Characteristics Levels of Manganese (Mn) in the Soil Profiles of Automobile Mechanic workshops In Ado Ekiti Land, Ekiti State

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Abstract: **Heavy metals such as manganese (Mn) in soils are of great environmental concern. Volumes of literature have been reported in damaging effects of excessive Mn in soils. Automobile mechanic activities remain one of the major indirect sources of manganese into the environment through indiscriminate dumping of wastes. In order to determine Mn content in the soil from the vicinity of automobile mechanic workshop and evaluate the contamination levels, the concentrations and distribution of Mn must be established for different physical and chemical phases of the soil. Different soil layers (0-15cm, 15-30cm and 30-4cm depth) were collected and analyzed for Mn contents using atomic absorption spectrophotometer (AAS). Soil texture, conductivity pH, total organic content and cation exchange capacity were also measured. Sequential extraction was also carried out to determine the geochemical phase of Mn. In the investigated soils, the range total of Mn concentration across the various sampling sites were 41.5- 50-1 mg/kg (0-15cm) 34.6 -37.3 mg/kg (15-30cm) and 21.2-27.8mg/kg (30-45cm),this revealed higher concentration of Mn in topsoil samples(0-15cm) than subsoil samples(15-30cm)(30-4cm). The results showed that enrichment factors (*E.F.*) of Mn were 0.21, 0.20 and 0.31 respectively while the geo accumulation index (*Igeo*) values of the metals in the soil under study indicate that they are uncontaminated with Mn. The mechanic wastes represent a potential source of heavy metal pollution to the environment. Although, the present level of Mn contamination of the soil does not call for alarm. Therefore, necessary steps should be taken to minimize the accumulation of metal. It is now recommended that a separate portion of land be set for automobile workshops called the mechanic village.**

I. INTRODUCTION

The potential toxicity of trace metals in soil or sediments is a function of their mobility and bioavailability, which depends on the metals' phase, chemical and physical processes that govern transformations between phases (Ahmed Wali *et al.*,2014). There is increasing awareness that heavy metals present in the soil may negatively affect human health and the environment (Singh and Kalamahad,2011). From the environmental point of view, all heavy metals are important because they cannot be biodegraded and are largely immobile in the soil system, so they accumulate and persist in the soil for a long period which results in levels that is harmful to humans upon both acute and chronic exposure (Mahatmaphule 2005).

The most frequently reported heavy metals regarding potential hazards and contaminated soil are Cd, Cr, Pb, Zn, Mn, Fe and Cu (Olayinka *et al.*,2017). The concentration of these toxic elements in the soils may be derived from various source: including anthropogenic pollution, weathering of natural high background rocks and metal deposits (Olayinka et.,2017). The contamination of the terrestrial environment by heavy metals has been occurring for millions of years from the natural weathering of the present rocks which precipitate metals into the terrestrial system. Humanity has helped to vastly increase this contamination through various activities e.g. metalliferous mining and smelting, biosolid disposal, fossil fuel combustion, traffic-related emission. Automobile mechanic activities remain one major indirect source of Mn into the environment trough indiscriminate dumping of wastes (Osakwe,2014).

II. EXPERIMENTAL METHOD

2.1 Study Area And Sampling

This investigation was carried out within auto-mechanic workshops at Adebayo and Ilawe streets in Ado Ekiti, Ekiti State, South-Western part of Nigeria. At each of the workshops investigated were three designated sites for soil sampling at 100meter apart. The control (background) samples were obtained from a location remote from the industrial zone and far removed from the influence of industrial activates at the university campus, EKSU at Adebayo street Ado Ekiti, Ekiti State. Sampling design for this study was based on two premises: first, the need to spread sample sites objectively over the study area and second, the need to ensure that site characteristics are adequately depicted. Soil sample was collected from three samplings depths 0-15cm, 15-30cm, and 30-45cm. The sampling was restricted to this zone because it provides the bulk of plant nutrients (Hasan *et al.*, 2014). The three different sites were dug with digger and shovel while samples were collected inside labelled polythene bags.

2.2. Materials Preparation

The soil samples were air-dried at room temperature (R.T.; 20- 25^{0}C) in the laboratory for about two weeks before being crushed with mortar and pestle into fine powder sieved through a 0.5mm mesh. These were later stored in labelled polythene bags.

