

## ANALYSIS OF PESTICIDAL RESIDUE IN SOIL OF MAIHAR REGION OF SATNA DISTRICT OF MADHYA PRADESH

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### ABSTRACT

*In this Analysis work five village of Maihar region of Satna District have been selected for collection of soil samples. Soil samples were collected by standard methods and these samples were analysed for pesticidal residues. In targeted various pesticides only pendimethalin was detected in selected three villages. The reason behind this is excessive use of pendimethalin in these villages and pendimethalin is slow biodegradable pesticide. Present concentrations of pendimethalin in soil of three villages are not so high to cause fatal adverse effect.*

**KEYWORDS:** *Maihar Region, Pesticidal Residue, Pendimethalin*

### INTRODUCTION

In Madhya Pradesh State, Maihar is a tehsil of Satna district and it is famous for goddess Sharda Temple. This tehsil is located at 24.27°N 80.75° and have average elevation of 376 meter. This area is hub of cement production and is known as limestone zone because of good quality of lime stone for cement production. Total population of this area is nearly 358725 and people of this area are mainly dependent on agriculture [1, 2]. There are farming of many crops takes place like wheat, rice, pulses (Chana, Arhar) including different type of vegetables like Bittergourd, tomato, onion, pumpkin & Mustard etc. The yield of crops is required in large amount due to increase in the population. Because of the requirement of high yield production the farmers are using different chemical compounds in which various type of pesticides [3] are used to kill unwanted pests and herbs. Residue of these pesticides in soil causes environmental problems because some pesticides are not biodegradable. Biodegradable pesticides are broken down by microbes and less harmless for environment whereas persist pesticides are those which may take months or years to break down [4]. Those pesticides which are slowly degraded are present in soil in the form of residue and cause problems.

In present study 05 villages have been selected for study purpose. These are Kakara, Jeetnagar, Podi, Bhadanpur, Kuseri. Collected soil samples from these villages were analyzed and it was found that in 3 of these villages the **Pendimethalin** in high concentration. A survey has been done for the health of people of the village in which pendimethalin was found. It was observed adverse effect was found in some people but not hazardous. In the future concentration of pendimethalin continued to increase in these villages then there may be fatal impact. Now it is very important to continue this study to find out how much pendimethalin is increasing every year and its effect on human health.

## MATERIAL AND METHODS

### Collection of Samples from Selected Sites at Maihar Region

Samples of soils were collected from Kakara, Jeetnagar, Podi, Bhadanpur, Kuseri villages of Maihar Tehsil of Satna district. About ½ kg samples from five farms of each village were collected following standard sampling pattern that is soil samples from each corner, mid of farm were taken mixed homogenously and representative ½ kg were taken for testing and send to Institute of pesticide Formulation Technology, (NABL & APEDA accredited laboratory), Gurugram Haryana for analysis of soil from different villages of Maihar region.

