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Evaluation of Agro-based Adsorbents for Oil Spill Remediation in Freshwater and Saltwater Environments: Kinetic and Adsorption Model Analysis

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Abstract

Environmental pollution caused by oil spills poses significant risks to both human health and ecosystems, particularly in regions like Nigeria's Niger Delta, where oil spills are frequent. Conventional methods for oil spill cleanup have limitations, which has prompted research into alternative, more effective techniques. This study investigates the use of agro-based materials, specifically plantain and banana species, combined with clay soil as adsorbents for oil removal in freshwater and saltwater environments contaminated with Premium Motor Spirit (PMS) and Automotive Gas Oil (AGO). The adsorbents were prepared using a calcination method and their effectiveness in adsorbing pollutants such as ammonia (NH_3), potassium (K), sulfur (S), copper (Cu), manganese (Mn), and phosphorus (P) was evaluated over time. Various kinetic models, including Langmuir, Freundlich, and Temkin, were applied to analyze the adsorption behavior. The results indicate that the adsorbents exhibited substantial removal of contaminants, with the efficiency varying depending on the adsorbent type, contaminant, and environmental conditions. The adsorption process showed a clear trend of initial rapid adsorption, followed by saturation and reversibility in some cases. These findings suggest that agro-based adsorbents could offer a cost-effective and sustainable solution for mitigating oil pollution in water bodies.

Keywords: Evaluation, Agro-based adsorbents, oil spill remediation, freshwater, saltwater environments

INTRODUCTION

A major issue facing the oil industry is environmental pollution, which can result from operational challenges, accidents, waste discharge, sabotage, and more recently, illegal artisanal refining of petroleum, as seen in Nigeria's Niger Delta. It is estimated that around 3 million metric tons of oil and its products are released into water bodies each year [1], which is a significant amount. The Niger Delta is considered one of the most polluted regions globally [2]. Reports indicate that between 1976 and 2015, there were around 16,476 oil spills, with about 3 million barrels of oil dumped into the environment. Unfortunately, about 70% of these spills were not recovered [3]. Pollution leads to severe consequences for both people and the environment, including health risks and ecosystem damage.

Since these issues cannot be entirely avoided, humanity must focus on finding ways to remove pollutants from the affected areas. Several traditional methods for cleaning up spills include manual clean-ups, the use of booms, skimmers,

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sorbents, burning, dispersants, natural methods, and bioremediation. Each of these techniques has its advantages and disadvantages. For example, while burning may offer a rapid cleanup method, it causes additional environmental problems. This drives the continuous search for improved cleanup techniques [4].

Recent advancements include the use of microorganisms for biodegrading pollutants. Bio-augmentation, a technique where microorganisms such as bacteria and fungi are added to polluted environments to break down contaminants, has been introduced [1-3]. Many studies have been conducted to find the most effective and cost-efficient ways to eliminate pollutants. One widely used cleanup process is adsorption [5]. Adsorption is a surface process where molecules are transferred from a fluid to a solid surface, either through physical forces or chemical bonds. Typically, this process is reversible, with the reverse process called desorption. Adsorption is often described using equations called isotherms (such as the Langmuir and Freundlich equations) that quantify the amount of substance on the surface based on the fluid's concentration. Temperature, an important environmental factor, influences adsorption and its parameters [6].

Adsorption is an effective separation method used in industries like chemicals, petrochemicals, and pharmaceuticals. It can be classified as either physical or chemical, based on the mechanism involved. Physical adsorption occurs due to van der Waals forces, while chemical adsorption happens through chemical bonds [7-9]. Adsorption involves the phenomenon where gases or solutes are absorbed onto solid or liquid surfaces. Molecules or atoms on a solid surface have residual energy from unbalanced forces. When certain substances come into contact with the surface, they are attracted and remain there. Depending on the forces at play, adsorption can be physical (driven by intermolecular forces like van der Waals) or chemical (driven by chemical bonds). Physical adsorption generally occurs at low temperatures, has a fast adsorption rate, low adsorption heat, and is non-selective. The weak intermolecular attraction causes minimal changes to the adsorbate molecules, and the adsorption energy is small, making it easier to separate the adsorbed substance. In contrast, chemical adsorption involves stronger forces and is typically more difficult to reverse [10, 11].

METHODOLOGY

Silicon Oxide Test Procedure

10 cm^3 of air-dried, sieved soil (particle size < 2 mm) was placed into a 75 ml plastic extraction bottle. To each bottle, 25 ml of a 0.5 M acetic acid extraction solution was added using an automatic dispenser, and the bottle was capped. The mixture was left to stand overnight (approximately 20 hours). Afterward, the bottle was shaken on a reciprocating shaker at 120 OPM for 50 minutes. The solution was then filtered, and the extract was collected into plastic containers. The ICP was standardized, and the silicon (Si) concentration in the soil extracts was measured.

