PARIPEX - INDIAN JOURNAL OF RESEARCH | Volume - 11 | Issue - 08 |August - 2022 | PRINT ISSN No. 2250 - 1991 | DOI : 10.36106/paripex

journal or A O	RIGINAL RESEARCH PAPER	Chemistry
MI MI	JDY OF RESIDUAL PESTICIDES IN SOIL OF LIHAR REGION OF SATNA DISTRICT IN LIHYA PRADESH	<b>KEY WORDS:</b> Pendimethalin, pesticidal residue, biodegradability, herbicide
Asha Kalwani	Department of Chemistry, Govt. Girl P.G. College	e,Satna-485001(M.P.) India.
Neelam		

# Neelam Richhariya

 $Department of Chemistry, Govt. Girl P.G. College, Satna-485001 (M.P.) \ India.$ 

In this study, some villages were selected for evaluation of pesticidal residue where excessive use of pesticides take place. Collected soil samples were analysed for various pesticidal residue. it was observed that only pendimethalin was detected in soil of Olea, Harnampur, and Berma in very small quantities. Residue of pendimethalin was obtained because pendimethalin is a slow biodegradable herbicide. Excessive use of pendimethalin may cause increment in residual concentration and can pose adverse effect. Other pesticides were not detected due to teither their good biodegradability or their fewer use.

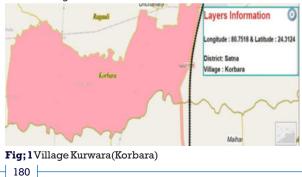
### INTRODUCTION

Maihar is a Tehsil of Satna district in the state of Madhya Pradesh, India. Maihar is known for the temple of the revered Mother Goddess Sharda Maa situated on Trikuta hill. In Maihar region main living hood of people is agriculture [1]. To produce high yield of crops farmers are using different type of pesticides[2] and repeated use of these pesticides are accumulated in soil and cause adverse effect when reach at high concentration. Due to this analysis of soil is necessary in a specific time for pesticidal residue in agricultural field [3,4]. Literature reveals that no study has been done for pesticidal residue in maihar region of Satna district. On this basis some villages were selected where farming is done by modern techniques for pesticidal residue in soil. The selected villages were Kurwara, Olea, Guraeya, Harnampur and Berma. Soil samples were collected from these villages and analyzed for various pesticidal residue. It was observed that in soil of villages Olea, Harnampur and Berma except pendimethalin no other pesticidal residues were obtained. It is due to excessive use of herbicide pendimethalin in this area. In other two villages no pesticidal residue was obtained above detectable limit. A door to door survey was done in villages Olea, Harnampur and Berma for health status of people who were using pendimethalin in their agriculture field. It was also found that no hazardous effect was observed because concentration of pendimethalin was not so high to cause serious bad effect.

# MATERIAL AND METHODS:

# **Collection Of Samples From Selected Sites At Maihar Region**

Samples of soils were collected from Kurwara, Olea, Guraeya, Harnampur and Berma villages of Maihar Tehsil of Satna district. About<sup>1</sup>/<sub>2</sub> 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 <sup>1</sup>/<sub>2</sub> 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.



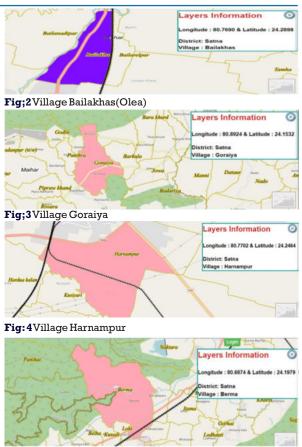


Fig.5 Village Berma

# Chemicals

Anhydrous sodium sulfate (Na2SO4, 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-Dichloranilline, 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,

www.worldwidejournals.com

#### PARIPEX - INDIAN JOURNAL OF RESEARCH | Volume - 11 | Issue - 08 | August - 2022 | PRINT ISSN No. 2250 - 1991 | DOI : 10.36106/paripex

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 (Na2SO4), pre washed with 20 ml methanol to reduce the absorption of pesticides in the sample during clean up. All the filtrate was collected into100 mL flat bottom round flask. Anhydrous sodium sulphate (Na2SO4) 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 2mL 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-Dichloranilline, 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-exoepoxide, 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 corelation co-efficient (r 2) & gt;0.99.