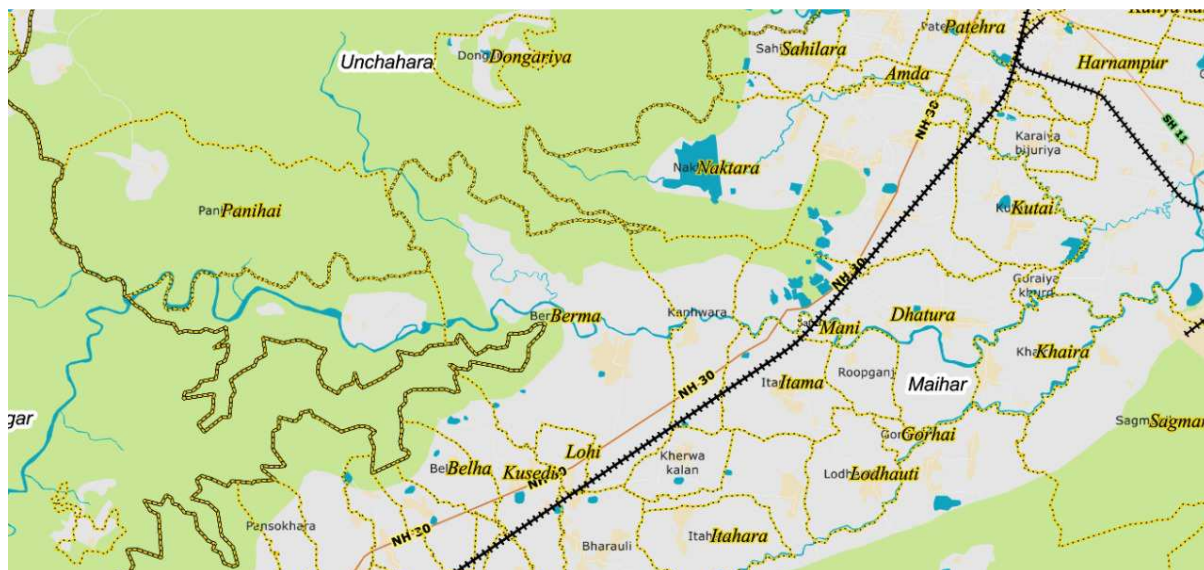


Figure 1: Selected Villages from Maihar Region.

### Chemicals

Anhydrous sodium sulfate ( $\text{Na}_2\text{SO}_4$ , AR grade), sodium chloride (Hi-Media, AR grade) methanol and n-hexane solvents of pesticide residue were grade purchased from Merck (Darmstadt, Germany). The soil samples were screened for 76 pesticides viz. Propoxur, 3,4-Dichloraniline, alpha-BHC, Pencycuron, Phorate, Thiometon, Dimethoate, Dazomet, Simazine, Atrazine, beta-BHC, Propetamphos, delta-BHC, Chlorothalonil, Paraoxon Methyl, Etrimfos, gamma-BHC (Lindane), Iprobenfos, Propanil, Metribuzin, Malaoxon, Alachlor, Transfluthrin, Metalaxyl (Mefenoxam), Pirimiphos-methyl, Linuron, Malathion, Metolachlor (S-Metolachlor), Chlorpyrifos, Fenthion, Flufenacet, Pendimethalin, Fipronil, Heptachlor-exo-epoxide, Chlorfenvinfos, Captan, o,p'-DDE, Butachlor, Endosulfan-I, Hexaconazole, Fipronilsulfone, Profenofos, p,p'-DDE, Myclobutanil, Oxyfluorfen, o,p'-DDD, Buprofezin, Kresoxim-methyl, Chlorfenapyr, Ethion, Triazophos, Benalaxyl, Propiconazole-1, Endosulfan sulfate, Propiconazole-2, p,p'-DDT, Diclofop-methyl, Propargite-1, Propargite-2, Etoxazole, Fenpropathrin, Fenazaquin, Phosalone, Pyriproxyfen, L-Cyhalothrin, Permethrin-1, Permethrin-2, Cyfluthrin-1, Cyfluthrin-2, Cyfluthrin-3, Cyfluthrin-4, Cypermethrin-1, Etofenprox Fenvalerate-1, Fenvalerate-2 (Esfenvalerate), Deltamethrin-1 (Tralomethrin deg.-1). The Certified Reference Material (CRM) was purchased from Sigma Aldrich (USA).

### Extraction of Soil for Pesticide Residue

The soil samples were dried, sieved and mixed homogeneously. 20 g sample was taken in 250 ml stoppered conical flask followed by addition of 50 ml of methanol (HPLC grade) to each conical flask. Each conical flask was stoppered and subjected shaking on auto-shaker for one hour at 160 rpm. After one hour of shaking, samples were left undisturbed on flat surface for half an hour for settling of soil particles. After half an hour, 20 ml of clear supernatant was pipetted out and filtered through anhydrous sodium sulphate (Na<sub>2</sub>SO<sub>4</sub>), pre washed with 20 ml methanol to reduce the absorption of pesticides in the sample during clean up. All the filtrate was collected into 100 mL flat bottom round flask. Anhydrous sodium sulphate (Na<sub>2</sub>SO<sub>4</sub>) bed was finally washed by 10 mL of methanol (HPLC grade) into same flat bottom round flask. The solvent was evaporated near to dryness on rotary evaporator (Buchi RP-300) under vacuum (500 mm of Hg) keeping constant temperature of water bath at 40°C. The sample was reconstituted by 2 mL n-hexane for instrumental analysis using gas chromatography mass spectrometry.

