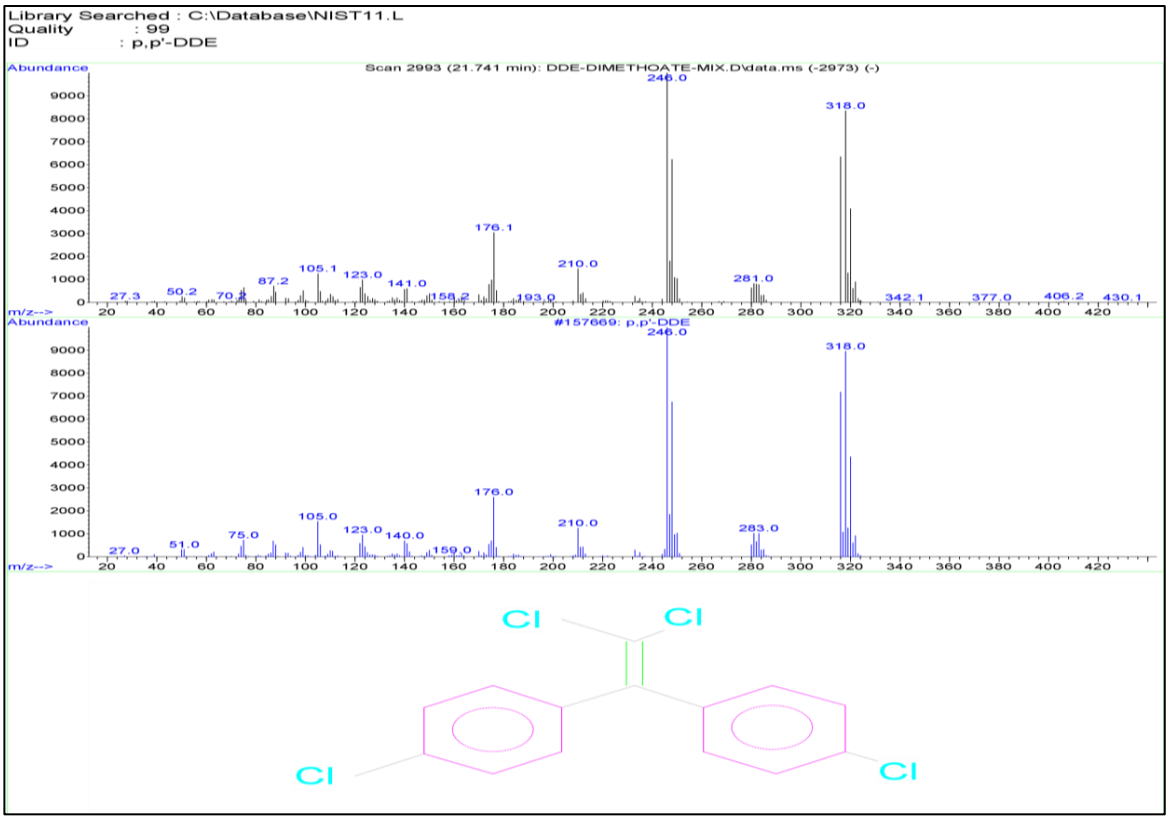

Figure 12: Chromatogram for 4,4' – DDE indicating the major and minor fragments

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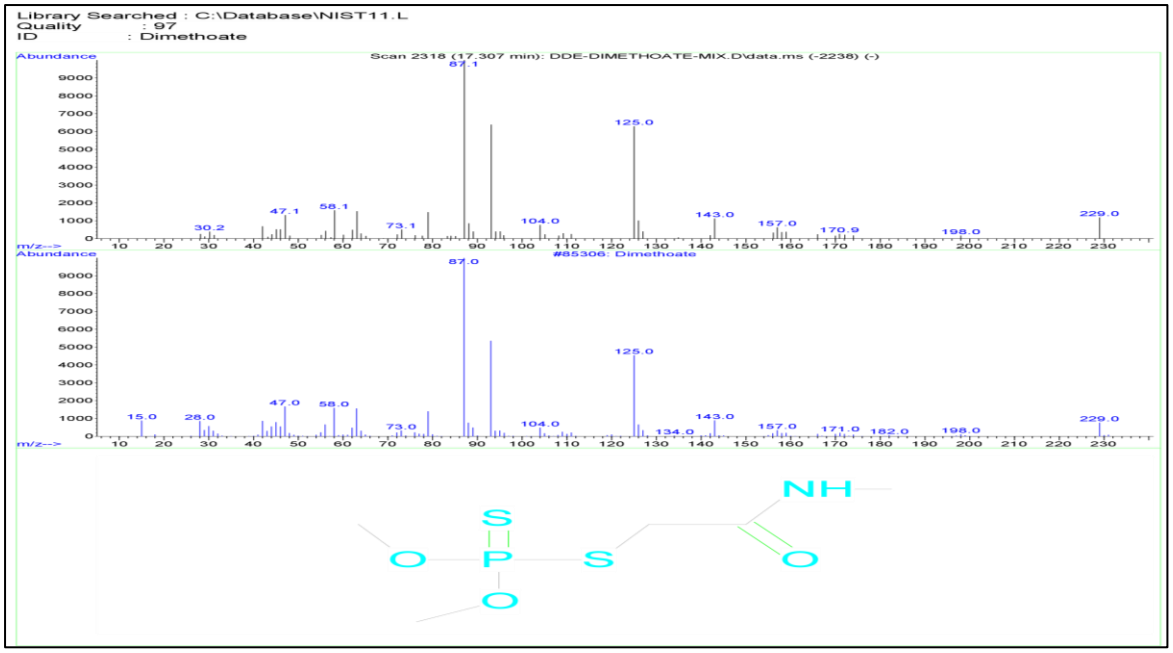

Figure 13: Chromatogram for Dimethoate showing the minor and major fragment