2.3 Analytical Determination Of Parameters

2.3.1 Determination soil pH

1. Procedure:(soil pH in 0.01m $CaCl_2$) suspension was prepared (10g of soil and 20ml of solution)
2. Occasionally stirring with a glass rod, the suspension was allowed to stand for about 30 minutes.
3. Then electrode of calibrated pH (pH Horiba pH meter D-5)was inserted into the partially settled suspension and pH of the soil was measured.

2.3.2 Soil Temperature/Relative Humidity (R.H.)

Temperature and R.H. reading were taken in the field with the appropriate instrument. These were done by digging a small hole (10cm depth) while the device was placed in it and covered. Reading was taken after 5minutes.

2.3.3 Soil particle size analysis

Particle size composition was obtained by the hydrometer method (Andres *et al*., 2014).

2.3.4 Determination of soil organic carbon (Walkley-Black method)

Walkley and Black,1934 as used by Juliana *et-al*.,2014.

1. A grounded representative sample was made to pass through a 0.5mm sieve.
2. Soil samples were weighed out in duplicate and transferred to 250ml Erlenmeyer flask.
3. With a pipette, 10ml of 1N $K_2Cr_2O_7$ solution was measured accurately into each flask and swirled gently to disperse the sediment.
4. Rapidly, 20ml concentration H_2SO_4 was added using an automatic pipette directing the stream into suspension. Immediately, swirling of the flask began unit the sediment sample and reagents were properly mixed, hence vigorous swirling for one minute. The breaker was rotated and the flask was allowed to stand on a sheet of asbestos for about 30 minutes
5. After standing for 30 minute, 100ml of distilled water was added.
6. 3-4 drops of indicator were added and titrated with 0.5N ferrous sulphate solution. As the end point was being approached, the solution took on a greenish cast and the changed to dark green. At this point, the addition of ferrous sulphate drop by drop until the color changed sharply from blue to red (maroon colour) in reflected light against a white background.
7. Blank titration was carried out in the same manner, but without sediment (steps 3,4,5 and 6) to standardize the dichromate.

2.3.5 Exchangeable acidity Determination (titration method)

Extraction with IN KCl:

1. 5g of air-dry soil (passed 2mm sieve) was weight into a 45 ml centrifuge tube, and 30ml of IN KCl added. The centrifuge was tightly covered and shaken for one hour on a reciprocal shaker using a rubber stopper.
2. The content was centrifuged for 15minutes at 2,000 rpm, and the clear supernatant was carefully decanted into a 100ml volumetric flask.
3. Another 30ml of IN KCl was then added to the same soil sample and was shaken for 30 minutes. Step 2 was repeated, and the clear supernatant also transferred into the same volume trick flask.
4. Step 3 was repeated for the third time, and the clear supernatant was again combined into the same volumetric flask. The volume was made up to mark with IN KCl.

Titration for H.H. and Al

1. 25ml of KCl extract was pipetted into a 250ml Erlenmeyer flask, and approximately 100ml of distilled water was added.
2. 5 drops of phenolphthalein were added and the solution was titrated with 0.5N NaOH to a permanent pink endpoint. A few more drops of an indicator were added to precipitate Al $(OH)_3$.
3. The amount of base used was equivalent to the total amount of acidity (H+Al) in the aliquot taken.
4. One drop of 0.05N HCl was added to the same flask to bring the solution back to the colourless condition, and 100ml of NaF solution added. While stirring constantly, the solution was titrated with 0.05N HCL until the colour of the solution disappeared. One to two drops of the indicator were added due to reappearance of colour and 0.05N HCL was continually added until the colour faded and did not return within 2 minutes. The milliequivalents of acid used were equal to the amount of exchangeable Al.

2.3.5 Determination of Exchangeable Ca, Mg, K, Na, and effective Cation Exchange Capacity (CEC)

1. 30ml of 1N NH_4OAC (Ammonium acetate), was added to 5g of the soil sample and shaken for 2 hours on a mechanical shaker.
2. The solution was centrifuged at 2,000 rpm for 5-10 minute and the clear supernatant was carefully decanted into a 100ml volumetric flask.
3. Another 30ml of NH_4OAC solution was added and the mixture was shaken for 30minutes, centrifuged and transferred the supernatant into the same volumetric flask.
4. Step 3 was repeated and the supernatant transferred into the same volumetric flask.

