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Effect of Chemically Modified Biowaste-Low Density Polyethylene Waste Composites for Sorption of Heavy Metals in Soil Samples collected at Waste Dumpsite in Uvwie Local Government Area of Delta State, Nigeria

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ABSTRACT: The objective of this paper was to evaluate the effect of chemically modified biowaste-low density polyethylene waste composites prepared in a 1:1 and 1:2 ratios for sorption of heavy metals in soil samples collected at waste dumpsite in Uvwie Local Government Area of Delta State, Nigeria using appropriate standard techniques. Data obtained show reduction of values of metal concentration across board, before and after treatment with sorbents. These average values of these parameters include pH (before: 6.01±0.23, after: 6.15±0.25), cadmium (before: 12.39±1.77; after: 9.24±3.24), chromium (before: 1.65±0.43; after: 1.26±0.29), iron (before: 744.38±207.77; after: 478.44±262.94), zinc (before: 12.83±3.12; after: 10.00±1.01), nickel (before: 28.72±4.70; after: 20.33±6.51), lead (before: 19.48±17.92; after: 12.85±4.44), and manganese (before: 16.41±4.46; after: 10.31±6.54). The sorption capacity of the modified sorbents was higher than that of the unmodified sorbents. Modification of the sorbents surface increases the surface characteristics thereby decreasing the concentration of heavy metals and increasing the sorption of these heavy metals from contaminated soils. The unmodified composite 1:2 (LDPE: sawdust) sorbed more metals than unmodified composite 1:1 because the increased proportion of sawdust provided a greater surface area, higher porosity, and more functional groups for metal adsorption. The modified composite 1:1 $CaCO_3$ and composite 1:2 $Mg(OH)_2$ sorbed Mn, Ni, Fe, Pb and Cd more than the other sorbents. The composite 1:1 SiO_2 and composite 1:2 $Ca(NO_3)_2$ sorbed Cr more than the other sorbents and least sorbents that sorbed Cr are composite 1:1 K_2CO_3 and composite 1:2 $Mg(OH)_2$ while composite 1:1$CaCO_3$ and composite 1:2 $Mg(OH)_2$ sorbed Zn more than the other sorbent. It is due to the varying chemical properties and interactions of the modifying agents with the specific metal ions. Each combination of composite ratio and modifying agent creates a unique sorbent with distinct sorption behaviors for different metals.

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Metals with a high atomic mass greater than 20 and density greater than 5 gcm^{-3} are known as heavy metals. Some heavy metals, such as iron, zinc, copper, manganese, chromium, molybdenum, and selenium, are necessary as trace components for proper running and maintenance of the human body's metabolism. However, these metals can become very toxic in higher amounts. When exposed to trace elements like mercury, lead, and plutonium, which are toxic and have no known beneficial effects, these elements accumulate over time in the bodies of humans and other living organisms. The higher the concentration, the more toxic the element. The health of people and other living things may be negatively impacted by excessive levels. Food, air, and drinking water are the primary sources of these heavy metals