Quantitative analysis:The Linearity concentrations of the water and soil samples were observed as follows for targeted compounds.

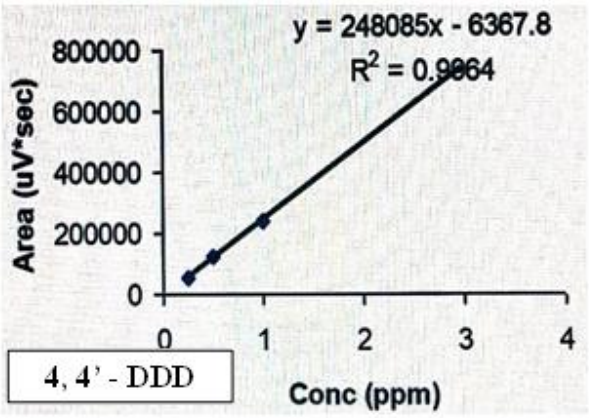

Figure 14: Calibration curve for 4, 4' – DDD

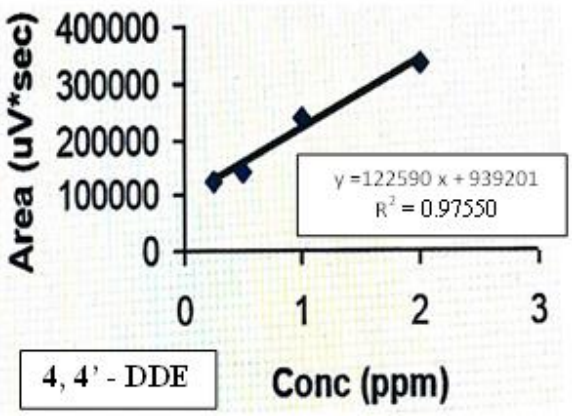

Figure 15: Calibration curve for 4, 4'-DDE

Retention times, quantitation ions and confirmation ions for pesticides analysed by GC/MS.