5. The flask was made to mark with the NH_4OAC solution.
6. K, Na, and Ca were determined on a flame photometer while Mg was determined on an atomic absorption spectrometer
7. Effective CEC was thus calculated by the sum of exchangeable bases (Ca, Mg, K, Na) and exchangeable Al and H expressed in meq/100g(Adamu *et al*., 2014)

2.3.6 Digestion of soil samples for heavy

In the present study, the method of Abegunde *et al*.,2018 was adopted in the digestion of soil sample. It involves weighing 2g of air-dried and homogenized soil sample in a beaker. This was followed by the addition of concentrated $HClO_4$(2ml) and concentration H.F. (10ml). The mixture, placed on a hot plate, was heated until almost dry. Subsequently, the second addition of $HClO_4$ (2ml) and H.F. (10ml), the mixture evaporates to near dryness. Finally, 2ml of $HClO_4$ was added and the sample evaporated until the appearance of white fumes. The residue obtained was dissolved in 12ml HCl and diluted to 25ml. This final solution was used for the determination of Mn metals using atomic absorption spectrophotometer (AAS).

2.3.7 Sequential Extractions of Heavy Metals in Soils

Sequential chemical extraction of Mn in the various soil profile was undertaken using a sequential extraction procedure described by Filqueiras *et a*l.,2002, which was modified by Ahmed(2014). Levels of Mn in the soil were analyzed using atomic absorption spectrophotometer, AAS (Alpha 4AAS, Chemical Tech. Analytical, Euro at Analytical chemistry Laboratory, University of Ibadan, Nigeria). Blanks and standards were used as a quality control measure. Detection limit was 0.01mg/1.Statistical analysis using the SPSS for windows 13.0 versions was later employed for data presentation

III. RESULT AND DISCUSSION

3.1 Physiochemical composition of soil.

The result of the physiochemical composition of soil samples were summarised in table 3.1. The pH across the entire study area ranged range from 6.5-7.1(10-15cm),6.4-6.7(15-30cm) and 5.6-6.6(30-45cm).The topsoils (0-15cm) across the study area suggest slightly acidic to neutral condition while the middle (15-30cm) and bottom layers(30-45cm) suggest slightly acidic condition.

The relative humidity (R.H.) of all soils levels ranged 56.5-61.4. This is a reflection of some climatic variation of the areas under study.The organic matter(OM) of all topsoils ranged from 2.12-3.04 %,middle layer 0.28-2.82 % and bottom soil 0.21-0.13% . The O.M. of the control area is much higher than any of the auto-mechanic workshops.The significant difference in the O.M. content of control and mechanic workshops indeed reflects a depletion of this parameter by wastes which are indiscriminately disposed within workshops.

The soil cation exchange capacity(CEC) of the mechanic workshops ranged from 9.69-10.74(10-15cm),5.21-.7.08(15-30cm) and 3.26-8.07(30-45cm). The CEC is high in all topsoils, followed by the middle soils and least in the bottoms(30-45cm). It is relatively high in control soils.

The concentration of total Mn in the soil under study range (Topsoils) from (41.2-50.1mg/kg) middle soil,(34.6-37.3mg/kg) and inner soil(21.2-27.8mg/kg).The concentration of (Mn) in all topsoils is high followed by the middle and least in the inner soil. All the soil samples showed a decreasing order of the sand>clay >silt in the particle size analysis.

However, the distribution of pH, R.H., O.M. and CEC among the soil samples showed significant impact of anthropogenic pollution by Mn.

Table 3.2 represents the binding fraction of the soil of automobile workshops.The distribution of Mn among specific various forms of soil, reflects both metal and soil characteristics(Isaac *et al*.,2015). The results revealed the different fractions of exchangeable (F_1) easily available (F_2) Mn, Ox (F_3), organic(F_4) amorphous(F_5) Fe oxide crystalline (F_6) and residual(F_7) (Isaac *et al*.,2015)

The exchangeable(F_1) and easily available (F_2) fractions in the extraction of generally represent the bioavailability of total metal(Mn). The sum of F_1 and F_2 as recorded in the table