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in human body (Singh *et al.*, 2020; Lane and Morel, 2009). According to studies, important nutrients that are needed for a variety of metabolic processes of humans and other living organisms include metals like copper, manganese, cobalt, chromium, selenium, iron, magnesium, molybdenum, nickel, and zinc. Many nutrient deficiencies related disorders or deficiency syndromes are triggered by an inadequate supply of trace amount of these metals (Tchounwou *et al.*, 2014). These critical heavy metals play special roles in several enzyme-catalyzed oxidation-reduction reactions in plants and animals, and are imporant components of these essential enzymes (Tchounwou *et al.*, 2014). Several health risks have been directly or indirectly linked to heavy metal toxicity, a dire situation which has been considered as a serious threat. Even if these metals lack any chemical roles, their toxic effects still cause damages to the human body thereby altering its optimal functioning (Monisha, *et al.*, 2012). The continued presence of heavy metals as environmental contaminants is a problem that is becoming increasely significant for evolutionary, ecological, nutritional, and environmental concerns (Jaishankar *et al.*, 2013; Nagajyoti *et al.*, 2010). Metals like arsenic, chromium, cadmium, copper, lead, mercury, zinc, and nickel are the heavy metals most abundant in wastewater and soil, with their attendant harm to human health and the environment (Lambert *et al.*, 2000). Heavy metals are launched into the surroundings via natural and human activities. Various reinforces of heavy metals consist of soil erosion, mining, herbal weathering of the earth's crust, urban runoff, business effluents, sewage discharge, insect or ailment management sellers carried out to crops, and lots of others (Monisha *et al.*, 2012). Heavy metal contamination presents substantial health risks to both animals and humans alike (Carolin *et al.*, 2017). For instance, low levels of cadmium in humans can cause "Itai-Itai" sickness, lung cancer, and renal and liver issues (Demim *et al.*, 2013). Consuming water that is infected with an excessive quantity of Cr can bring about ulcers, pores and skin inflammation, and lung congestion (Miretzky and Cirelli, 2010). Chest pain, diarrhoea, gastrointestinal discomfort, nausea, pulmonary fibrosis, and renal oedema are some of the health issues linked to ingesting or inhaling nickel dust. Leaking lead into the environment has been linked to cancer, kidney and neurological system disorders, and mental retardation (Qu *et al.*, 2013; Cechinel *et al.*, 2013). The most dangerous form of copper is Cu^{2+}, which can cause anemia, headaches, renal damage, and even death (Awual *et al.*, 2015; Tang *et al.* 2014). A high zinc intake in humans can cause anemia, fever, vomiting, and allergic reactions (Petcu *et al.*, 2015). Mercury consumption can harm the kidney, brain, respiratory system, and reproductive organs (Singh *et al.*, 2014). Surface modifiers play a key role in altering the sorption behavior of heavy metals in soil and water. These modifiers, also known as amendments or additives, are substances that are applied to the soil or water to modify their physicochemical properties and enhance the sorption of heavy metals. Sorption of heavy metals is a vital feature in environmental pollution remediation. These techniques have the cap potential to take away contaminants through adsorption onto sorbents. However, sorbent choice for pollution remediation lies on the materials' sorption capacity, pH of sorption and the contaminants distribution coefficient (Alby *et al.*, 2018). To increase the sorption capacity, the sorbent can be modified physically or chemically. For instance, activated carbon is known as a good adsorbent for the treatment of wastewater and soil, but it has several drawbacks, including high energy consumption coupled with high cost. Other common sorbents studied are graphene-based composites, clays, magnetic nanoparticles, agricultural waste products (Tang *et al.*, 2014). Therefore, the objective of this paper was to evaluate the effect of chemically modified biowaste-low density polyethylene waste composites prepared in a 1:1 and 1:2 ratios for sorption of heavy metals in soil samples collected at waste dumpsite in Uvwie Local Government Area of Delta State, Nigeria.

MATERIALS AND METHODS

Sampling: The soil samples were collected very close to the waste dumpsite at back of Tetfund building at the Federal University of Petroleum Resources, Effurun in Uvwe Local Government Area of Delta State, Nigeria within the coordinates 5°34"23'N and 5°50"38E. The major activity around the sampling site is agricultural activity. The low-density polyethylene waste was randomly collected from different locations within the premises of the University by hand picking. The Saw dust was collected from Udu Bridge Saw Mill in Udu, Warri, and Delta State.

Sample preparation: The soil samples were taken to the Department of Chemistry Laboratory, Federal University of Petroleum Resources, Effurun, where the unwanted materials were removed from them by hand picking. The saw dust samples were hand-picked to remove unwanted particles and sun dried for one month to remove moisture and pass through 0.4mm mesh sieve The low-density polyethylene wastes were washed with tap water, rinsed with distilled water and air dried to remove moisture.

