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Phytoremediation Potentials of Sunflower (*Helianthus annuus*) on Soil Contaminated with Organochlorine Pesticide Residue

S Garba and L Dauda

Department of Chemistry Nigerian Defence Academy Kaduna, Nigeria.

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Abstract: The physicochemical properties of IAR soil and the phytoremediation potential of *Helianthus annuus* (Sunflower) plant on organochlorine pesticide residue were determined using standard methods. Results of the physicochemical analyses showed that IAR soil had a pH range of 4.73 – 4.93, organic matter content range of 0.71 – 2.39 and particle size range of: 10% - 12% of clay, 42% - 46% of silt and 44% - 46% of sand. Results of the absorption of organochlorine pesticide residue showed that Sunflower absorbed a mean concentration of 3.4556 ± 3.0174 mg/kg of alpha-BHC, 0.4873 ± 0.3638 mg/kg of beta-BHC, 3.4884 ± 6.0400 mg/kg of Heptachlor, 2.2781 ± 1.86238 mg/kg of Aldrin, 1.4494 ± 1.3549 mg/kg of gamma-BHC, 6.7012 ± 5.7216 mg/kg of delta-BHC, 37.3056 ± 19.9992 mg/kg of Heptachlor-epoxide, 88.8459 ± 72.9678 mg/kg of Endosulfan I, 5236.2737 ± 0 mg/kg of Dieldrin, 0.9224 ± 0 mg/kg of Endrin, 21.7438 ± 21.4416 mg/kg of p,p' DDD, 12.5867 ± 0 mg/kg of Endosulfan II, 13.0872 ± 12.9082 mg/kg of p,p'-DDT, 1858.8949 ± 1816.4019 mg/kg of Endrin Aldehyde, 852.0080 ± 1147.7685 mg/kg of Endosulfan sulphate and 16.5957 ± 19.2893 mg/kg of Methoxychlor. The results of this work showed that sunflower could be used for the absorption of organochlorine pesticides residue especially on loamy soils.

Keywords: Organochlorine, Sunflower, Deionized water

INTRODUCTION

One major productivity enhancing practice among farmers in Nigeria is the use of chemicals (pesticides, herbicides and fertilizers) to enhance production and productivity in agriculture,¹

Pesticides are substances or organisms designed to kill or control pests like insects, fungi, weeds, or rodents. Organochlorine pesticides (OCPs) have been widely used throughout the world since the middle of the 20th century and have been used globally, mostly in public health activities and more so in an attempt to combat vector borne diseases in agriculture and animal production². OCPs are known to be toxic to humans, animals and most aquatic life. Significant exposure occurs through the consumption of contaminated agricultural products containing residues of these pesticides or their degradation products³. A simple method, therefore, to remove, detoxify or immobilize environmental contaminants especially organochlorine pesticides residue in a growth matrix (Soil, Water or Sediments) through the natural, biological, chemical or physical activities and processes of the plants⁴ is known as phytoremediation. Phytoremediation can be used to remove metals (e.g. Ag, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, and Zn), radionuclides (e.g. ⁹⁰Sr, ¹³⁷Cs, ²³⁹Pu, ²³⁴U, ²³⁸U) and certain organic compounds⁵.

Sunflower plant - a fast-growing, deep-rooted industrial oil crop with a high biomass, also known for its ability to remove heavy metals such as zinc or copper as well as several radionuclides from contaminated environment⁶⁻⁸ was used as the phytoremediator of organochlorine pesticides residue on loamy soil collected from Institute for Agricultural Research (IAR) farm located within Zaria metropolis.

EXPERIMENTAL

Sampling location and collection of sample: The soil sample was collected from a research farm of the Institute for Agricultural Research (IAR) located within Zaria metropolis of Kaduna state, Nigeria. A standard stainless steel soil auger was used to take topsoil and subsoil samples at a depth of 20 cm and 50 cm respectively. The soil sample was collected at different strategic locations of the farm, then mixed together to give a composite sample.