Table 3.1 Chemical compositions (Mean ± S.D) of soil samples

Specification	Soil Depth(cm)	Soil 1	Soil 2	Soil 3	Control
pH	0-15	7.1±0.2	6.9±0.1	6.5±0.2	6.7±0.1
	15-30	6.7± 0.1	6.7± 0.1	6.4± 0.1	5.7± 0.1
	30-45	5.6± 0.2	6.6± 0.2	6.2± 0.2	5.7± 0.2
Relative Humidity		61.4	56.5	58.3	59
Organic matter(%)	0-15	3.04±0.11	2.67±0.09	2.12±0.12	7.21±0.10
	15-30	2.82± 0.20	1.27±0.10	0.28±0.06	5.27± 0.07
	30-45	1.31± 0.13	0.69±0.13	0.21±0.01	4.51± 0.09

CEC(meq/100kg)	0-15	10.74±1.31	9.69±1.85	9.85±1.89	11.17±0.15
	15-30	6.87± 1.03	7.08±1.15	5.21±0.77	10.29±0.11
	30-45	8.07± 1.34	6.58±1.22	3.26±0.79	9.52± 0.09
Mn(total,mg/kg)	0-15	45.5±0.7	41.2±0.8	50.1±0.5	9.5±0.3
	15-30	34± 0.6	34.6± 0.9	37,3± 0.5	7.2± 0.2
	30-45	27.8± 0.3	24.9± 0.6	21.2± 0.6	4.3± 0.1
Particle size Distribution	Sand(%)	67±3	63±3	71±4	55±2
	Clay(%)	24±3	26±2	22±3	34±2
	Silt(%)	09±2	11±3	09±3	11±2
	T/C	SCL	SCL	SCL	SCL

S.D: Standard Deviation/C: Textural class:SCL: Sand Clay Loam represents a minor percentage of the extracts. Consequently, the metal can be said to have a low index of availability within the study area.

The percentage of Mn found in the MnOx (F3) is relatively low compare with other fractions. In fact, least concentrations were recorded across the soil profile within this fraction.It ranged from 0.8-1.6(0-15cm),0.5-0.9(15-30cm) and 0.2-0.3(30-45cm) with a decreasing order down the soil profiles.The levels of Mn within this fraction are released under reducing conditions. Properly with stronger reducing agent, more Mn would be released. Levels of the metal associated with organic matter are represented in fraction 4(F4). This fraction is relatively high and next to F7.Highest levels within this fraction are recorded in the middle layer(15-30cm) across the various soils. This fraction, F4 represents a laerger portion of the total metal concentration.' Level of Mn recorded in this fraction is released under strong oxidixing conditions,thus,this fraction constituents a source of conditions potentially available Mn in the solis (AhmedWali *et al*.,2014).

From table 3.2, the greatest part of Mn in the studied soil was associated with the residual fraction (F7) across the soil profile.This Fraction,called the first phase corresponds to the fractions which cannot be mobilized. The concentration of Mn within this fraction(F7) which decreasaes down the soil profile ranged from 22.0-28.5(0-15cm)16.4-19.49(15-30cm) and 8.6-11.1(30-45cm) Filqueiras *et al*.,(2002).

Table 3.3 represents the elemental contamination (C.F.s). The C.F.s of the entire study area of auto-mechanic workshops increases down the soil layers. This is a reflection of the leaching of Mn from top soil(0-15cm) to the middle(15-30) and bottom soil(30-45) soil layers respectively.

(Tao zhu,2014).

Table 3.4 represents the enrichement Factor(E.F.) and geoaccumulatio idex(*Igeo*) with respect to the natural background.

The E.F. is defined as;

$$EF = (M/Fe)_{sample} / (M/Fe)_{background} \quad (1)$$

$(M/Fe)_{sample}$ =th**e** ratio of metal and Fe concentraton of the sample and $(M/Fe)_{background}$ =the ratio of metal and Fe concentration of background(Guilherme *et al*.,2011)

The mean E.F. of soil under study ranged between 0.21-0.31.This shows that the study soils are relatively enriched but with a low concentration of Mn.