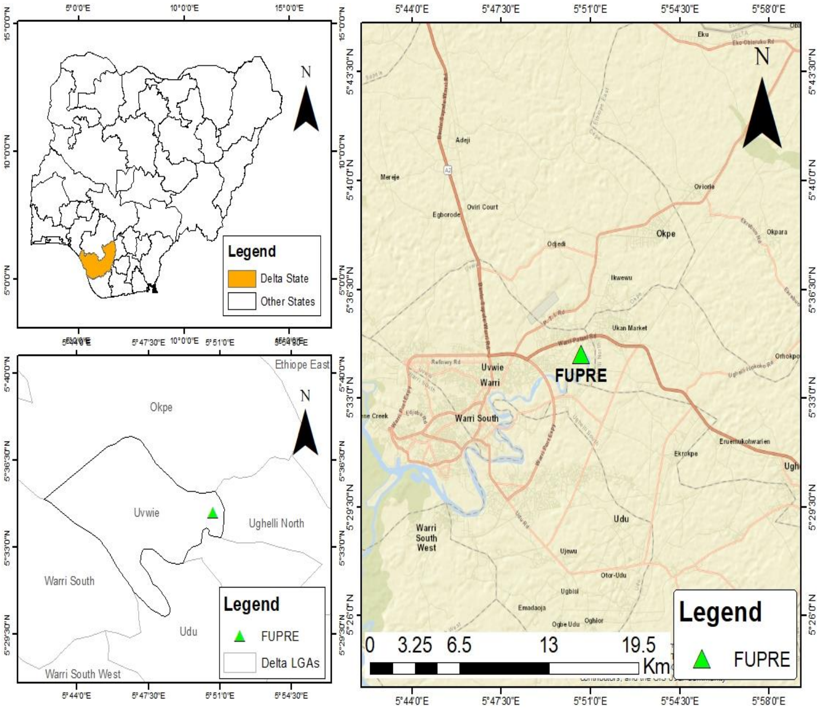


Fig. 1: Location of the sampling site

Preparation of composite/ modification: The LDPE sachet wastes were fed into the pelleting machine to produce intermediate nodules which were channeled to the cutter segment, which the machine churned out as pellets. The pellets and the sawdust were mixed in different ratios by weight, 1:1, 1:2. The ratio 1:1 same amount of sawdust and the LDPE pellets while the ratio 1:2 is 1 of LDPE pellets and times 2 of sawdust. The different ratios were fed into the extruding machine set at 250 ^{o}C. The hot mixed composite was collected in 30cm length and width 5 inch and thinness 1 cm metallic molds. The molds were greased with olive oil, 20 g of the chemicals was weighed, some portion of the weighed chemicals ($CaCO_3$, $Ca(NO_3)_2$, $Mg(OH)_2$, K_2CO_3 and SiO_2) was sprinkled on the greased mold which is used to collect the hot composite afterward, the other potion of the chemicals was sprinkled on the surface of the composite before cooling of the composite. The modified composite was carefully scaled out of the molds after cooling of the composites.

Sorption study: Prior to the metal sorption study, a portion of each of the soil samples was collected and prepared for heavy metal content determination using atomic absorption spectrophotometer (AAS). The heavy metals determination of untreated soil serves as a baseline reference 24 hours after treatment. The composites (sorbents) were buried in different portions of the soil samples which were thoroughly shaken for intimate mixing. A portion of each of the soil samples was also collected 24 hours after burying the sorbents in order to determine heavy metal content using AAS analysis.

*Ph. determination:*The pH of soil was determined before and 24 hours after burying the sorbents. 10 g of each of the soil samples was sieved through a 2-mm mesh into a 50 mL beaker. 10 mL of distilled deionized water was added into the beaker and was allowed to stand for 30 minutes and stirred contiuously with a glass rod within the duration of standing. The electrode (probe) of the pH meter was thereafter inserted into the partly settled mixture and then the pH recorded.

Determination of metal concentration: Heavy metal concentration in soil samples were determined following standard procedures recorded by Mikkonen and Tummavuori (2014) with some adjustments. Soil samples before and after treatment were analyzed using the Atomic Absorption Spectrometer (GBC 932 AA). The samples were air dried in an oven at 105 ^{o}C for 24 hours to remove moisture. The dried samples were grinded into fine powder with mechanical grinder, and sieved with a 2 mm mesh. A digestion process was then performed by the addition of 20 mL of aquaregia (mixture of concentrated nitric acid and hydrochloric acid 1:3) to 1 g of filtered sand in a 250 mL conical flask, placed on a hot plate at 180 ^{o}C for 30 minutes under reflux in a fume hood, and then allowed to cool. The digested sample was then filtered using a 0.45 µm filter paper to remove any remaining undigested solids. The filtered sample is then transferred to a 100 mL volumetric flask, and then diluted to the mark with distilled deionized water. The AAS instrument was then used to measure the absorbance of light by the analyte ions at a wavelength 190 – 850 nm. Prior, calibration standards were prepared with known concentrations of each metal ions to be determined. The slope (m) and intercept (b) of the calibration curve was determined using linear regression. Heavy metal concentration (in mg/kg of soil) is determined as follows:

$$Conc.(mg\ per\ kg) = \frac{1000x(A-b)}{\mathrm{mV}} \quad (1)$$

Where: A = Absorbance of heavy metal sample (nm); x = initial soil weight (g); b = intercept of calibration curve; m = slope of calibration curve; V = final volume after digestion.