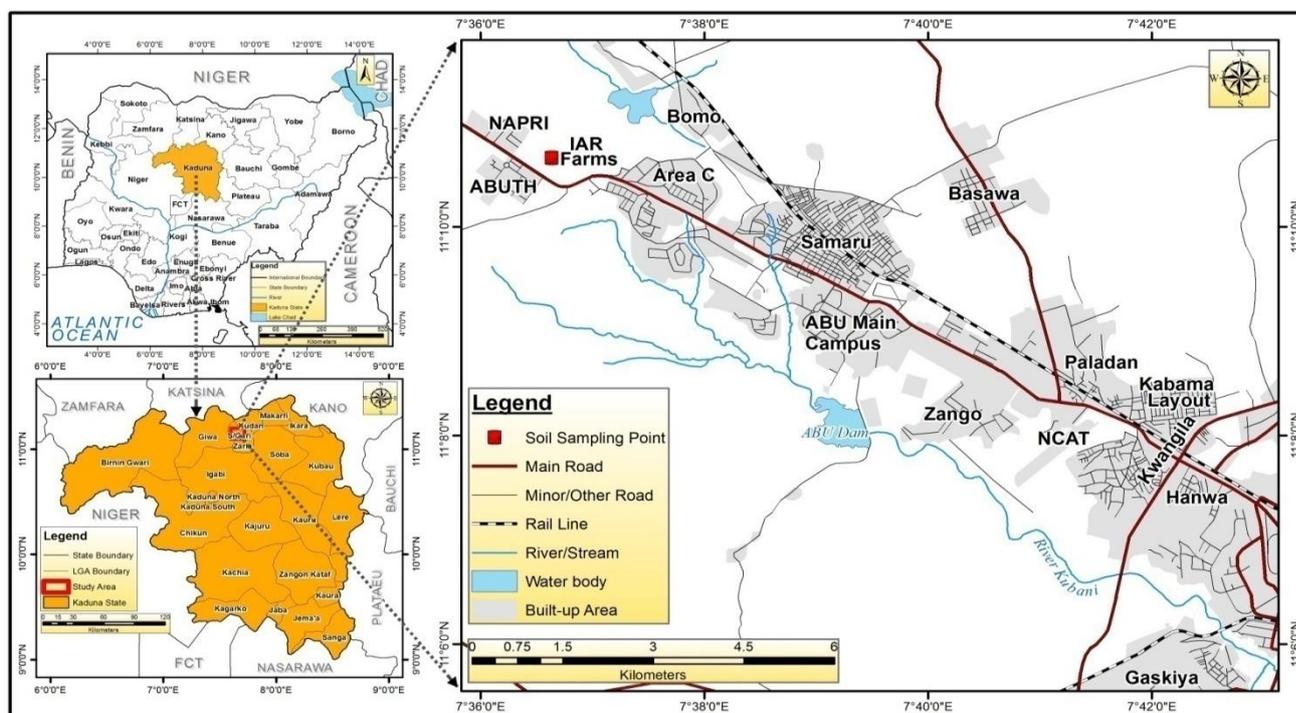


Figure 1: Map of Zaria showing Soil Sampling Point

Source: Modified from the Administrative Map of Sabon Gari L.G.A.

Sample Treatment/Preservation: A part of the composite soil sample collected (about 30 g) was air-dried in the laboratory for 1 week, ground in agate mortar and sieved through a 2 mm mesh, then stored in black polythene bag prior analysis. The remaining soil sample was divided and spiked with standard concentrations of organochlorine pesticides (1% and 2%). The Sunflower seeds were planted for a period of 30 days before harvesting. After harvesting, the soil was treated as above, while the roots and shoots were air-dried and ground to powder separately prior analysis.