To calculate the Si content in the soil, use the following formula:
Si (mg/L) in soil = Si in soil extract (mg/L) * 2.5. (3.6)

Some soil testing laboratories in the Northeast have begun offering a modified version of the acetic acid test for Si, replacing colorimetric Si determination with ICP analysis. This modification is based on a method that has been used for years by the University of Florida soils laboratory. Since glassware can leach silicon into samples, it is essential to use plastic containers for Si analysis [4-5].

Electrical Conductivity (EC) Test

Electrical conductivity (EC) increases with temperature. While the ideal temperature for measurement is 25°C, EC can also be determined at other known temperatures using the appropriate temperature coefficients. EC determination is commonly used for diagnosing, surveying, and monitoring soil salinity and assessing the effectiveness of leaching and drainage systems [6].

Sample Preparation for EC Measurement

Air-dry the soil, or dry it in an air-forced oven at temperatures below 35(±5)°C. Then, grind and sieve the sample to a size of ≤ 2.0 mm.

Calibration of Conductivity Meter

To calibrate the conductivity meter, follow the instrument's instructions, using a NIST or equivalent traceable 0.147 dS/m (or higher concentration) KCl solution.

Procedure for EC Measurement

i. Calibrate the conductivity meter according to the manufacturer's guidelines.
ii. Rinse the conductivity cell thoroughly with deionized or distilled water.

EC Determination

Weigh 20 g of air-dried soil and transfer it into a 250 ml polyethylene bottle. Add 100 ml of deionized or distilled water (1:5 w/v ratio) to the container, close it with a bottle cap, and place it horizontally in a reciprocating shaker. Shake the mixture for 60 minutes at 180 oscillations per minute. After shaking, remove the bottle from the shaker and allow it to stand for 30 minutes. Carefully immerse the conductivity probe into the supernatant without disturbing the sediment. Take the conductivity reading once it stabilizes. Rinse the probe thoroughly with deionized/distilled water and blot off any excess water. Report the electrical conductivity (EC) in dS/m at 25°C. Ensure that the sample is at room temperature (20–25°C) during measurement.

Conceptual Scanning Electron Microscope (CSEM) Approach

The Conceptual Scanning Electron Microscope (CSEM) technique was employed to examine the morphological characteristics of the raw materials used for adsorbent formulation. This process involves preparing the samples by removing debris, followed by scanning to assess their morphology. The aim is to understand the structure of the materials and evaluate their potential for oil adsorption.

Conceptual Procedure for X-Ray Fluorescence (CPXRF) and Energy Dispersive X-Ray Fluorescence (EDXRF)

In this study, Energy Dispersive X-Ray Fluorescence (EDXRF) was used to identify the elements present in the adsorbent materials. The XRF analysis was conducted using the SKYRAY EDX3600B X-ray fluorescence spectrometer, which provides fast and precise analysis of complex compositions. The instrument is capable of detecting elements ranging from magnesium (Mg, atomic number 12) to uranium (U, atomic number 92) with high resolution.

Sample Preparation: Non-homogeneous samples were crushed to achieve a fine, uniform size and then pelletized.

Testing Procedure

i. Initial preparation.
ii. Calibration and initialization using a pure silver standard.
iii. Selection of the appropriate calibration curve for the analysis.
iv. Testing of the samples.
v. Data collection and analysis using Excel output.
vi. Finalization of the process.

Surface Area Analysis of Samples

The surface area of the raw materials used for adsorbent formulation was determined using a Micrometrics Instrument Corporation Flowsorb 23000, based in the USA. Krypton gas was used for single-point surface area measurement. Samples were dried at 400°C for 2 hours, then cooled to room temperature before analysis. Surface area was evaluated by converting the obtained value into specific surface area (m^2/g) by dividing the result by the sample's weight.

Experimental Procedure for Fresh and Saltwater Environments

Freshwater was tested to ensure that any residual oil on the agro-based material was removed. The formulated adsorbents were then exposed to freshwater contaminated with Premium Motor Spirit (PMS) and Automotive Gas Oil (AGO). The performance of the adsorbents was monitored by measuring changes in Total Petroleum Hydrocarbons (TPH), ammonia, nitrogen, iron, sulfur, copper, and other pollutants. The ability of the adsorbents to remain intact in the water without dissolving was observed, along with their effectiveness in reducing contaminant concentrations.