RESULTS AND DISCUSSION

The US EPA's soil screening levels (SSLs) for heavy metals and pH alongside values before and after treatment with the various composites are presented in Table 1. The concentrations of individual heavy metals sorbed by various composites are presented in Figure 2.

Effect of Surface Modifiers on pH.: The results obtained from the physicochemical analysis of the soils are shown on Table 1. Soil pH is a widely accepted parameter that influences the availability of micro-nutrients and heavy metals in the soil to plants. The pH of the soil from the dumpsite of the Federal University of Petroleum Resources Effurun was found to be in the range of acidic region, with an average of 6.01±0.23. There was a considerable increase in soil pH values across board, with an average of 6.15±0.25 when sorbents were introduced. These sorbents work through ion exchange and neutralization reactions that reduce the concentration of hydrogen ions in the soil, leading to an increase in pH. By incorporating these materials, the overall soil pH is adjusted upwards, making the environment less acidic and more suitable for certain crops and enhanced heavy metal sorption. According to Okuo *et al.*, (2014), some Nigerian soils, particularly in the forest and savannah zones, have a pH between 5.70 and 6.50. This pH range was considered to be typical for common soils that encourage microbes and plant growth. Further, the US EPA also provides guidelines for soil pH. The recommended pH range of soil varies, and this variation depends on the land use as well as the types of plants being grown. Generally, the guidelines for soil pH is 6.0-6.8 for residential and agricultural soil, 6.0-7.0 for gardening and landscaping soil, and 5.5-7.5 for forestry and wildlife habitats (US EPA, 2021). Though soil pH outside these ranges can be detrimental to plant growth, the soil used for this study falls within the residential and agricultural guidelines, indicating no potential environmental concerns.

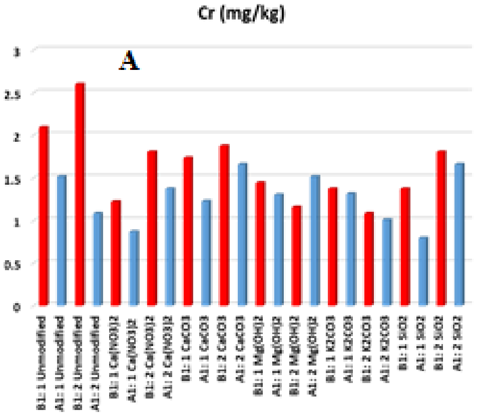
Effect of surface modification of composites: The accumulation of heavy metals in soil poses a significant threat to environmental sustainability and human health (Kabata-Pendias, 2011). Conventional methods for heavy metal removal, such as chemical precipitation and ion exchange, have limitations due to high operational costs and secondary pollution (Wang *et al.*, 2017). Surface modification of materials has emerged as a promising strategy to enhance heavy metal sorption from soil. The mechanism of heavy metal sorption on surface-modified materials involves complexation, ion exchange, and precipitation reactions (Brigatti *et al.*, 2017). Surface modification can increase the availability of functional groups, such as hydroxyl, carboxyl, and amino groups, which can form complexes with heavy metals (Crini, 2005). Additionally, surface modification can alter the surface charge of materials, enhancing electrostatic attraction between the material surface and heavy metal ions (Ngah & Hanafiah, 2008). Several studies have demonstrated the efficacy of surface-modified materials in removing heavy metals from soil. For example, surface-modified zeolites exhibited high sorption capacity for lead and zinc (Wang *et al.*, 2017). Similarly, surface-modified graphene oxide showed improved sorption capacity for cadmium and mercury (Li *et al.*, 2018). Based on the results (Table 1), the composite ratio (1:2) unmodified sorbed higher metal concentration for most of the metals than composite ratio (1:1). For example, composite ratio (1:2) unmodified 10.31 mg/Kg of Cd, 1.08 mg/kg of Cr, 247.21 mg/Kg of Fe, 9.68 mg/Kg of Zn, 23.27 mg/Kg of Ni, 11.46 mg/Kg of Pb, 5.29 mg/Kg of Mn while composite ratio (1:1) unmodified absorbed 9.37 mg/Kg of Cd, 1.51 mg/Kg of Cr, Zn of 10.26 mg/Kg, Ni of 17.14 mg/Kg, Pb of 15.28 mg/Kg, and Mn of 9.05 mg/Kg. This could be because of the increase in the amount of biowaste to LDPE which increased number of pore spaces and the matrix thereby making it easier for increased sorption of the metals. The composite modified with Calcium Nitrate ($Ca(NO_3)_2$, calcium carbonate ($CaCO_3$), magnesium hydroxide ($Mg(OH)_2$), potassium carbonate (K_2CO_3), and silica gel (SiO_2) sorbed more metals than the unmodified composite at their different ratios 1:1 and 1:2. .