Analyses of Physicochemical Properties

Determination of soil particle size: The hydrometer method of particle size analysis of soil as described by Agbenin⁹ was used. A weighed quantity (50 g) of 2 mm sieved soil sample was transferred into a 600 cm³ beaker. 25 cm³ of Calgon and 400 cm³ of tap water were added then the mixture was transferred into high speed stirrer container and stirred for 15 minutes. The soil suspension was then transferred to a 1L capacity cylinder and diluted to mark and then stirred with a glass stirring rod. The hydrometer was then inserted and readings were taken at the following time intervals (Note: hydrometer was inserted 20 seconds before the time of reading was due)

4: 48 minutes = silt and clay (<0.002mm)

5 hours = clay (< 0.002mm)

The temperature of the soil suspension after each reading was taken and the following calculations were made using the formulae below

$$\text{Clay (g Kg}^{-1}\text{)} = \frac{H \text{ (gL}^{-1}\text{)} \times 1000}{W \text{ (g)}} - 1$$

(Where 1 is the calgon correction factor)

$$\text{Silt + Clay (g Kg}^{-1}\text{)} = \frac{M \text{ (gL}^{-1}\text{)} \times 1000}{W \text{ (g)}} - 1$$

$$\text{Silt (g Kg}^{-1}\text{)} = \{\text{Silt + Clay}\} - \text{Clay}$$

$$\text{Total sand (g Kg}^{-1}\text{)} = 1000 - (\text{Silt} + \text{Clay})$$

From the above,

$$\text{Weight of soil (oven-dry basis)} = W \text{ (g)}$$

$$\text{Hydrometer reading after 4min 48 seconds} = M \text{ (gL}^{-1}\text{)}$$

$$\text{Hydrometer reading after 5 hours} = H \text{ (gL}^{-1}\text{)}$$

Determination of soil pH: Soil pH was determined using a pH meter in a 0.01M CaCl₂ solution as described by Agbenin⁹. An amount (10 g) of 2 mm sieved soil was weighed into a 50 cm³ beaker. 20 cm³ of 0.01M CaCl₂ solution was added and stirred. The suspension was allowed to stand for 30 minutes with occasional stirring. Before the pH of the suspension was taken, the pH meter was pre calibrated using 4.0 and 7.0 pH buffers at the same temperature with the suspension. A glass electrode was used (the electrode was not allowed to reach the bottom of the container) to take pH readings by the pH meter after every 30 seconds. Readings were made in triplicate to get the average readings

Determination of Organic matter content: Soil organic matter content was determined in the soil sample following the procedure given by Ryan *et al.*¹⁰. A portion (1 g) of soil sample was weighed and transferred into a 500 cm³ beaker. A volume of 0.5M potassium dichromate solution (10 cm³) and 20 cm³ concentrated sulphuric acid were added separately. The suspension was mixed thoroughly and allowed to stand for 30 minutes. Deionized water (200 cm³) and 10 cm³ of concentrated orthophosphoric acid were added to the suspension. The mixture was allowed to cool for 10 minutes then 10 – 15 drops of diphenylamine indicator was added and the suspension was thoroughly mixed using magnetic stirrer. The resulting solution (25 cm³) was pipetted and transferred into a conical flask and was titrated against a 0.5M ferrous ammonium sulphate solution, until the colour of the suspension changed from violet blue to green. A blank titration was carried out using the same procedure as above excluding the soil sample. The titration was repeated thrice and the organic matter content was determined using the following formulae:

$$\% \text{ oxidizable organic carbon (w/w)} = \frac{[V_{\text{blank}} - V_{\text{sample}}] \times M}{\text{Soil dry weight (g)}}$$

$$\% \text{ Total organic carbon (w/w)} = 1.334 \times \% \text{ Oxidizable organic carbon}$$

$$\% \text{ Organic Matter (w/w)} = 1.724 \times \% \text{ Total Organic Carbon}$$

Where M is molarity of ferrous ammonium sulphate; V_{blank} is volume of ferrous ammonium sulphate in blank; V_{sample} is volume of ferrous ammonium sulphate in sample.

