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Heavy Metal Concentration and Physicochemical Changes of Soil Treated with Spent Engine Oil and Organic Manure (Poultry) After 12 Weeks of Growing *Cyperus compressus* **L.**

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ABSTRACT: Heavy metal concentration and physicochemical changes of soil treated with spent engine oil (SEO) and poultry manure after 12 weeks of growing *Cyperus compressus* was this study's aim. Poultry manure (PM) was 10 % of 4000g (400 g) while the soil was 3600 g. Spent engine oil was added to 4000 gr of mixed soil and PM at different concentrations: 0 mL (control), 40 mL (1 %), 120 mL (3 %) and 220 mL (5 %) v/w oil-in-soil and allowed to stand for seven days in the sack bags before planting. Field observation was carried out. At the end of 12 weeks of growth, the plants were removed. Initial soil and raw SEO samples were subjected to heavy metal analysis (Zn, Hg, Mn, Fe, Pb, Cu, Cr, and Cd). Thereafter, soil after 12 weeks of *Cyperus compressus* culture were also subjected to physical, chemical and heavy metal analysis. The data obtained were subjected to T-test and Analysis of Variance (ANOVA) test respectively. The result obtained showed that there was significant difference (P≤0.05) in the concentration of initial heavy metals in untreated soil and that of raw SEO. Treatment effects on differences in mean was significant ($P \le 0.05$) at all levels, to soil physical parameters, chemical parameters, and heavy metal parameters treated with varying levels of SEO. However, Pb was not detected in the soil samples at all levels. Finally, the plant has demonstrated its potentials in heavy metal uptake, hence, this *C. compressus* can be adopted for recovering hydrocarbon-polluted soil.

Keywords: *Cyperus compressus,* Heavy metal, Hydrocarbon, Sample, Spent engine oil

Introduction

Nigeria has maintained its number 1 rank and 6th largest oil-producing country in the world and oil spillage as a result of oil mining, can introduce heavy metals such as copper, zinc, and lead into the environment which are toxic to plants and damage soil ecosystems (Ekundayo *et al*., 2002). Spent engine oil contains a mixture of different chemicals which include low to high molecular weight $(C_{15}-C_{21})$ compounds, lubricants additives, decomposition products, and heavy metals which are harmful to the soil and human health (Duffus, 2002). The spent engine oil (SEO) is discarded unmonitored by motor mechanics in Nigeria into gutters, water drains, open vacant plots, and farms (Anoliefo and Vwioko, 2001). Atuanya, (2007) reported that waste engine oil causes a breakdown of soil texture, followed by soil dispersion. However, *C. compressus* belongs to the genus *Cyperus*, family Cyperaceae is also known by various other names, such as yellow nutsedge, tiger nut, chufa, iron water chestnut, underground chestnut, underground walnut, earth almond, ginseng fruit, and ginseng bean (Yang, 2017). Cyperaceae are a plant family of grass-like monocots, comprising 5600 species with a cosmopolitan distribution in temperate and tropical regions (Taheri *et al*., 2021).

Methods for the remediation of soils polluted with petroleum products include traditional, chemical, and biological technologies (Nwankwo, 2014). The problems related with them include the fact that they are