For each experiment, 100 ml of PMS or AGO was added to 1 liter of water in a plastic container. A 10 g sample of the formulated adsorbent, with various mix ratios and particle sizes, was immersed in the contaminated water. The mixture was stirred and maintained at room temperature throughout the experiment. At 1-hour intervals over 6 hours, small samples were collected for analysis. The adsorbent was removed and weighed after each interval, and the sample's concentration was analyzed. After each measurement, the adsorbent was returned to the container for further analysis. All samples were stored in a cool environment at 4°C to prevent any changes in composition. The initial weight of the plastic containers, along with the weight of the pollutant and adsorbent, was measured to calculate the total process weight.

At each 1-hour sampling, the mixture was stirred before collecting a sample for gas chromatography (GC) analysis. The weight of the mixture after removing the adsorbent was recorded, along with the final weight of the contaminated container after the adsorbent was replaced. The procedure ensured accurate tracking of the contaminant removal process.

Kinetic of Langmuir Model

The concept of Langmuir Adsorption Isotherm Model was used in evaluating the adsorption process. The concept of Langmuir model expressed the adsorption at the stage of isothermal state and the expression is given as:

$$\frac{Ce}{qe} = \frac{1}{K_L} + \frac{a_L Ce}{K_1} \quad (1)$$

where, Ce = concentration of absorbate at equilibrium (mg/l),

q_e = Amount adsorbed per unit mass of the adsorbent (mg/g),

K_1 = Affinity between adsorbent and adsorbate and a_L= Langmuir constant related to the energy of adsorption

The kinetic of Langmuir model was obtained from Atef (2009).

Kinetic of Freundlich Model

The concept of kinetic model of Freundlich adsorption was considered and the expression is

$$Log q_e = Log K_f + \frac{1}{n} Ce \quad (2)$$

where, K_f = Freundlich constant linked to the adsorption capacity, and $\frac{1}{n}$ = Freundlich constant corrected to the adsorption intensity

The Weber et al (1974) concept was used in expressing the Freundlich adsorption model kinetic

Kinetic of Temkin Model

The concept of the Temkin model equation is commonly used in describing the equilibrium adsorptive characteristics between binary phases comprising the adsorption system. The concept of Temkin isotherm model was demonstrated (Surya-Dash *et al*., 2020) as given below.

$$q_e = a + bInCe \quad (3)$$

Where, a and d b are constant interrelated to energy and capacity of adsorption.

The results obtained from these models are discussed in the next chapter.

RESULTS AND DISCUSSIONS

Figure 1 shows the variation of Potassium (K), Ammonia (NH_3), Sulphur (S), Nitrogen (N), Copper (Cu) and Manganese (Mn) characteristics in container 1 containing plantain Ogoni red with fresh water medium contaminated with AGO with increase in adsorption time period using adsorbent mixture of 1:9 10% to 90% ratio of plantain Ogoni red (POR) to clay soil sample of both with particle size of 150μm. Table 1 the degree of the parameters shown in Figure 1 revealed decrease in Potassium (K), Ammonia (NH_3), Sulphur (S) and Copper (Cu) from zero hour to 3 hours samples before a sudden increase in the same parameters. This revealed that after 3 hours the adsorbent has attained supper saturated point, hereby reversible process was experienced in which the adsorbed parameters are leached back to the system. For the case of Manganese (Mn), continues decrease in concentration was experienced with increase in time. This revealed that saturation and supper saturation point has been achieved. In the case of Nitrogen (N), an increase was experienced from above zero hour to 2 hours before sudden decrease was experienced and constant values achieved showing saturation points or supper saturated. The Figure 1 further demonstrates the magnitude of parameters in container 1 pollution level with AGO as K > NH_3 > S > Cu > N > Mn.

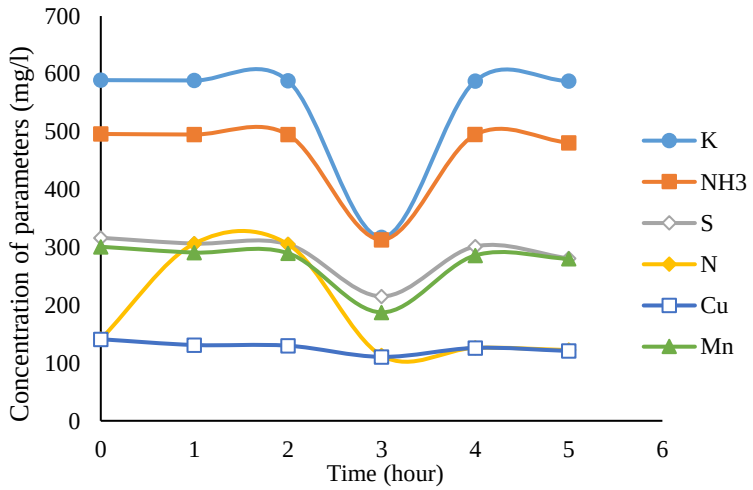

Figure 1. Variation of concentration of parameter versus time for Plantain Ogoni Red in Fresh Water & AGO and clay adsorbent