S.No.	Target compounds	Retention time (min)	Quantitation ion (m/z)	Confirmation ions (m/z)
1.	4,4'- DDD	22.5	235	165,235
2.	4,4' – DDE	21.739	256	176,246,318
3.	Dimethoate	17.235	125	87,125
4.	α-Endosulfan	21.3	241	195,241

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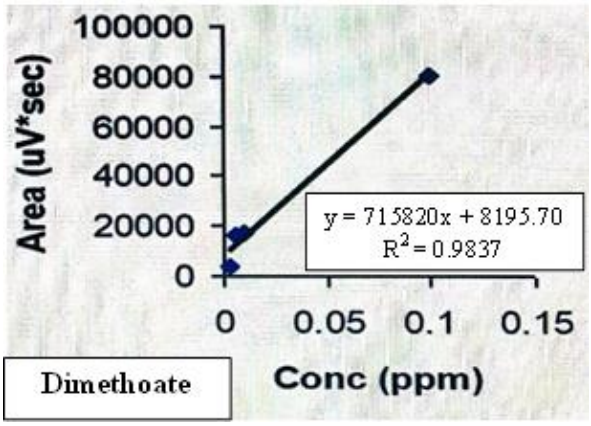

Figure 16: Calibration curve for Dimethoate

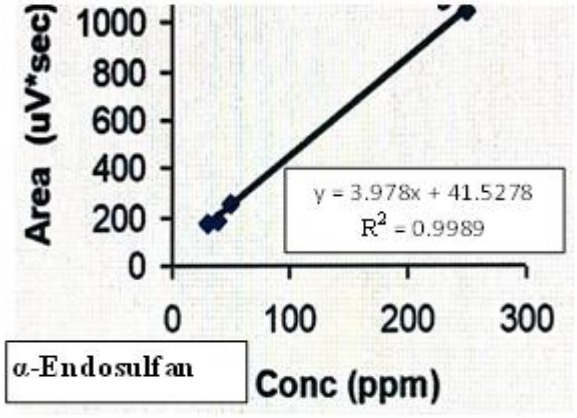

Figure 17: Calibration curve for α-Endosulfan

Results and Discussion

A simple accurate and precised GC-MS method for the determination of Dimethoate, 4,4'- DDD, 4,4' – DDE, α-Endosulfan pesticide was developed. From the chromatogram, retention time was found to be 17.235min for Dimethoate, 21.739min for 4,4'-DDE, 22.518min for 4,4'-DDD and 21.316min for α-Endosulfan with a correlation coefficient (r2) of 0.9967. The limit of detection (LOD) was calculated and found to be 0.11 µg and limit of quantification (LOQ) was found to be 0.33 µg. In selected areas, water samples precision values % RSD values were found to be 0.170 and in soil samples precision values were found to be 0.205 respectively. The peaks are clearly indicated that the compounds are fairly enriched after extraction which is the main point of extraction. Figure 14 to 17, indicates the calibration curves for the different compounds namely 4,4'- DDD, 4,4'- DDE, Dimethoate and α-Endosulfan. These calibration curves were plotted to determine the different concentration in the soil and water samples in Guntur District. The chromatograms show that the retention times of 4,4'- DDD (22.518min), 4, 4'- DDE (21.739min), Dimethoate (17.235min) and α-Endosulfan (21.316min).

The molecular weights of the target compounds are 4,4'- DDD (318), Dimethoate (229), 4, 4'- DDE (318) and α-Endosulfan (404). The calibration curve shows the 0.00736ppm with %RSD 4.7 in soil and 0.074436ppm with %RSD 7.0 in water sample for 4,4'-DDD, 0.637490ppm with %RSD 3.2 in soil and 0.606208ppm with %RSD 4.3 in water for 4,4'-DDE, 0.637490ppm with %RSD 3.2% in soil and 0.543720ppm with %RSD 4.2 in water for Dimethoate and 0.00623ppm with %RSD 5.2 in soil and 0.0056ppm with %RSD 5.2 in water for α-Endosulfan. All compounds showed a % RSD of less than 10 in Bapatla, Nizampatnam, Repalle, Bhattiprolu and Kolluru areas. These values of validation parameters indicated that the analysis using the GC-MS method is quite acceptable. The calibration curve shows the different concentrations that were tested by dilution of the 1ppm mixture to determine the lowest detection limit of the mixture. This was done to determine the lowest concentration of the mixture of the compounds that could be detected by the GC-MS.