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complex technology and are expensive to run (Nwankwo, 2014). Phytoremediation is defined as the use of plants and associated soil microbes to reduce the concentrations or toxic effects of pollutants in the environment (Greipsson, 2011). Phytoremediation is widely accepted as a cost-effective ecological restoration technology (Greipsson, 2011). Moreover, Ffertilizers in the form of urea-phosphate, N-P-K, and ammonia have been used to enhance growth of the soil microbial populations (Nwankwo, 2014). Some workers observed increased biodegradation (Ijah and Antai, 2003, Ahiamadu and Suripno, 2009), while in the others there was no increase in biodegradation rates (Leahy and Colwell, 2000). The above methods main disadvantages are ecological impact and no successful clean-up of petroleum pollutants (Nwankwo, 2014). Moreover, the availability of artificial fertilizers, cost and their ecological impact especially in Nigeria with high rainfall power (Nwankwo, 2014). The introduction of poultry manure as an organic fertilizer is a cleaner option because poultry manure slowly releases its nutrients, acts as a soil conditioner at a reduced cost, accessible, and has higher microbial diversity (Nwankwo, 2014). Understanding the immediate threat caused by the abandonment of spent engine oil remains of great importance as this will further proffer a solution that will aid the conservation, and sustainable use of the soil in order to promote healthy farming. With this in view, the aim was to study the heavy metal concentration and physicochemical changes of soil treated with spent engine oil and organic manure (poultry) after 12 weeks of growing *C. compressus*.

Materials and methods

Collection of soil and plant samples: This study was done in the screen house of the Department of Plant Science and Biotechnology, Michael Okpara University of Agriculture, Umudike. Umudike is located within Longitude 07⁰34'' E, Latitude 05⁰29'' N and at an elevation of 122 m above sea level [National Root Crops Research Institute, Umudike (NRCRIU), 2020]. Plant samples were collected from the surroundings of Michael Okpara University of Agriculture, Umudike. Samples of spent engine oil were collected from motor mechanics shops around MOUAU. Soil samples were collected at the depth of $0 - 15$ cm in crop farms around Michael Okpara university of Agriculture, Umudike. Initially untreated soil sample and spent engine oil were taken to the laboratory for heavy metal analysis.

Research design: The research design of this experiment was completely randomized designed having only one factor (spent engine oil). 400 g of poultry manure was constant to amount of soil needed (3600 g) which amounts to 10 % of 4000 g. While the spent engine oil was varied in 1 %, 3 % and 5 % to the soil and poultry manure content. Control samples had zero levels of spent engine. Each sample was replicated three times. Soil samples were sieved with 2 mm sieve and was mixed in the following way; Negative control (0 g of Poultry manure, 4000 g farm soil), Positive control (400 g of poultry manure, 3600 g of farm soil and zero mL of SEO), 1 % (400 g of poultry manure, 3600 g of farm soil and 40 mL of SEO), 3 % (400 g of poultry manure, 3600 g of farm soil and 120 mL of SEO), 5 % (400 g of poultry manure, 3600 g of farm soil and 220 mL of SEO). The treatment samples were allowed to stand for seven days. Subsequently, three de-leafed plants obtained with the roots and of same heights were planted in each sack bags filled with the above-mentioned treatment. At the end of 12 weeks of transplanting, the plants were removed, the soil were then taken to the laboratory for physical, and chemical constituent evaluation.

Soil porosity: The macro and the micro-pores were the two types of pore size measured . The samples used for the determination of saturated hydraulic conductivity were also used to determined the macro-porosity of the samples (W_{sat}) and the weight after 24 hours of mounting on the tension table at 60cm tension (W₆₀) by the volume of the core (Vc) then multiplying by100%.

$$
Maccro-porosity = \frac{W_{sat} - W_{eq}}{Vc} \times 100\%
$$

Also in the same sample, micro-porosity was determined by dividing the difference between the weight of sample after 24 h of mounting on the tension table at 60 cm tension (W_{60}) and the weight of oven dried sample at 105 °C for 48 h (W₀) by the volume of the core (V_c), then multiplied by 100%.

$$
Micro - porosity = \frac{W_{60} - W_0}{V_c} \times 100
$$

Soil pH: Soil pH was determined with the use of pH indicator (meter). Apparaus used were test tube, pH meter, weighing balance, spatula, beaker. Five (5 g) of the soil samples were weighed into 5 g of distilled water in a test tube and vigorously stirred. The pH was obtained using pH indicator and read after 3 seconds. It was cross matched with the colour scale pH of 6.5 will be obtained for the soil sample without oil while pH of 6.0 was obtained for the waste or used oil containnated soil.