Table 1. Comparison of Percentage Removal (Adsorbed) of Parameters for Plantain Ogoni Red and Clay Soil Formulated Adsorbent in Salt Water Medium Contaminated with PMS.

Element/ Time (hour)	**Percentage Removal (Adsorbed) of Parameters (%)**				
	1	**2**	**3**	**4**	**5**
Potassium	0.00	0.01	5.16	0.16	0.32
Ammonia	0.13	0.15	3.09	0.00	1.93
Phosphorus	-40.96	-17.83	1.45	3.61	8.43
Nitrogen	7.48	8.33	9.59	11.22	12.51
Sulphur	2.05	2.29	7.48	3.08	7.63
Iron	0.60	0.66	6.42	6.83	8.05
Copper	9.82	10.94	16.56	14.72	16.69
Manganese	0.00	0.06	0.69	7.07	7.99
Silica	24.00	26.76	30.98	36.00	49.87
Chlorine	9.18	10.23	12.45	14.05	17.35

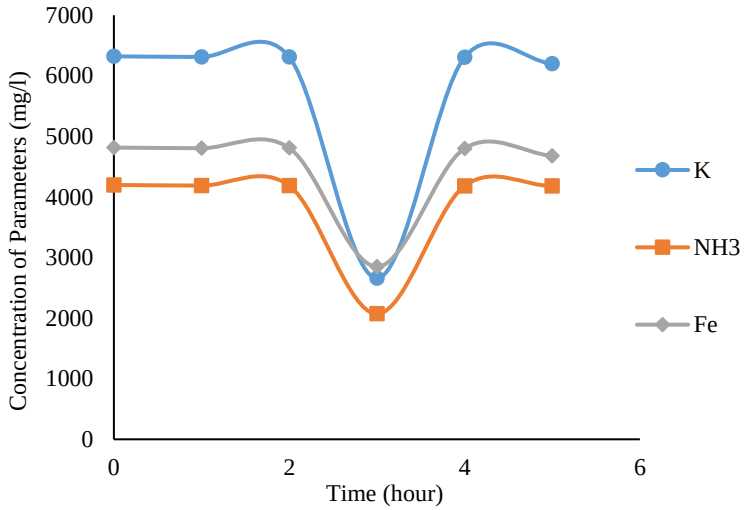

Figure 2. Variation of concentration of Potassium (K), Ammonia (NH3) and Iron (Fe) versus time (hour) for Plantain Ogoni Red in Salt Water & PMS and clay adsorbent

Figure 2 depicts the results of an adsorbent mixture with a 600 μm particle size plantain Ogoni red (POR) clay soil sample . Potassium (K), ammonia (NH3), and iron (Fe) concentrations decreased with increased contact time for a period of more than one hour to three hours, and a sudden increase in the concentration of the parameters under investigation was observed, revealing the effect of the adsorbent's leachate into the fresh water container 1. The outcome further demonstrates that, both in the forward and backward adsorption processes, the concentrations of potassium (K) and iron (Fe) are greater than those of ammonia (NH3). The research further revealed that the formulated adsorbent mixture possesses the characteristics of adsorbing potassium (K), ammonia (NH_3), and Iron (Fe) for the period of 3 hour effectively without reversable process taking place in the medium. The same process of calcine was carried out during the preparation of the adsorbent.