The geoaccumulatio idex(Igeo) introduced by muller (1969) used by Arena *et al*.,2014 was also used to assess metal pollution in soils at express as:

$$Igeo=\log_2\left[\frac{Cn}{1.5Bn}\right] \text{--}(2)$$

Table 3.2 Results of heavy metal (Mn) binding Fractions

Soil	soil Depth (cm)			Fraction				
		F1	F2	F3	F4	F5	F6	F7
Soil1	0-15	0.9	2.7	1.2	7.3	7.3	3.4	24.5
	15-30	1.5	2.3	0.7	10.2	4	5.5	17.1
	30-45	2.2	1	0.3	5.5	2.8	2.9	11.1
MEAN		1.5	2	0.7	7.6	4.7	3.9	17.6
S.D		0.6	0.8	0.4	3.3	2.3	1.3	6.7
SOIL 2	0-15	0.9	1.2	0.8	8.5	8.8	3.2	22
	15-30	1.9	2.3	0.5	11.7	5.5	1	16.4
	30-45	1	1.5	0.3	7.4	2.2	1.6	8.6
MEAN		1.3	1.7	0.5	9.2	5.5	1.1	15.7
S.D		0.5	0.6	0.3	2.2	3.3	1.1	6.7
SOIL 3	0-15	0.7	2.4	1.6	7.1	8.5	4.9	28.5
	15-30	2	1.8	0.9	11	4.6	1.3	19.4
	30-45	1.3	1	0.2	6.8	2.9	1.9	9
MEAN		1.3	1.7	0.9	8.3	5.3	2.7	19
S.D		0.6	0.7	0.7	2.3	2.8	1.9	9.7
CONTROL	0-15	0.9	0.4	1.3	0.2	0.6	1	5.3

	15-30	0.5	0.5	nd	0.4	0.9	0.7	3.2
	30-45	Nd	10.2	Nd	0.9	0.5	Nd	1.9
MEAN		0.7	3.6	1.3	0.5	0.7	0.8	3.5
S.D		0.2	5.5	0	0.3	0.2	0.2	1.7

F1 = exchangeable; F2= Easily available;F3=MnOx; F 4 =Org;

F5 = FeOx(amorphous);F6=FeOx(crystalline): F7=Residual

Table 3.3 Elemental contamination factors (CFs) of soils

Element	Soil layers	Soil 1	Soil 2	Soil 3	Natural Background Concentration
Mn	0-15	4.7	4.34	5.27	95 ±03
	15-30	4.82	4.81	5.18	7.2± 0.2
	30-45	6.47	5.79	4.93	4.3 ± 0.1

Average natural background concentration(± SD,n=3) -----(3)

Where Cn is the measured concentration of the examined metal in the soil and Bn is the geochemical background concentration of the same metal. Factor 1.5 is the background matrix correction factor due to lithogenic effect.

The Igeo of all soils under study ranged from 0.48-0.55. These results suggest that the soil from auto-mechanic lies within uncontaminated to moderately contaminated with Mn (Khan *et al*.,2018)

Table 3.4 The mean of E.F. and *Igeo* classes of the metals studied with respect to the natural background

ELEMENT	soil 1	soil 2	soil 3
Enrichment Factor Mn	0.21	0.2	0.31
I_{goe} *class Mn*	0.50	0.46	0.55

Normalizing element, Fe, with natural background value of 204.7mg/kg

A >5,extremely contaminated;4-5strongly to extremely strongly contaminated;3-4.strongly contaminated;2-3,moderately to strongly contaminated;1-2,moderately contaminated;0-1,uncontaminated;<0,practically uncontaminated(Khan *et al*.,2018)

IV. CONCLUSION

This survey has helped to determine the level of Mn in the three soil layers which ranged from Topsoil (0-15cm)(41.2-50.1mg/kg) middle soil (15-30cm) (34.6-37.3 mg/kg) and inner soil(21.2-27.8mg/kg). The concentration of (Mn) in all topsoils was high followed by the middle and least in the inner soil samples collected from the vicinity of automobile mechanic workshops in Ado Ekiti, (Adebayo(EKSU) and Ilawe road areas) The soil pollution in the present study was assessed using *E.F.* and *Igeo*. The calculated mean of the E.F. which ranged from 0.2-0.31 showed that all the soils are slightly enriched anthropogenically with Mn. The mean results of *Igeo* of all soils under study ranged from 0.48-0.55. Based on literature classification, it allowed us to conclude that, for analyzed metal (Mn). The soils understudy can be described as uncontaminated with Mn.

However, the level of Mn in the studied areas was not high when this study was carried out, so there is no serious implication for health hazard

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