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Phosphorus content: This was determined by the Bray and Kutz (1945) method with the use of absorption spectrophotometer. One gramme (1 g) scoop of soil and 10.00 mL of extractant were mixed together for 5min. This was further shaken to give a blue colour. The intensity of the blue colour filtrate development was treated with ammonium molybdate – hydrochloric acid solution and aminonaphthol – sulfonic acid solution. The colour was measured using an absorption spectrophotometer at 640nm. The result was calculated in ppm.

Potassium content: Apparatus used were test tube, colour scale, beaker and spatula. A test tube was placed into cavity of the thermoformed lining and filled with 0.7 % nitric acid. Potassium test sticks were removed as required and then the container was resealed immediately. A test stick was dipped into the solution to be tested so that the reaction zone was completely moist. Excess liquid was shaken off. A test stick was placed into the test tube which was filled with 0.7 % nitric acid and then left for one minute. The test tube was removed and compared with the color scale. In the presence of potassium, the test paper turned yellow to orange red.

Total organic carbon: Total organic carbon (TOC) was determined by the Walkey and Black (1934) wet dichromate oxidation method.

Total nitrogen: Total nitrogen was measured by the macro Kjedahi digestion procedure as described by Bremner (1965).

Cation exchange capacity: Cation exchange capacity (CEC) was determined by the ammonium acetate displacement method as described by Chapman (1965).

Analysis of heavy metals: Bio-available or soluble concentration of heavy metal was determined by Aqua Regia method (Chen and Ma, 2001). Conventional aqua regia digestion was performed in 250- mL glass beakers covered with watch glasses. A well-mixed sample of 0.5000 g was digested in 12 mL of aqua regia on a hotplate for 3 h at 110 °C. After evaporation to near dryness, the sample was diluted with 20 mL of 2 % (v/v with H₂O) nitric acid and transferred into a 100 mL volumetric flask after filtering through Whatman no. 42 paper and diluted to 100 mL with DDW. The filtrates were analyzed for Zn, Hg, Mn, Fe, Pb, Cu, Cr, and Cd using atomic absorption spectrophotometer. The values were compared with the widely used normal and critical levels of total concentration of heavy metal for the contaminant limit (c), p index was calculated as the ratio between the heavy metal content in the soil and the toxicity criteria (the tolerable levels).

Data analysis: Data collected was subjected to descriptive statistics to obtain the means and standard deviations. T-test analysis was used to compare the difference in the heavy metal properties of soil and spent engine oil. Means of the laboratory analysis were subjected to analysis of variance (ANOVA). Statistically significant means at 5 % probability were separated using Ducan multiple range test (DMRT). All the test was done using statistical package for social sciences (SPSS) version 26.

Results

Heavy metal content of experimental soil and spent engine oil is presented in Table 1. From the result, Zn in the soil was 0.025 mg/kg while that of spent engine oil was 9.310 mg/kg. Hg in the soil was 0.025 mg/kg while that of spent engine oil was 7.250 mg/kg. Mn in the soil was 0.615 mg/kg while that of spent engine oil was 33.725 mg/kg. Fe was in the soil was 0.465 mg/kg while that of spent engine oil was 14.885 mg/kg. Pb in the soil was 0.000 mg/kg while that of spent engine oil was 119.300 mg/kg. Cu in the soil was 0.000 mg/kg while that of spent engine oil was 10.825 mg/kg. Cr in the soil was 0.035 mg/kg while that of spent engine oil was 17.270 mg/kg. Cd in the soil was 0.045 mg/kg while that of spent engine oil was 14.360 mg/kg. There was significant difference ($P \le 0.05$) in the concentration all the heavy metals in soil and that of spent engine oil.