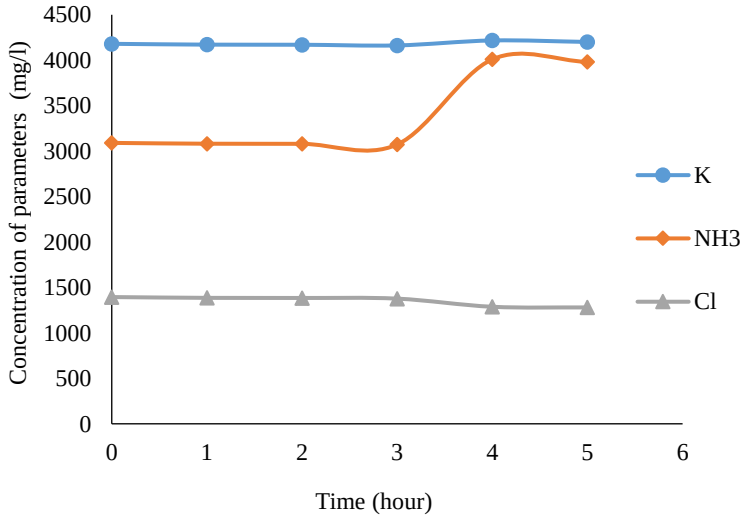

Figure 3. Variation of concentration of Parameters versus time (hour) for Plantain Agbagba in Salt Water & PMS and clay adsorbent

Figure 3 shows an increase in Potassium concentration from zero hour to 3 hour, followed by a sudden decrease in hour 3 with a slight increase in contact time hour 4, before a decrease took effect in hour 5. Whereas, Ammonia (NH3) demonstrated a linear flow from hour 0 to hour 2 and then a decrease was observed in hour 3 before a sharp increase in hour 4 and slight decrease in hour 5. Also, Chlorine shows a very smooth decrease from hour 0 to hour 3 before a noticeable decrease in hour 4, before attempting

to increase in hour 5 for plantain Agbagba with clay soil samples of particle size of 150μm in container 1 contaminated with PMS in salt water medium. Table 2 the increase and decrease in parameters demonstrated may have been brought on by the adsorbent's leaking into the contaminated medium (salt water) prior to the adsorption process of the adsorbent starting to adsorb the parameters in the medium for the duration of one hour or two hours. The binding strength of the adsorbent was able to survive for a duration of more than 5 hours. The adsorbent was produced utilizing the calcine method at temperatures between 500°C and 600°C. The obtained result does, however, highlight the importance of the produced adsorbent in treating contaminated salt water with PMS.

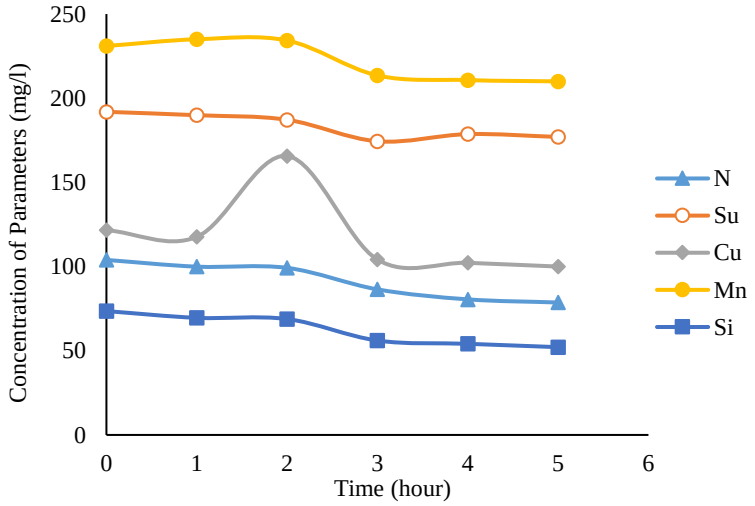

Figure 4. Variation of Concentration of Parameters versus Time (hour) in container 4 containing Banana Ogoni Red in Fresh Water & AGO and clay adsorbent (R4:6, 1.18mm).

Table 2. Comparison of Percentage Removal (Adsorbed) of Parameters for Plantain Agbagba 1 and Clay Soil Formulated Adsorbent in Salt Water Medium Contaminated with PMS.

Element/ Time (hour)	Percentage Removal (Adsorbed) of Parameters (%)				
	1	2	3	4	5
Potassium	0.22	0.24	0.42	-0.88	-0.50
Ammonia	0.29	0.33	0.57	-29.75	-28.82
Phosphorus	-76.92	-40.00	32.62	38.46	63.69
Nitrogen	0.07	1.08	14.76	8.30	10.12
Sulphur	2.98	3.38	5.80	38.52	39.49
Iron	4.81	5.45	9.35	28.50	29.46
Copper	4.47	5.07	8.70	10.11	11.49
Manganese	2.94	3.33	5.72	33.27	34.39
Silica	14.29	16.01	26.44	29.73	32.96
Chlorine	0.65	0.73	1.26	7.67	8.20

Figure 4 shows the variation of Nitrogen (N), Sulphur (Su), Manganese (Mn), Copper (Cu) and Silicon (Si) versus Time. These parameters were into container 4 containing banana Ogoni Red specie with fresh water sample contaminated with AGO. The ratio of the absorbent was R4:6 with particle size 1.18mm and it shows the parameters decrease with an increase in time. The hour under review was from hour 0 to hour 5. The result demonstrated that the concentration of Manganese of Banana Ogoni Red specie in fresh water contaminated with AGO was higher than Nitrogen, Sulphur, Copper and Silicon as shown in Figure 4.