Farshid et al. reported that the DDE in water samples showing mean concentration of 0.055ppb with 0.003 standard deviation at range 0.05-0.14ppb. DDE is detected in 84% of water samples whereas DDE in sediment samples gave mean concentration 9.840ppb with 2.85 standard deviation at range 12.27-30.43ppb which was detected in 96% of sediment samples. As well as in water samples the Endosulfan showing 0.046ppb mean concentration with 0.009 SD at range 0.30-0.46 was detected in 60% of water samples and in sediment samples it is 10.622ppb mean concentration with 1.72 SD of range 4.12-15.22ppb which was detected in 46% of sediment samples [7] which were less than the results obtained in this investigation.

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J.C. Akan et al. reported that 4,4'-DDD mean concentrations in fish samples are 3.12µg/g with standard deviation 0.06 in liver, 2.04 µg/g with 0.02 SD in gills, 1.93 µg/g with 0.11SD in stomach and 1.22 µg/g with 0.01SD in flesh. The Endosulfan in fish samples mean concentrations are 5.64 µg/g with 0.22 SD in liver, 3.21 µg/g with 0.31 SD in gills, 2.94 µg/g with 0.15SD in stomach and 1.75 µg/g with 0.11SD in flesh [8].

Sergiane et al. has been reported that the Dimethoate showed 10.34 min retention time having 87, 125, 93 monitor ions whereas 125 ions are used for quantification and 0.704 Kow with vapour pressure 0.25pa having one segment [9].

Elipiniki et al. reported that the Dimethoate in Olive oils showed vapour pressure 8.25E-06 mmHg (25^0C), solubility in water at 25000(21^0C) having log Kow 0.78 with MRL 2000 µg/kg and the α-Endosulfan showed vapour pressure 3.00E-06mmHg (25^0C), solubility in water at 0.51 (20^0C) having log Kow 3.83 with MRL 50 µg/kg. The α-Endosulfan shows 75.0 ± 9.5 initial elution and 96.0 ± 8.0 optimized elution. Dimethoate shows mean concentration of 120.6 and relative standard deviation with 0.8. The Dimethoate showed 15.02min retention time, LOD 1µg/L, LOQ 3 µg/L with linear range 5-200 µg/kg, R^2 0.9980 and %Relative standard deviation 8.9%. Where as in α-Endosulfan, the retention time is 27.19min, LOD 0.1 µg/L, LOQ 0.3 µg/L, linear range 5-500 µg/kg with R^2 0.9974 and %RSD is 8.5. G. Mariani et al. reported the α-Endosulfan showing 53.62 RSD% with internal standards recovery percentage (ISR %) is 114 and 0.94-4.7 intervals, 4,4'-DDE showed 7.83 RSD% with 114 ISR% and 6.2-11 intervals and 4.4'-DDD showing 7.20 RSD% with 2.0-3.7 intervals [10].

Yoann Fillatre et al. reported that the Dimethoate shows the retention time 3.4min, declustering potential 11V and Selective Reaction Mode I show 230.0/199.0 quantification ions, collision energy is 13V, whereas in Selective Reaction Mode II it is 230.0/125.0 confirmation ions and collision energy is 29V. The Dimethoate having R^2 0.9964 with linearity range 0.1-1 µg/L, LOD is 0.023 µg/L, LOQ is 0.08 µg/L and maximum %RSD is 10.6% [11].