* (p≤0.05), ** (p≤0.01), *** (p≤0.001), NS (not significant) (p>0.05)

Particle size composition of soil treated with spent engine oil and poultry manure before vegetation is presented in Table 2. From the result, sand ranges from 55.20±0.00 % of Negative control sample to 80.00±0.28 % of 5 % sample. Treatment effect on difference in mean was significant (P≤0.05). Silt ranges from 11.45±0.07 % of 3 % sample to 22.00±0.00 % of negative control sample. Treatment effect on difference in mean was significant (P≤0.05). Clay ranges from 4.95±0.21 % of 1 % sample to 23.80±0.28 % of negative control sample. Treatment effect on difference in mean was significant (P≤0.05).

Table 2: Particle size composition of soil treated with spent engine oil and poultry manure before vegetation

Particle Size Composition	Sand $(\%)$	$Silt (\%)$	Clav(%)
Po control	$69.95 \pm 0.21^{\rm b}$	17.50 ± 0.14^b	12.95 ± 0.21 ^d
Ne control	$55.20 \pm 0.00^{\mathrm{a}}$	22.00 ± 0.00 ^d	23.80 ± 1.41 ^e
1%	75.95 ± 0.21 °	19.45 ± 0.07 °	4.95 ± 0.21 ^a
3%	78.00 ± 0.28 ^d	$11.45 \pm 0.07^{\text{a}}$	11.00 ± 0.28 ^c
5%	80.00 ± 0.28 ^e	11.50 ± 0.14 ^a	$9.00+0.28b$
Total	$71.82 + 9.45$	$16.38 + 4.48$	12.34 ± 6.67
$P \leq 0.05$	$0.000***$	$0.000***$	$0.000***$

* (p≤0.05), ** (p≤0.01), *** (p≤0.001), NS (not significant) (p>0.05). Means \pm standard deviation with different superscript alphabet are significantly different at alpha 0.05. PO (Positive), Ne (Negative).

The chemical composition of experimental soil treated with spent engine oil and poultry manure is presented in Table 3a. From the result, pH ranges from 4.55 ± 0.07 of negative control sample to 7.15 ±0.07 of positive control sample. Phosphorus (P) ranges from 26.15 ± 0.07 of 5 % sample to 128.55 ± 6.68 of negative control sample. Treatment effect on difference in mean was significant (P≤0.05). Nitrogen (N) ranges from 0.10±0.00 of 5 % sample to 0.29 ± 0.01 of negative control sample. Treatment effect on difference in mean was significant (P≤0.05). Organic carbon (OC) ranges from 2.43±0.01 of negative control sample to 3.46±0.01 of 5 % sample. Treatment effect on difference in mean was significant (P≤0.05). Organic matter (OM) ranges from 2.02±0.01 of positive control sample to 5.98±0.04 of 5 % sample. Treatment effect on difference in mean was significant (P≤0.05). Calcium (Ca) ranges from 4.01±0.01 of 5 % sample to 13.01±0.01 of positive control sample. Treatment effect on difference in mean was significant (P≤0.05).

* (p≤0.05), ** (p≤0.01), *** (p≤0.001), NS (not significant) (p>0.05). Means \pm standard deviation with different superscript alphabet are significantly different at alpha 0.05. PO (Positive), Ne (Negative).

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The Chemical composition of experimental soil treated with spent engine oil and poultry manure is presented in Table 3b. From the result, Magnesium (Mg) ranges from 2.45 ± 0.07 cmol/kg of 5 % sample to 9.01 ± 0.01 cmol/kg of positive control sample. Treatment effect on difference in mean was significant (P≤0.05). Sodium (Na) ranges from 0.12 ± 0.00 of 1 % sample to 0.36 ± 0.00 cmol/kg of negative control sample. Treatment effect on difference in mean was significant (P≤0.05). Exchangeable Acidity (EA) ranges from 0.57±0.01 cmol/kg of 1 % sample to 1.40±0.06 cmol/kg of negative control sample. Treatment effect on difference in mean was significant (P≤0.05). ECEC ranges from 7.60±0.04 cmol/kg of 5 % sample to 23.30±0.02 cmol/kg of positive control sample. Treatment effect on difference in mean was significant (P≤0.05). Base saturation (BS) ranges from 88.44±0.01 of 5 % sample to 95.63±0.02 cmol/kg of 1 % sample. Treatment effect on difference in mean was significant ($P \le 0.05$).