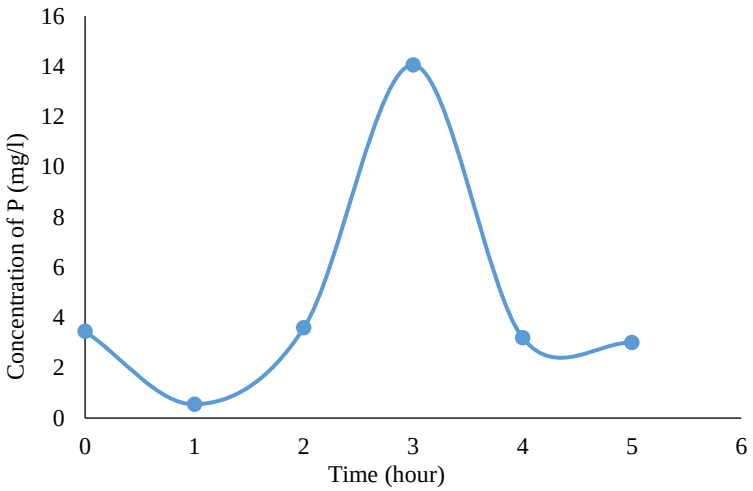

Figure 5. Variation of Concentration Phosphorus (P) versus Time (hour) for Banana Ogoni Red in Fresh Water & AGO and clay adsorbent.

Figure 5 shows decrease in Phosphorus (P) concentration with increase in contact time for mixture of formulated adsorbent ratio of R2:8 of Banana Ogoni Red of 10% to clay soil of 90% of particle size of $300\mu m$. The reduction in parameter concentration indicates that the adsorbent is successful in reducing these measured parameters. Since the container was contaminated with AGO, the medium and adsorbent were combined to raise a drop-in process parameter. The variations in the parameters in Figure 5 shows that no reversible condition was seen, which further shows that the saturation threshold has not yet been achieved.

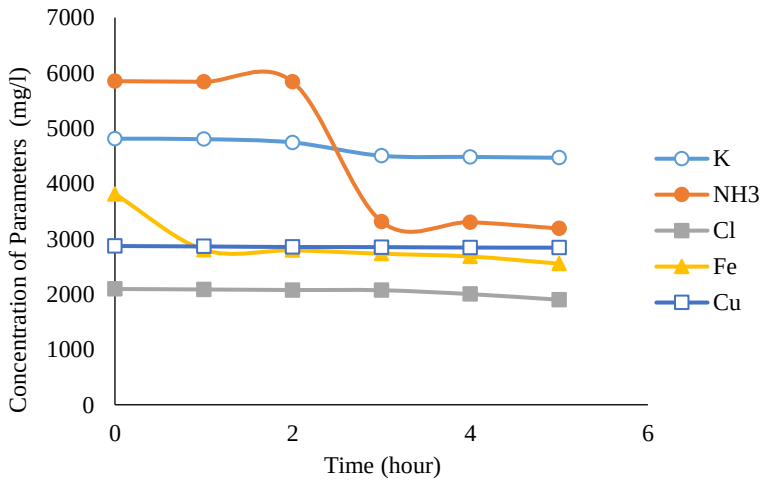

Figure 6. Variation of Concentration of Parameters versus Time (hour) for Native Banana in Salt Water & AGO and clay adsorbent (R1:9, 150μm).

Figure 6 shows decrease in Potassium (K), Ammonia (NH3) and Chlorine (Cl), Iron (Fe) and Copper (Cu) concentration with increase in contact time for mixture of formulated adsorbent ratio of 1:9 of Native banana of 10% to clay soil of 90% of particle size of $150\mu m$. The reduction in parameter concentration indicates that the adsorbent is successful in reducing these measured parameters. Since the container was contaminated with AGO, the medium and adsorbent were combined to raise a drop-in process parameter.