### Standard Stock Solutions

The stock solution of Certified Reference Materials (CRM) of pesticide was prepared. Individual pesticide weighed in volumetric flask of 10 ml. maximum up to 4mg, which was dissolved in few drops of HPLC grade acetone and make up to the mark of standard volumetric flask with HPLC grade hexane. Standard stock and working standard solution were stored in deep freezer at -20°C. 76 pesticides mixture (Organochlorine, Synthetic Pyrethroids and Herbicides) which are commonly used in India viz Propoxur, 3,4-Dichloraniline, alpha-BHC, Pencycuron, Phorate, Thiometon, Dimethoate, Dazomet, Simazine, Atrazine, beta-BHC, Propetamphos, delta-BHC, Chlorothalonil, Paraoxon Methyl, Etrimfos, gamma-BHC (Lindane), Iprobenfos, Propanil, Metribuzin, Malaaxon, Alachlor, Transfluthrin, Metalaxyl (Mefenoxam), Pirimiphos-methyl, Linuron, Malathion, Metolachlor (S-Metolachlor), Chlorpyrifos, Fenthion, Flufenacet, Pendimethalin, Fipronil, Heptachlor-exo-epoxide, Chlorfenvinfos, Captan, o,p'-DDE, Butachlor, Endosulfan-I, Hexaconazole, Fipronilsulfone, Profenofos, p,p'-DDE, Myclobutanil, Oxyfluorfen, o,p'-DDD, Buprofezin, Kresoxim-methyl, Chlorfenapyr, Ethion, Triazophos, Benalaxyl, Propiconazole-1, Endosulfan sulfate, Propiconazole-2, p,p'-DDT, Diclofop-methyl, Propargite-1, Propargite-2, Etoxazole, Fenpropathrin, Fenazaquin, Phosalone, Pyriproxyfen, L-Cyhalothrin, Permethrin-1, Permethrin-2, Cyfluthrin-1, Cyfluthrin-2, Cyfluthrin-3, Cyfluthrin-4, Cypermethrin-1, Etofenprox, Fenvalerate-1, Fenvalerate-2 (Esfenvalerate), Deltamethrin-1 (Tralomethrin deg.-1). standard solution were prepared at six different concentration levels of **10, 25, 50, 100, 250, 500 ppb** all gave good response for MS/MS detector were considered for study.

### Linearity

The calibration graph was plotted with spiked samples (matrix match) of six different concentrations of standard mixture solutions. The mixed standard stock solution used for spiking soil samples was prepared with acetone and n hexane. The standard mixtures were analyzed by GCMS/MS at each concentration level. Calibration curve were plotted at six concentration levels with correlation co-efficient ( $r^2$ ) > 0.99.

### Sample Analysis as per Following Steps

- Preparation of calibration solutions and injection to GCMSMS.
- Analysis of prepared soil samples.
- Calculation of residue content by linearity graph.
- Injection of Reagent Blank and solvent blank sample to prevent false positive reporting

### RESULT AND DISCUSSION

The calibration graph was plotted with spiked samples (matrix match) of six different concentrations of standard mixture solutions. The mixed standard stock solution used for spiking soil samples was prepared with acetone and n hexane. The standard mixtures were analyzed by GCMS/MS at each concentration level. Calibration curve were plotted at six concentration levels with correlation co-efficient ( $r^2$ ) > 0.99. Chromatograms were obtained for soil samples of all five villages are mentioned following.