Sample Extraction and Clean-up

Extraction of Organochlorine Residues from Soil Sample: The organochlorine pesticide residue was extracted from the soil sample before planting and after harvesting by a method described by Ize-Iyamu *et al.*¹¹, using a Soxhlet extractor. A portion (10 g) of each soil sample (before planting and after harvesting) and 20 g of anhydrous sodium sulphate were ground into dry powder. The ground sample was extracted with 150 cm³ of a mixture of Acetone and n-Hexane (2:1) using a Soxhlet extractor. After extraction, the extract was transferred into a round bottomed flask connected to a pre-weighed receiver through a Liebig condenser and concentrated to about 20cm³ on a water bath maintained between 50 and 55°C. The remaining solvent in the concentrated extract was evaporated to about 5 cm³ using a rotary evaporator.

The same procedure was repeated using plant sample (roots and shoots respectively)

Sample Clean-up: The different soil and plant extracts were cleaned up as described by Ize-Iyamu *et al.*¹¹, using 2 g of activated silica gel which was packed in a column of about 1 cm internal diameter and approximately 10 cm long. The column was loaded with 10 cm³ n-Hexane, while the sample extract was dissolved in 5 cm³ of n-Hexane before it was loaded also into the column. Elution of each of the sample was done with 50 cm³ of ethyl-acetate and n-Hexane mixture (9:1). The eluents were then concentrated on a rotary evaporator at about 45 °C. The almost-dry concentrates were dissolved in 2 cm³ Acetone and transferred into vials for subsequent injection into the Gas Chromatograph.

Sample analysis for organochlorine pesticide residue: Organochlorine pesticide residues were analyzed by Gas Chromatography with the ⁶³Ni selective electron-capture detector. The column used was DB-17 (30m x 250um x 0.25um). The carrier gas and makeup gas were Helium and Nitrogen respectively with a 2.0 mL /min flow rate, employing the split less mode. 1.0µL of the final extract

was injected at a temperature of 250 °C. The oven temperature was kept at 150 °C at the initial then it was increased to 280 °C at a rate of 6 °C/ min. The total run time was 21.67 minutes. The detector was maintained at 290 °C, peak identification was performed by the GC-ECD software (Agilent 7890A GC-ECD).

RESULTS AND DISCUSSION

The soil particle size distribution as shown in **Table 1** showed that the soil analysed was basically a loamy soil due to higher percentage of sand content as compared to silt and clay. IAR soil had an average of 45.33% sand content, with 44.00% of silt and 10.67% of clay. Soils having high sand content are reported to facilitate leaching and removal of pesticide residues from surface soils ¹². The soil sample had a mean pH of 4.81. This pH value lies within the recommended range for proper growth and efficient uptake of nutrients and compounds from soil. The soil had relatively moderate organic matter content, a mean of 1.27. Soil pH, organic matter and clay contents are known to influence the dynamics and behavior of both inorganic and organic pollutants in soil¹³⁻¹⁵.

Table 1: Results of the physicochemical properties of the IAR soil

	pH	Mean ± SD	Organic Matter (%)	Mean ± SD	Clay (%)	Mean ± SD	Silt (%)	Mean ± SD	Sand (%)	Mean ± SD
Ic	4.76	4.81	0.71	1.27	12.00	10.67	42.00	44.00	46.00	45.33
Farms	4.93	±	0.71	±	10.00	±	44.00	±	46.00	±
	4.73	0.09	2.39	0.79	10.00	0.94	46.00	1.63	44.00	0.94