Sample Analysis as per following steps-I. Preparation of callibration solutions and injection to GCMSMS.

ii. Analysis of prepared soil samples.

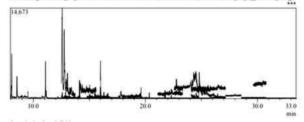
iii. Calculation of residue content by linearity graph.

iv. Injection of Reagent Blank and solvent blank sample to prevent false positive reporting

### **RESULTS AND DISCUSSION:**

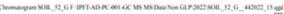
The calibration graph was plotted with spiked samples (matrix match) of six different concentrations of standard mixture solutions. The mixed standard stock solution used for spiking soil samples was prepared with acetone and n hexane. The standard mixtures were analyzed by GCMS/MS at each concentration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

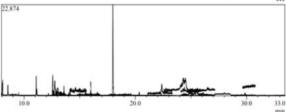
Chromatogram SOIL\_52\_F F /IPFT-AD-PC-001-GC MS MS/Data/Non GLP/2022/SOIL\_52\_F\_442022\_14.gpd



#### Fig: 6 Village Kurwara (Korbara) Soil Analysis Results

Analyzed	4/5/2022 4 28 18 AM
Sample Type	Unknown
Level #	-1
Sample Name	SOIL 52 G
Sample ID	
Vial #	14
Injection Volume	1.00
Data File	F IPFT-AD-PC-001-GC MS MS Data/Non GLP 2022 SOIL 52 G 442022 15 apd
Method File	F-IPFT-AD-PC-001-GC MS MS Data Non GLP 2022 MRM SCOPE 2022 ggm
Tuning File	C GCMSsolution System Tunel TUNING NS WITH COLUMN FLM 2 CID ON-OFF 04042022 2 gg
(Comment)	
	C:GCMSsolution/System/Tunel/TUNING_NS_WITH COLUMN_FLM 2_CID ON-OFF_04042022_2.0

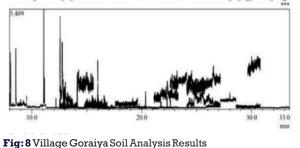




#### Fig: 7 Village Bailakhas (Olea) Soil Analysis Results

Analyzed	4/5/2022 5/15/18 AM
Sample Type	Unknown
Level#	
Sample Name	SOIL 52 H
Sample ID	
Vial #	15
Injection Volume	1.00
Data File	F IPFT-AD-PC-001-GC MS MS Data/Non GLP 2022/SOIL 52/11 442022/16 ggd
Method File	F IPFT-AD-PC-001-GC MS MS Data Non GLP 2022 MRM SCOPE 2022 apr
Tuning File	C GCMSsolution System Tunel TUNING NS_WITH COLUMN_FLM 2_CID ON-OFF_04042022_2.ggt
[Comment]	

Chromatogram SOIL\_52\_H F /IPFT-AD-PC-001-GC MS MS/Data/Non GLP/2022/SOIL\_52\_H \_\_442022\_16 qgd



181

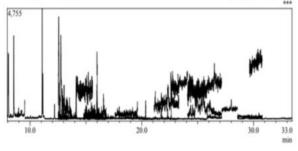
www.worldwidejournals.com

# PARIPEX - INDIAN JOURNAL OF RESEARCH | Volume - 11 | Issue - 08 | August - 2022 | PRINT ISSN No. 2250 - 1991 | DOI : 10.36106/paripex