Table 3b: The chemical composition of experimental soil treated with spent engine oil and poultry manure

* (p≤0.05), ** (p≤0.01), *** (p≤0.001), NS (not significant) (p>0.05). Means \pm standard deviation with different superscript alphabet are significantly different at alpha 0.05. PO (Positive), Ne (Negative).

Heavy metal composition of the experimental soil treated with spent engine oil and poultry manure after growth in Table 4. From the results, Zinc (Zn) ranges from 0.03 ± 0.02 mg/kg of the negative control sample to 0.28±0.02 mg/kg of the 5 % sample. Treatment effect on difference in mean was significant (P≤0.05). Mercury (Hg) was only detected in Negative control sample at 0.03 ± 0.01 mg/kg. Treatment effect on difference in mean was significant (P≤0.05). Manganese (Mn) ranges from 0.06 ± 0.00 mg/kg of the 5 % sample to 6.05 ±0.01 mg/kg of the positive control sample. Treatment effect on difference in mean was significant (P≤0.05). Iron (Fe) ranges from 0.00 ± 0.00 mg/kg of the positive control sample to 3.80 ± 0.02 of 1 % sample. Treatment effect on difference in mean was significant (P≤0.05). Lead (Pb) was not detected in all the samples. Copper (Cu) ranges from 0.00±0.00 mg/kg of the 3 % sample to 2.42±0.02 mg/kg of the 1 % sample. Treatment effect on difference in mean was significant (P≤0.05). Chromium (Cr) ranges from 0.00 ± 0.00 mg/kg of the positive control sample to 0.41 ± 0.01 mg/kg of the 1 % sample. Treatment effect on difference in mean was significant (P≤0.05). Cadmium (Cd) ranges from 0.00 ± 0.00 mg/kg of the 3 % sample to 0.63 ± 0.01 mg/kg of the 5 % sample. Treatment effect on difference in mean was significant (P≤0.05).

Table 4: Heavy metal composition of the experimental soil treated with spent engine oil and poultry manure after growth

	Zn (mg/kg)	Hg (mg/kg)	Mn (mg/kg)	Fe (mg/kg)	Pb (mg/kg)	Cи (mg/kg)	Cr (mg/kg)	Cd (mg/kg)
Po Control	$0.07 \pm 0.01^{\rm b}$	0.00 ± 0.00^a	6.05 ± 0.01 ^e	$0.00+0.00^a$	ND	0.18 ± 0.00 ^c	$0.00+0.00^a$	0.37 ± 0.01 °
Ne Control	0.03 ± 0.02^a	$0.03 \pm 0.01^{\circ}$	0.62 ± 0.01 ^a	0.47 ± 0.01 ^c	ND.	$0.12 \pm 0.01^{\rm b}$	$0.04+0.01b$	$0.05 \pm 0.01^{\circ}$
1%	0.23 ± 0.01 ^c	$0.00+0.00^a$	0.33 ± 0.01^b	$3.80 + 0.02^b$	ND.	$2.42+0.02^e$	0.41 ± 0.01 ^e	0.48 ± 0.02 ^d
3%	0.21 ± 0.01 °	$0.00+0.00^a$	4.33 ± 0.01 ^d	$0.35 \pm 0.01^{\circ}$	ND.	$0.00+0.00^a$	0.06 ± 0.00 ^c	$0.00+0.00^a$
5%	$0.28 + 0.02d$	$0.00+0.00^a$	$0.06 + 0.00^a$	0.92 ± 0.02 ^d	ND	$0.57+0.01d$	0.15 ± 0.01 ^d	0.63 ± 0.01 ^e
Total	$0.16+0.10$	$0.01 + 0.01$	$2.28 + 2.58$	$1.11 + 1.45$	$\overline{}$	$0.66 + 0.95$	$0.13+0.15$	$0.30+0.26$
$P \le 0.05$	$0.000***$	$0.000***$	$0.000***$	$0.000***$	٠	$0.000***$	$0.000***$	$0.000***$
WHO/FAO	50.00	270.00		425.50	85.00	36.00	100.00	85.00