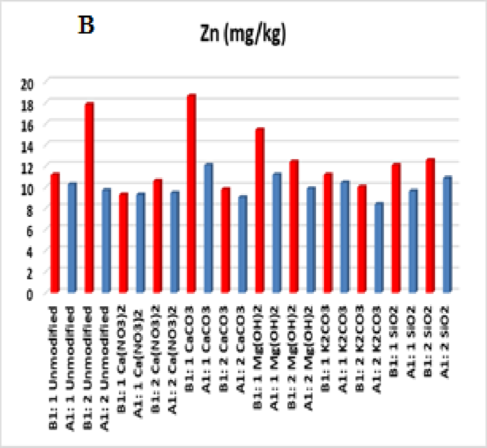
Table 1: pH and Heavy Metal Values of Soil Before and After Treatment with Composites

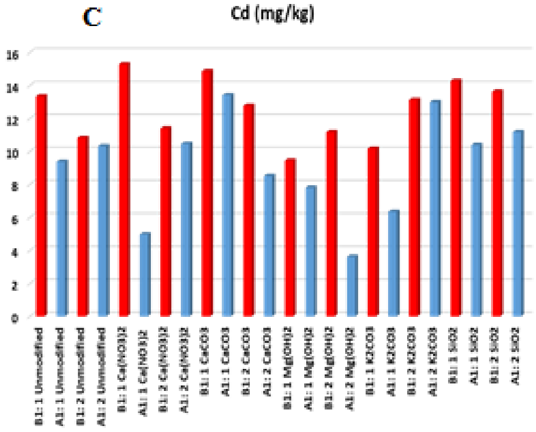
Parameters		**pH**	**Cd mg/Kg**	**Cr mg/Kg**	**Fe mg/Kg**	**Zn mg/Kg**	**Ni mg/Kg**	**Pb mg/Kg**	**Mn mg/Kg**
Tolerable limits (US EPA, 2021)		6-8	1.8-39	230-390	-	1500-3000	35	400	-
1:1 Unmodified	Before	6.06	13.340	2.091	769.907	11.166	28.382	74.223	17.181
	After	6.09	9.374	1.514	531.037	10.263	17.145	15.283	9.054
1:2 Unmodified	Before	6.01	10.816	2.596	952.270	17.817	24.336	13.806	13.806
	After	6.28	10.311	1.082	247.218	9.689	23.277	11.462	5.298
1:1 + $Ca(NO_3)_2$	Before	5.58	15.287	1.226	564.427	9.278	35.734	22.828	14.865
	After	6.25	4.975	0.865	175.942	9.278	14.705	3.371	2.536
1:2 + $Ca(NO_3)_2$	Before	6.03	11.393	1.803	251.070	10.592	26.006	12.168	6.421
	After	5.62	10.455	1.370	671.020	9.442	23.662	11.077	14.416
1:1 + $CaCO_3$	Before	5.86	13.412	1.731	977.313	18.638	33.808	16.438	22.667
	After	6.18	14.854	1.226	998.503	12.070	28.896	20.066	23.823
1:2 + $CaCO_3$	Before	6.32	12.763	1.875	561.859	9.77 1	29.666	17.048	15.283
	After	6.47	8.509	1.658	290.882	9.032	19.264	8.926	6.710
1:1 + $Mg(OH)_2$	Before	6.18	9.446	1.298	822.561	15.436	22.121	9.889	18.108
	After	6.38	7.787	1.442	382.706	11.166	20.355	15.250	9.192
1:2 + $Mg(OH)_2$	Before	6.04	11.176	1.514	751.927	12.398	23.373	3.403	15.283
	After	6.09	3.605	1.154	104.024	9.853	8.476	15.700	1.060
1:1 + K_2CO_3	Before	6.28	10.167	1.370	925.301	11.166	24.529	12.072	24.336
	After	6.48	6.345	1.310	353.168	10.427	11.430	8.348	6.357
1:2 + K_2CO_3	Before	5.64	12.979	1.082	668.451	10.017	29.859	16.278	15.315
	After	5.89	13.123	1.009	640.840	8.375	29.377	15.796	14.865
1:1 + SiO_2	Before	6.18	14.277	1.370	803.297	12.070	31.689	18.718	16.856
	After	6.15	10.383	0.793	711.473	9.606	21.929	14.640	14.705
1:2 + SiO_2	Before	5.99	13.628	1.803	884.205	12.562	35.092	16.888	16.824
	After	5.96	11.176	1.658	634.419	10.838	25.492	14.319	15.668