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Analyzed       : 4/4/2022 11:46:05 PM
Sample Type    : Unknown
Level #       : 1
Sample Name    : SOIL_52_A
Sample ID     :
Vial #        : 8
Injection Volume : 1.00
Data File     : F:\IPFT-AD-PC-001-GC MS MS\Data\Non GLP\2022\SOIL_52_A_442022_9.qgd
Method File   : F:\IPFT-AD-PC-001-GC MS MS\Data\Non GLP\2022\MRM SCOPE 2022.qgm
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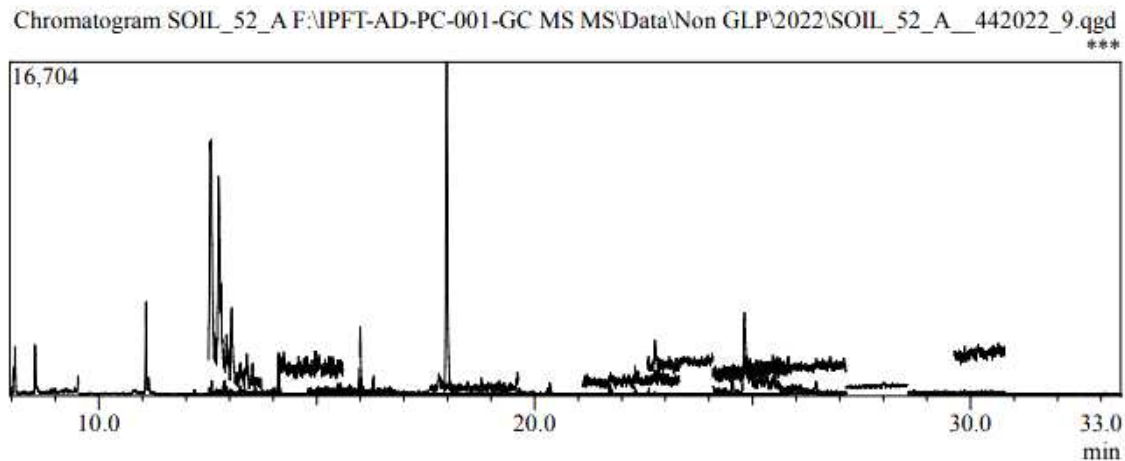


Figure 2: Village Kakara Soil Analysis Results.

Analyzed : 4/5/2022 12:33:13 AM  
 Sample Type : Unknown  
 Level # : 1  
 Sample Name : SOIL\_52\_B  
 Sample ID :  
 Vial # : 9  
 Injection Volume : 1.00  
 Data File : F:\PFT-AD-PC-001-GC MS MS\Data\Non GLP\2022\SOIL\_52\_B\_442022\_10.qgd  
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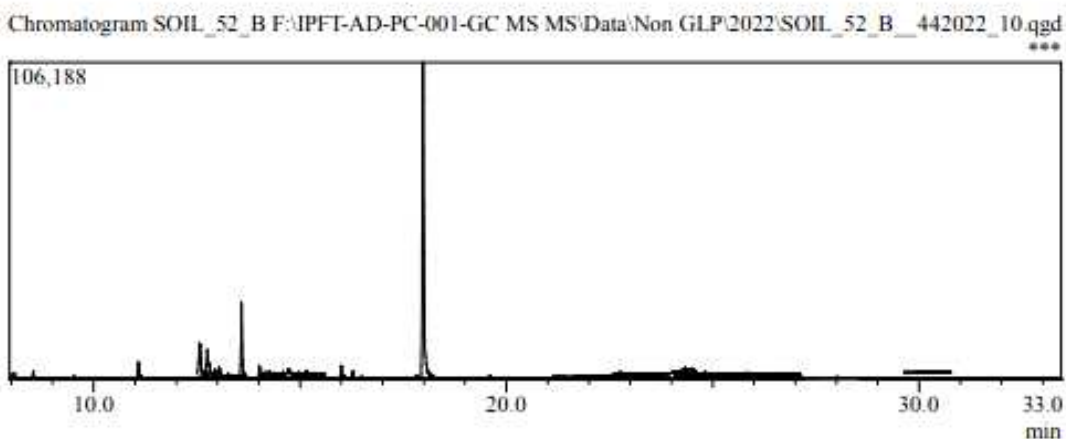