M.L Cervera et al. reported that the retention times for α-Endosulfan, 4,4'- DDE and 4,4'-DDD is 25.55mins, 26.62mins and 28.16mins and their quantification ions are 169.9690 m/z, 246.0003 m/z and 235.0081 m/z. At three spiked levels (0.01, 0.05 & 0.5 mg/kg) of α-Endosulfan, the average recoveries and RSD% in orange is 0, 91(13),73(6), LOQ 0.05mg/kg, in apples is 0,71(34), 93(11), LOQ 0.5mg/kg, in carrot 93(22), 99(7), 93(7), LOQ 0.01mg/kg, in tomato's is 94(20), 76(9), 96(5) and LOQ is 0.01mg/kg. In 4,4'- DDE, the LOQ in oranges, apples, carrots and tomato's is 0.01mg/kg. In 4,4'- DDD, the LOQ is 0.01mg/kg [12].

M. Roszko et al. reported that the α-Endosulfan gave 0.1 mg/kg^{-1} fortification level, 96% recovery (n=3) and relative standard deviation is 13%. The Dimethoate shows 0.1 mg/kg^{-1} fortification level 88% recovery and RSD is 2%. Whereas the 4,4;-DDD and 4,4'-DDE showing 0.4 mg/kg^{-1} fortification, 79% recovery and RSD is 5% [13].

S.Enbaia et al. reported that the Endosulfan showed mean concentration of 0.0003ppm with standard deviation 0.006ppm and 4,4'-DDD having mean 0.0021ppm with standard deviation 0.05ppm were detected in fish tissues[14].

Razia Sultan et al., reported that the Organochlorines i.e. 4,4'-DDE showing 97.8% recovery and relative standard deviation 5.2%. The limit of detection of 4,4'-DDE is 0.002mg/kg and limit of quantification is 0.005mg/kg. DDD showing 95.4% recovery and relative standard deviation 4.7% with LOD 0.002mg/kg and LOQ 0.005mg/kg [15].

Conclusion

The conditions of GC-MS were successfully optimized and the method developed was applied in the determination of selected pesticides studied. From the results carried out for the analysis in this study, it was

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A Peer Reviewed & Refereed, International Open Access Journal
Vol.4.Issue.1.2017 (Jan-Mar) ISSN:2455-0221(P), 2394-2606(O)

revealed that dichloromethane was the best solvent system for the determination of the selected pesticides in soil and Ethyl acetate in water. The analysis time was shortened and peak resolution was good in most cases 20 - 800 m/z.

Solid Phase Extraction, Gas Chromatography-Mass Spectroscopy were used for the separation and quantification of organochlorine, and organophosphates pesticides in the Guntur region. The methods applied were very effective since the presence of 4,4'-DDD, 4,4'-DDE, Dimethoate and α - Endosulfan was clearly confirmed. This study has shown that SPE with DCM, Ethyl Acetate is an accurate and reliable and effective method for the determination of target compounds at low concentrations in the environment. Further, it has been established that, although DDT has been banned, it is still present in the environment. The sources of these Organochlorine and organophosphorous compounds in the water systems might be from industrial effluents and or from diffuse sources such as run-off from agricultural lands. Thus, the presence of target compounds and some of its degradation residues in water systems can be attributed to their wide usage before their banning. Since they are persistent enough and degrade very slowly they accumulate in the soil and they are transported down to the water sources. It is recommended that the organochlorine and organophosphates be analysed using Gas chromatography- Mass spectroscopy as a reliable and accurate method of analysis. SPE is a cheap and best method for extracting the pesticide compounds which give quick and fast results during experiment. Pesticides should be tested in soil and sediments which might give a more precise conclusion of these pesticides. Furthermore, regular monitoring is needed to evolve a strategy to manage the environmental hazards due to these pesticides to avoid pollution of our water sources.

ACKNOWLEDGEMENTS

This work was financially supported by University Grants Commission – SERO, Hyderabad and the authors are grateful to UGC for their constant support. We are very much thankful to the Principal and Dept. of Chemistry, Govt. College for Women (A), Guntur for giving support and encouragement for carrying out the research work.

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A Peer Reviewed & Refereed, International Open Access Journal
Vol.4.Issue.1.2017 (Jan-Mar) ISSN:2455-0221(P), 2394-2606(O)

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