Ic = IAR Farms

The concentration of organochlorine pesticide residue absorbed by the plant (**Table 2**) showed that 3.4556 ± 3.0174 mg/kg, 0.4873 ± 0.3638 mg/kg, 1.4494 ± 1.3549 mg/kg and 6.7012 ± 5.7216 mg/kg of α -BHC, β -BHC, γ -BHC and δ -BHC respectively was absorbed. The detection of the isomers of Benzene hexachloride may be due to its use by farmers as a fungicide on seed grains such as wheat, barley and oats¹⁶ or its occurrence as an impurity in other fungicides at low levels ¹⁷. The plant also absorbed 3.4884 ± 6.0400 mg/kg, 37.3056 ± 19.9992 mg/kg and 16.5957 ± 19.2893 mg/kg of Heptachlor, Heptachlor epoxide and Methoxychlor respectively. The presence of Heptachlor with its epoxide could be attributed to its use by farmers to control ants, cutworms, maggots, termites, thrips, weevil wireworms and many other insect pests in both cultivated and uncultivated soils. It also controls insects and pests of humans and domestic animals¹⁸. Heptachlor epoxide had higher concentrations than Heptachlor in the Sunflower plant probably because Heptachlor is moderately persistent in soil where it is mainly transformed into its epoxide. Aldrin was detected at a concentration of 2.2781 ± 1.86238 mg/kg. Dieldrin however had a concentration of 5236.2737 ± 0 mg/kg. Aldrin and Dieldrin are highly effective insecticides for soil dwelling pests and for the protection of wooden structures against termites and wood borers. Sunlight and bacteria can change Aldrin into Dieldrin¹⁹ most probably the reason for its higher concentration than Aldrin. Central nervous system excitation is the primary adverse effect in humans in cases of Aldrin or Dieldrin intoxication. Other symptoms include: headaches, dizziness, general malaise, and muscle twitching etc ¹⁹. Sunflower had a concentration of 0.9224 ± 0 mg/kg of Endrin and 1858.8949 ± 1816.4019 mg/kg of Endrin Aldehyde. Endrin is basically used as a rodenticide. Endrin and its metabolite Endrin Aldehyde are lipophilic thus tend to accumulate in fatty tissues of organisms living in water. Its acute poisoning in humans affects primarily the nervous system.

A concentration of 21.7438 ± 21.4416 mg/kg of p,p DDD and 13.0872 ± 12.9082 mg/kg of p,p DDT were detected respectively. p,p DDE however was not detected. DDT is known to undergo metabolic conversion and dehydrochlorination. Presence of metabolites of DDT which is DDD and DDE encountered in this study is probably due to such metabolic processes²⁰. Also, its high persistence in the environment is a likely reason for the presence of DDT and its metabolites. The soil half-life for DDT is from 2 to 15 years²¹. Endosulfan I, Endosulfan II and Endosulfan sulfate were detected in Sunflower in high concentrations because the pesticide spiked to the experimental soils contained high percentage (35%) of Endosulfan. 88.8459 ± 72.9678 mg/kg, 12.5867 ± 0 mg/kg and 852.0080 ± 1147.7685 mg/kg concentrations of Endosulfan I, Endosulfan II and Endosulfan sulfate respectively were detected. Endosulfan which is marketed under various trade names such as Thionex 35EC, Endocel 35EC is a broad spectrum insecticide acting as a contact and stomach poison used for the control of bean aphids and thrips and is now commonly used by subsistence farmers in Nigeria²². Sunflower had a higher concentration of Endosulfan I than Endosulfan II, a probable reason being that Endosulfan II is thermally unstable and may be slowly converted to Endosulfan I in the environment.