Figure 3: Village Jeetnagar Soil Analysis Results

Analyzed : 4/5/2022 1:20:13 AM  
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 Sample Name : SOIL\_52\_C  
 Sample ID :  
 Vial # : 10  
 Injection Volume : 1.00  
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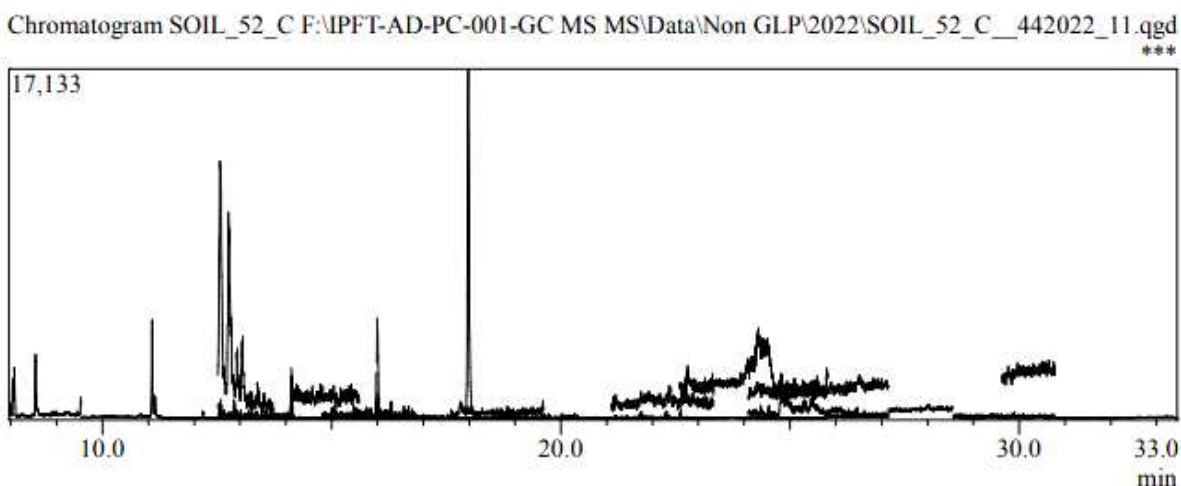
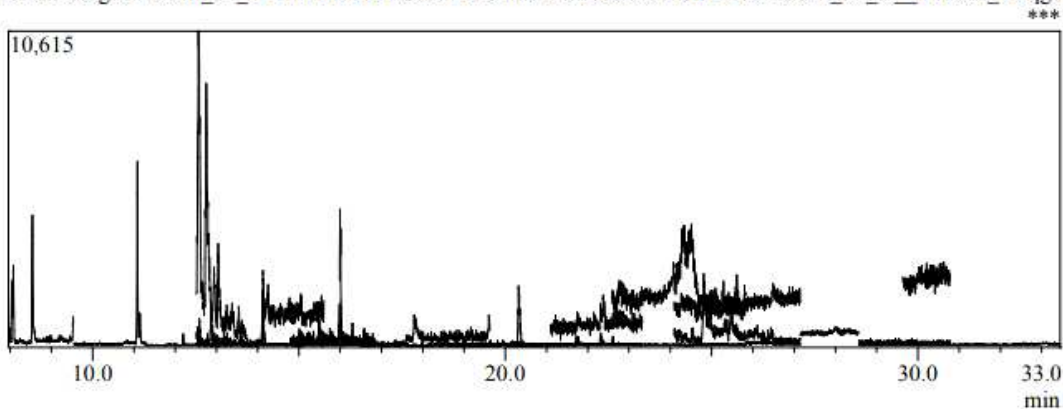


Figure 4: Village Podi Soil Analysis Results.