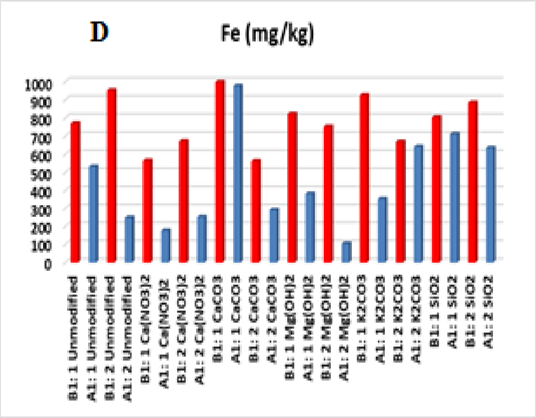
The increase in adsorption capacity of the modified composites was as results of the improved or modified surface characteristics of the composites (Ofudje *et al.*, 2015). The results obtained from composite ratio (1:1, 1:2) modified with Calcium Nitrate ($Ca(NO_3)_2$, calcium carbonate ($CaCO_3$), magnesium hydroxide ($Mg(OH)_2$), potassium carbonate (K_2CO_3), silica gel (SiO_2), There was high adsorption of Cr in composite 1:1 SiO_2 modification which decreases the level of contamination of Cr in the Soil compared to $Ca(NO_3)_2$, $CaCO_3$, $Mg(OH)_2$ and K_2CO_3 (1:1 SiO_2 > $Ca(NO_3)_2$, > $CaCO_3$ > $Mg(OH)_2$ > K_2CO_3. While composite ratio 1:2 the sorption of Cr is high with composite ratio 1:2 $Ca(NO_3)_2$ compared with the other modified composites, (1:2) $Ca(NO_3)_2$ > $CaCO_3$ > SiO_2 > K_2CO_3 > $Mg(OH)_2$). The modification of the composites with SiO_2 and $Ca(NO_3)_2$ improved adsorption capabilities. $Ca(NO_3)_2$ dissociates into Ca^{2+} and NO^{3-}, Ca^{2+} can facilitate the formation of complexes with Cr leading to higher absorption. SiO_2 have high surface area and contain silanol groups (-SiOH) on it surface increasing adsorption sites for Cr thereby leading to increased Cr adsorption. Santos *et al.* (2012) modified sugarcane bagasse with NaOH for the adsorption of Cr, the result obtained shows maximum adsorption capacity of Chromium increased from 20.34 mg g^{-1} by unmodified sugarcane bagasse to 58.00 mg g^{-1} by modified sugarcane bagasse. Modified composite ratio (1:1) $CaCO_3$ adsorbed more Zn than other sorbents. The modified sorbent with calcium carbonate ($CaCO_3$) possesses ion exchange properties. The calcium ions on the sorbent surface exchange with zinc ions in the soil, effectively reducing the concentration of Zn in the soil and immobilizing it on the sorbent. The affinity of calcium ions for zinc is higher than that of some other metal ions, leading to preferential absorption of zinc. Sorption of Cd, Pb, Ni, Mn and Fe shown in Fig. 2 (a-f) is high with modified composite ratio (1:1) $Ca(NO_3)_2$ than the other the modified composites while modified composite ratio (1:2) there was high adsorption of Cd, Pb, Ni, Mn and Fe with $Mg(OH)_2$. Calcium nitrate and magnesium hydroxide have ion exchange properties, allowing them to exchange their cations with the metal ions present in the soil. The exchange capacity of these sorbents might be higher for Cd, Fe, Ni, and Mn, leading to more efficient removal compared to other sorbents without strong ion exchange capability. And also it is because the modified sorbent have high surface area and contain functional groups that have affinity for Cd, Pb, Ni, Mn and Fe ions, these heavy metals can form complexes with the sorbent resulting in more effective heavy metal removal.