* (p≤0.05), ** (p≤0.01), *** (p≤0.001), NS (not significant) (p>0.05). Means \pm standard deviation with different superscript alphabet are significantly different at alpha 0.05. PO (Positive), Ne (Negative).

Discussion

Heavy metal constituent of spent engine oil and that of the soil were screened for heavy metal composition. It was observed that soil concentrations of heavy metal were very low when compared to raw spent engine oil concentrations of heavy metal which were higher. Hence, the significant variation (P≤0.05) existing between the soil heavy metal and raw spent engine. This confirms that spent engine oil contaminates the soil with toxic heavy metals. This finding was in line with observation of Kashif *et al*. (2018) who compared fresh and unused engine oil with spent engine oil and confirmed that higher Pb, Cu, Cr, Ni and Fe were found in spent engine oil. Physical parameters of the control soil were significantly (P≤0.05) lower than the spent engine oil treated soil samples. The result of this study was not in agreement with the report of Nwite *et al*. (2016) who stated that physical properties of soil following kerosene oil contamination and poultry manure amendment had no significant (P>0.05) treatment effect on bulk density of kerosene oil contaminated soil. Higher sand levels, lower silt and clay were observed in the treated soil than the original control samples. This infer that such soil will have lower nutrient holding capacity. Previous studies on hydrocarbon oil contamination of soil by Mbah *et al*. (2009), Nwite (2013) and Ogbohodo *et al*. (2001) reported similar findings. They argued that the treatment loosened soil compaction and increased its total porosity, improved aggregate stability as well as moisture content of soil.

Varying levels of chemical constituent was observed in this present study. Hydrogen ion concentrations of the negative control were more acidic. This was in confirmation with Ediene *et al*. (2016) reported higher acidity in the control than those treated soil samples. The higher organic matter, organic carbon, Ca, and Mg were found in treated soil and the positive control; hence, this may be connected to carbon reach nature of spent engine oil and poultry manure. However, lower concentration of nitrogen, phosphorus and potassium (NPK) which were important for plant growth and development were recorded for 1 % and 3 % spent engine oil treated samples. Similarly, this study agreed with Osaigbovo *et al*. (2012) who reported low nitrogen, phosphorus and potassium (NPK) in soil treated with spent engine oil and amended with fertilizer. Control soil samples were higher in nitrogen levels than spent engine oil treated plant samples. However, the positive control plant samples had higher phosphorus and potassium (NPK). Hence, confirms the ability of plant to take up available nutrients from the soil.

World Health Standards for heavy metal availability in soil and Food and Agricultural Organization standards were higher than all the observed heavy metal levels across the samples. However, increased amount of heavy metal was observed in soil samples treated with spent engine oil. This suggests that spent engine oil has the possibilities to cause soil heavy metal toxicity. Stout *et al*. (2018) opined that spent engine oils contain metals, which upon entering soils may pose risks to human health or the environment.

Conclusion

The physical, chemical and heavy metal were significant $(P \le 0.05)$ to changes in their concentrations. Higher organic matter and carbon was seen in the soil treated with spent engine oil. Higher heavy metal was observed the in spent engine oil than those of the farm soil. Confirming spent engine oil as soil heavy metal toxicant. This indicates the potentials of *C. compressus* in phytoremediation.

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