Table 2: Results of the mean concentration (mg/Kg) of organochlorine pesticide residue absorbed by Sunflower

Organochlorine Compound	Mean \pm SD (mg/kg)
α -BHC	3.4556 ± 3.0174
β -BHC	0.4873 ± 0.3638
Heptachlor	3.4884 ± 6.0400
Aldrin	2.2781 ± 1.86238
γ -BHC	1.4494 ± 1.3549
δ -BHC	6.7012 ± 5.7216
Heptachlor Epoxide	37.3056 ± 19.9992
Endosulfan I	88.8459 ± 72.9678
PP DDE	0 ± 0
Dieldrin	5236.2737 ± 0
Endrin	0.9224 ± 0
PP DDD	21.7438 ± 21.4416
Endosulfan II	12.5867 ± 0
PP DDT	13.0872 ± 12.9082
Endrin Aldehyde	1858.8949 ± 1816.4019
Endosulfan Sulfate	852.0080 ± 1147.7685
Methoxychlor	16.5957 ± 19.2893

ND = Not detected (below instrument detection limit)

From **Table 3** below, Raw IAR soil had the highest concentrations of Endosulfan sulfate and dieldrin with 184.2944 ± 0.0047 mg/kg and 122.6104 ± 0.0902 mg/kg concentrations while β -BHC and γ -BHC had the lowest concentrations of 0.0106 ± 0.0018 mg/kg and 0.1294 ± 0.0012 mg/kg. Endosulfan II however was not detected in the raw IAR soil. The high concentrations of Endosulfan sulfate in the soil, is evidence of its continuous use by farmers in Nigeria²². After planting, spiking and harvesting, the soil was found to have high concentrations of γ -BHC and Endosulfan sulfate with concentrations of 10.8352 ± 0.1157 mg/kg and 9.5414 ± 0.0206 mg/kg. While the lowest

concentrations were detected for Endrin with a concentration of 0.0594 ± 0.0035 mg/kg and p,p DDD with a concentration of 0.0563 ± 0.0009 mg/kg. Dieldrin, δ -BHC and p,p DDE were however, not detected. The IAR soil has a moderate retentive capacity. This is probably due to its clay content. Soil clay contents play very important role in the retention or accumulation of organochlorine pesticides. Organochlorine pesticides are known to bind with clay to form bound residues²³.

Table 3: Results of the concentration of organochlorine pesticide residue present in the soil before planting and that retained in the soil after harvesting

Organochlorine Pesticide residue	Soil before planting	Soil after planting, spiking and harvesting
	Ic (mg/kg)	Ic (mg/kg)
α -BHC	0.1804 ± 0.0045	0.9245 ± 0.0032
β -BHC	0.0106 ± 0.0018	1.3543 ± 0.0204
Heptachlor	0.0355 ± 0.0025	0.4868 ± 0.0040
Aldrin	0.3239 ± 0.0099	1.8406 ± 0.0047
γ -BHC	0.1294 ± 0.0012	10.8352 ± 0.1157
δ -BHC	0.6297 ± 0.0006	0.0000 ± 0.0000
Heptachor Epoxide	1.6242 ± 0.0179	0.2509 ± 0.0007
Endosulfan I	1.4027 ± 0.0056	0.3630 ± 0.0208
PP DDE	0.8723 ± 0.0122	0.0000 ± 0.0000
Dieldrin	122.6104 ± 0.0902	0.0000 ± 0.0000
Endrin	2.0226 ± 0.0046	0.0594 ± 0.0035
PP DDD	1.2329 ± 0.0301	0.0563 ± 0.0009
Endosulfan II	0.0000 ± 0.0000	4.6436 ± 0.0200
PP DDT	10.6625 ± 0.0220	0.0223 ± 0.0010
Endrin Aldehyde	12.0728 ± 0.0014	1.2624 ± 0.0022
Endosulfan Sulfate	184.2944 ± 0.0047	9.5414 ± 0.0206
Methoxychlor	7.6382 ± 0.0568	0.1596 ± 0.0045

CONCLUSION

Sunflower has a great potential as a phytoremediator of organochlorine pesticide residue, especially on loamy soils. Farmers, therefore, can conveniently plant sunflower on their farms at intervals so as to decontaminate the soils of organochlorine pesticide residue and thus increase crop yield.

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Corresponding author: S Garba ,

Department of Chemistry Nigerian Defence Academy Kaduna, Nigeria.

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