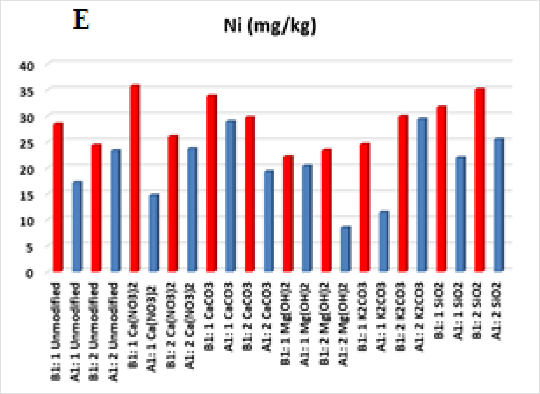
In the presence of magnesium hydroxide, the metal ions are converted into insoluble metal hydroxide precipitates which increases their absorption capacity and efficiency. Hayati and Mahmoodi, (2012) studied the effect of surface modification on activated carbon using NaOH for the adsorption of red (Acid Red 14) and blue (Acid Blue 92) dyes in an aqueous solution. The results obtained from their study show that the activated carbon treated with NaOH improved the surface properties of the adsorbent, increasing the adsorption capacity of sorbents from 2.50 and 0.69 mg/g to 9.17 and 11.77 mg/g for Acid Red 14 and Acid Blue 92 dyes respectively. And also, Ofudje *et al.* (2015) in their study employed the unmodified as well as alkaline-modified coconut shaft as biosorbents for Pb^{2+} removal from aqueous solution. The increase in the adsorption capacity of alkaline treated coconut shaft in their study was as a result of the improved surface characteristics of the adsorbent.











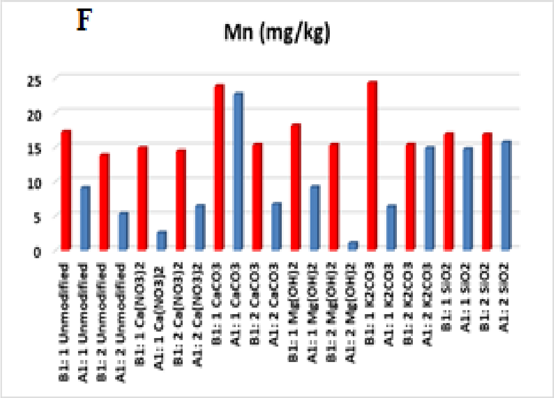


Fig 2 (A-F): Concentration of heavy metals sorbed by various composites

Conclusion: The research focused on the effect of chemically modified biowaste-low density polyethylene waste composites prepared in a 1:1 and 1:2 ratios for sorption of heavy metals in soil samples. Data obtained show reduction of values of metal concentration across board, before and after treatment with sorbents, and the sorption capacity of the modified sorbents was higher than that of the unmodified sorbents. Results showed that sorbents' surface modification increased the surface characteristics thereby reducing the concentration of heavy metals and increasing the sorption of the heavy metals from contaminated soils. Each combination of composite ratio and modifying agent creates a unique sorbent with distinct sorption behaviors for different metals. Overall,

Declaration of Conflict of Interest: The authors declare no conflict of interest.

Data Availability Statement: Data are available upon request from the corresponding author.

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