Analyzed : 4/5/2022 2:07:21 AM  
 Sample Type : Unknown  
 Level # : 1  
 Sample Name : SOIL\_52\_D  
 Sample ID :  
 Vial # : 11  
 Injection Volume : 1.00  
 Data File : F:\IPFT-AD-PC-001-GC MS MS\Data\Non GLP\2022\SOIL\_52\_D\_442022\_12.qgd  
 Method File : F:\IPFT-AD-PC-001-GC MS MS\Data\Non GLP\2022\MRM SCOPE 2022.qgm  
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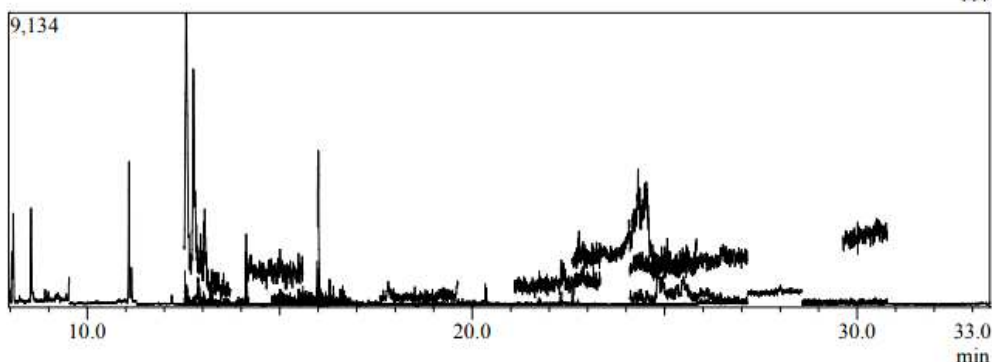
Chromatogram SOIL\_52\_D F:\IPFT-AD-PC-001-GC MS MS\Data\Non GLP\2022\SOIL\_52\_D\_442022\_12.qgd



**Figure 5: Village Bhadanpur Soil Analysis Results.**

Analyzed : 4/5/2022 2:54:19 AM  
 Sample Type : Unknown  
 Level # : 1  
 Sample Name : SOIL\_52\_E  
 Sample ID :  
 Vial # : 12  
 Injection Volume : 1.00  
 Data File : F:\IPFT-AD-PC-001-GC MS MS\Data\Non GLP\2022\SOIL\_52\_E\_442022\_13.qgd  
 Method File : F:\IPFT-AD-PC-001-GC MS MS\Data\Non GLP\2022\MRM SCOPE 2022.qgm  
 Tuning File : C:\GCMSsolution\System\Tune1\TUNING\_NS\_WITH COLUMN\_FLM 2\_CID ON-OFF\_04042022\_2.qgt  
 [Comment] :

Chromatogram SOIL\_52\_E F:\IPFT-AD-PC-001-GC MS MS\Data\Non GLP\2022\SOIL\_52\_E\_442022\_13.qgd



**Figure 6: Village Kauseri Soil Analysis Results.**

From the above five chromatogram obtained from soil analysis of soil samples of villages Kakara, Jeetnagar, Podi, Bhadanpur, Kuseri. Although study was done for different pesticidal residue but only pendimethalin was detected in three villages Kakara, Jeetnagar, Podi while in Bhadanpur and Kuseri no pesticidal residues were obtained above detectable limit. Concentrations of pendimethalin were obtained for Kakara 21.08ug/kg, Jeetnagar 54.34ug/kg, Podi 20.93ug/kg.

## CONCLUSION

People of Maihar region of Satna district are using different chemicals, pesticides, fertilizers to increase yield of crops. Present study was done only on amount of pesticidal residue present in soil of selected villages of Maihar region. Use of pesticide is common in all agricultural area and excessive amount of pesticide accumulated in soil but most of pesticides are biodegradable and no residues are obtained for such pesticides. In selected villages only pendimethalin was obtained in villages Kakara, Jeetnagar and Podi because of excessive use of pendimethalin in these villages. Except this pedimethalin is slowly biodegradable pesticide so their concentration can increase if excessive repeat use done in agricultural field. In spite of detection of pendimethalin in villages Kakara, Jeetnagar and Podi it is not very dangerous level because LD<sub>50</sub> of pendimethalin is 5000mg/kg.

## DISCLAIMER

The authors alone are responsible for the content and writing of the paper.

## REFERENCES

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