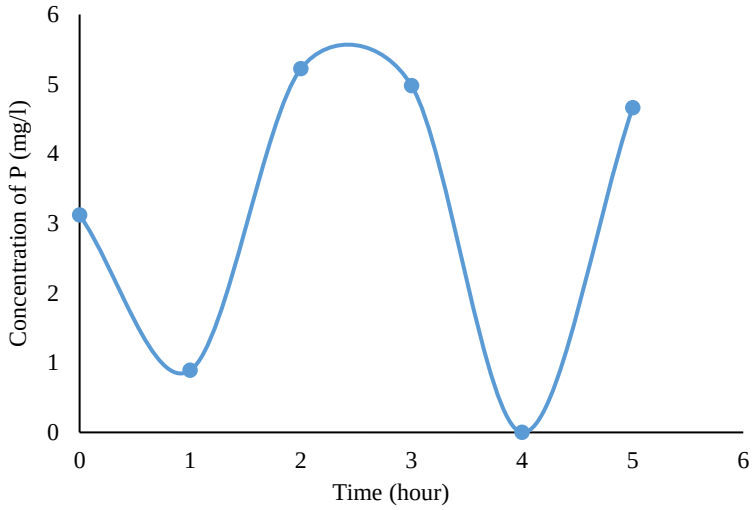

Figure 7. Variation of Concentration of Phosphorus versus Time (hours) for Native Banana in Salt Water & AGO and clay adsorbent.

Figure 7 shows the variation of the concentration of Phosphorus versus contact time in container 1 of native banana in salt water contaminated with AGO. The analysis was carried out using the clay adsorbent ratio 1:9 of particle size 150 μm. The result demonstrated a decrease in Phosphorus concentration from 0 hour to hour 1, then a sharp increase occurred between hour 2 and 3 before a sharp decrease in hour 4 and ended up with an increase in hour 5. The same process of calcine was carried out during the preparation of the adsorbent.

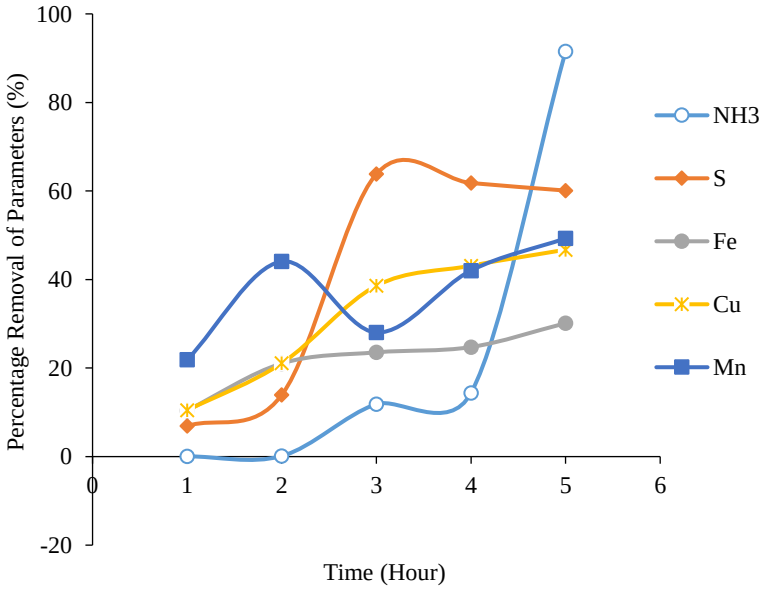

Figure 8. Comparison of Percentage Removal (Adsorbed) of Parameters Versus Time for Banana Ogoni and Clay Soil Formulated Adsorbent in Fresh Water Medium Contaminated with AGO.

Figure 8 shows the comparison of percentage removal or adsorbed of ammonia (NH_3), Sulphur (S), Iron (Fe), Copper (Cu) and Manganese (Mn) with respect to increase in contact time in container 4 containing Banana Ogoni Red and clay soil sample (CSS) formulated adsorbent mixed ratio of 4:6 of particle size of 1.18mm in fresh water medium contaminated with AGO. Increase in percentage adsorption of parameters (NH_3, S, Fe, Cu and Mn) by the adsorbent was experienced within the period of 1 to 3 hours before sudden decrease in contact time.