Analyzed	: 4/5/2022 6:02:11 AM
Sample Type	Unknown
Level #	1 Contractor
Sample Name	:SOIL 52 1
Sample ID	
Vial #	16
Injection Volume	:1.00
Data File	: F/IPFT-AD-PC-001-GC MS MS/Data/Non/GLP/2022/SOIL 52 1 442022 17 and
Method File	F-IPFT-AD-PC-001-GC MS MS Data/Non GLP 2022/MRM SCOPE 2022.cgm
Tuning File	COCMStolution/System/Tunel/TUNING NS WITH COLUMN FLM 2 CID ON-OFF 04042022 2.egt
[Comment]	

- benign synthesis of some novel biologically active 7-hydroxy-4-methyl coumarin derivatives, Current Research in Green and Sustainable Chemistry, 5, 100260,
- P Singh, S Sahu, PK Singh, N Singh (2018) Study of Association of Soil Parameters with Various Density Classes of Forests of Mukundpur, Satna, Forest Division and Madhya Pradesh. J Soil Sci Plant Health 2.
- 4- B. Gevao, K.T. Semple, K.C. Jones, Bound pesticide residues in soils: a review, Environmental Pollution, 108, 1,2000, 3-14

Chromatogram SOIL\_52\_1 F:/IPFT-AD-PC-001-GC MS MS/Data/Non GLP/2022/SOIL\_52\_1\_442022\_17.ggd



### Fig: 9 Village Harnampur Soil Analysis Results

Sample Type	Unknown
Level #	1 million 1
Sample Name	SOIL_52_3
Sample ID	
Vial #	17
Injection Volume	1.00
Data File	F :IPFT-AD-PC-001-GC MS MS Data/Non GLP 2022 SOIL_52_J_442022_18 qpd
Method File	F/IPFT-AD-PC-001-GC MS MS Data/Non GLP/2022 MRM SCOPE 2022 gpm
Tuning File	C GCMSsolution/System/Tunel/TUNING_NS_WITH COLUMN_FLM 2_CID ON-OFF_04042022_2.ggt
[Comment]	

Chromatogram SOIL\_52\_J F:IPFT-AD-PC-001-GC MS MS/Data/Non GLP:2022/SOIL\_52\_J\_442022\_18.qpd

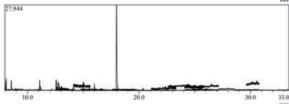


Fig: 10 Village Berma soil analysis results

Above fig.6-10 chromatogram obtained from soil analysis of soil samples of villages Kurwara, Olea, Guraeya, Harnampur and Berma exhibited that no any pesticidal residue above detectable limit except pendimetalin. Pendimethalin detected in villages Olea(23.45ug/kg), Harnampur(15.44ug/ kg) and Berma(26.04ug/kg) in low concentrations and no pesticidal residue was obtained in Kurwara and Guraeya. This analysis work was done for many pesticides which were being used in this area but only pendimethalin was obtained as residue. Door to door survey in these villages was resulted no adverse effect.

### CONCLUSION:

In present analysis work soil samples from selected villages villages Kurwara, Olea, Guraeya, Harnampur and Berma were analysed for pesticidal residues. It was observed only pedimethalin was detected in village Olea, Harnampur and Berma. Due to very low concentration of residual pendimethalin in soil of above three village no adverse effect observed when door to door survey has been done. Pendimethalin is slow biodegradable herbicides and due to this reason it was accumulated more than easily biodegradable pesticides. Excessive use of pendimethalin in this area may increase amount of residue and can cause harmful impact.

#### Disclaimer

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

## **REFERENCES:**

- Arvind Prasad Dwivedi(2016). Physico-Chemical Characteristics of Soil in three Different Areas of Maihar City, International Journal of Advanced Research in Chemical Science, 3(9), 37-48.
  Shailendra Yadav, Sushma Singh, Chitrasen Gupta (2022) Environmental