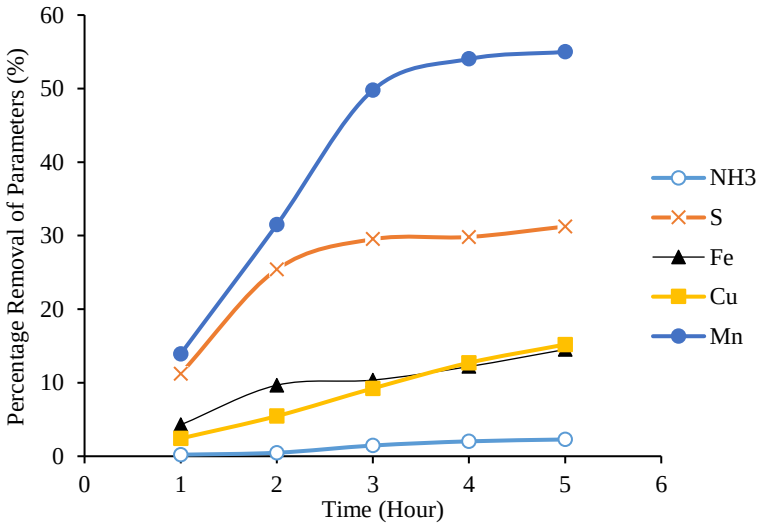

Figure 9. Comparison of Percentage Removal (Adsorbed) of Parameters Versus Time for Native Banana and Clay Soil Formulated Adsorbent in Salt Water Medium Contaminated with AGO.

Figure 9 compares the percentage removal or adsorbed of ammonia (NH_3), sulphur (S), iron (Fe), copper (Cu), and manganese (Mn) with respect to an increase in contact time in native banana and clay soil sample (CSS), which was developed as an adsorbent mixed ratio of 4:6 with a particle size of 1.18mm in salt water medium contaminated with AGO. Before the contact duration abruptly decreased, the adsorbent experienced an increase in the percentage of parameters (NH_3, S, Fe, Cu, and Mn) that were adsorbed.

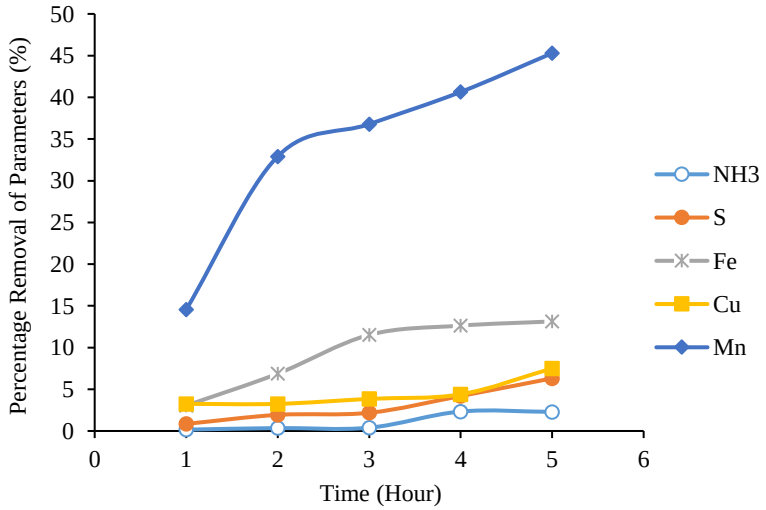

Figure 10. Comparison of Percentage Removal (Adsorbed) of Parameters Versus Time for Native Banana and Clay Soil Formulated Adsorbent in Fresh Water Medium Contaminated with PMS

Figure 10 compares the percentage removal or adsorbed of ammonia (NH_3), sulphur (S), iron (Fe), copper (Cu), and manganese (Mn) with respect to an increase in contact time in container 1 containing native banana and clay soil sample (CSS), which was developed as an adsorbent mixed ratio of 1:9 with a particle size of 150 μm in fresh water medium contaminated with PMS. The parameters all demonstrated increases from the first day to the last day; however, the volume of the percentage removal was in this order; Manganese (Mn) > Iron (Fe) > Copper (Cu) > Sulphur (Su) > Ammonia (NH_3). Increase in percentage adsorption of parameters (NH_3, S, Fe, Cu and Mn) by the adsorbent was experienced within the period of 1 to 3 hours before sudden decrease in contact time.

CONCLUSION

The study demonstrates that agro-based materials such as plantain and banana species, combined with clay soil, can be effective adsorbents for removing petroleum-based contaminants from both freshwater and saltwater environments. The adsorbents were able to significantly reduce the concentration of various pollutants, including ammonia, potassium, sulfur, and metals like copper and manganese, over a period of time. The adsorption process was highly influenced by the type of adsorbent, contaminant concentration, and environmental conditions. The kinetic analysis using Langmuir, Freundlich, and Temkin models revealed that the adsorption followed a pattern of rapid initial removal followed by saturation, with some degree of reversibility in certain cases. Overall, this research highlights the potential of utilizing locally available, low-cost materials for environmental cleanup, particularly in regions affected by oil spills. Further studies are needed to optimize the adsorbent preparation and investigate long-term stability and environmental impacts of these materials in real-world applications.

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