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Surface Water Remediation Using Zinc Oxide/Plantain Peelings Nanoparticles within Ogoni Creek of Niger Delta, Nigeria

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Abstract

This study aimed to evaluate the efficacy of zinc oxide/plantain peelings nanoparticles for oil spill contaminated surface water remediation within Ogoni creek of Niger Delta, Nigeria. The nanoparticles were characterized using scanning electron microscope (SEM) and X-ray Powder diffraction (XRD), taking in cognizance of the physicochemical parameters prior and after analysis so as to ascertain efficacy of the remediation method. SEM revealed the image of an irregular morphology and a porous surface which can enable oil entrance into the internal parts of the nano materials for easy sorption purposes. The XRD revealed a hexagonal wurtzite structure with a particle size of 22nm. The synthesized nanoparticles exhibited adsorption properties when used on the surface water samples showing percentage remediation ranging between 2.38 - 8.56% (SWa); 2.16 - 8.83% (SWb); and 53.33-77.62% (SWc). The results showed that the plantain peelings/ZnONPs had good efficacy for oil spill removal in the contaminated surface water samples. Kinetics of remediation gave a linear graph and took a pseudo-first order reaction with R^2 values close to 1. (R^2 = 0.978, 0.985 and 0.963). ANOVA revealed that there is a significant difference ($p<0.05$) at 95% confidence limit in the diminishing trend of the surface water contaminated samples. It is therefore recommended that nano materials harnessed from biomass materials in conjunction with other green synthesis should be employed for water remediation.

Keywords: Adsorption, Nanoparticles, Physicochemical, Remediation, Surface water

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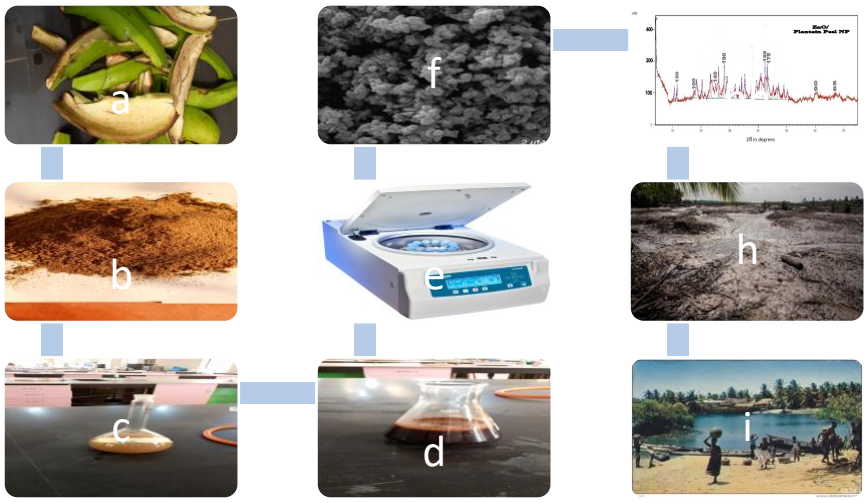
Graphical Abstract 1



Plantain peels (b) Dried and ground plantain peels powder (c) Filtrate of plantain peel extract (d) Filtrate + Zinc acetate (e) Centrifuge and oven dry (f) SEM (g) XRD (h) Oil spill contaminated water site

1.0 Introduction.

Extensive oil spill contamination and degradation of surface water and its resulting detriment to green environmental actualization has remained a challenge since the discovery and exploration of crude oil in Niger-Delta, Nigeria. Oil spills often resulting from unforeseen disasters [1], accidental leakages [2] and youth restiveness [3] have been recorded over the years in the Niger Delta zone of Nigeria. Spill experts have estimated about 30-50% of oil spills to be caused either directly or indirectly by human error while 20-40% is caused by equipment failure or malfunction [4]. Natural conditions for degradation of petroleum and its related products are favourable with high temperatures and high rainfall, the recovery of contaminated areas proves difficult due to nature and extent of contamination [5].

Serious global health impacts ranging from physical and mental disorders, organ dysfunction, neurological disorder, respiratory problem cancer, reduced life expectancy, weakening of the body's immune system, respiratory problems and death have often resulted from

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oil spill and related issues [6]; [7];[8] while other health related issues emanating from oil pollution may involve high levels of emotional stress and psychological distress resulting from living under such environmental adversity[9].

Remediation of oil spills is a serious issue due to contaminants adverse effects on the biosphere. Oil spreads on the top surface of water and form a horizontal smooth and slippery surface known as slick. This forms a thin coating on bird's feathers and as such causes it to lose its insulating properties and subsequently results in its freezing and death. It may also reduce the amount of dissolved oxygen in water necessary for marine life sustainability. Oil spill has toxic impact on aquatic animals and damages their food resources and habitats. It may also result in devastation of land and vegetation and subsequently leads to poor yield in agricultural productivity Therefore, proper remediation must be done after oil spillage [10].

Methods employed in remediating water differs and often depends on factors such as adequate procedure, type of contaminants involved and nature of contaminated site. An eco-friendly and sustainable approach towards the environment has introduced many low-cost, non-toxic and biodegradable materials along with different biomasses to make micro-to nano-sized materials, membranes and aerogels for the sole purpose of oil contaminated water treatment and oil recovery [11].

Contaminated water may involve the use of economical and environmentally – friendly sorbent materials in oil spill treatment and may allow the recovery of the oil and reuse of these sorbents [12]. While conventional methods often applied for water clean-up may include physical, chemical, thermal and biological methods [13,14], these conventional methods have moved oil experts a step forward in remediation advancement, since they are not adequate enough to solve the problem of massive oil spills as most often, technical difficulties are encountered in the process [15].

The synthesis of Kapok fiber using Polybutylmethacrylate silica nanoparticles [16]; cotton/SiO_2 nanoparticle modified with Octadecyltrichlorosilane [17] and Kapok fibre based on Fe_3O_4 nanoparticles with Dopamine modification [18] for oil sorption in water medium has been reported.

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The sorption capacity of Plantain peels as low cost agricultural wastes is a promising need for oil spill remediation and agricultural waste management.

. Therefore the necessity to enhance the applicability of nanomaterials in remediating oil spilled surface water in the Niger Delta region of Nigeria is of paramount importance.

1.1 Statement of Problem

The Niger Delta region of Nigeria has faced unprecedented negative environmental impacts in recent times due to the menace caused by oil spill. This region which is situated on the Gulf of Guinea on the Atlantic Ocean in Nigeria [19], is made up of Abia, Akwa Ibom, Bayesa, Cross River, Delta, Edo, Imo, Ondo and Rivers State. The region consists of diverse ecosystems of mangrove swamps, fresh water swamps, rain forest and is the largest wetland in Africa and has been among the ten most important wetland and marine ecosystems in the world [9]. The Niger Delta region is noted with a remarkable history of oil and gas exploration dated back from 1956 but as a result of frequent oil spillage in both terrestrial and aquatic environment, poor vegetation, surface and ground water contamination, and land degradation has rendered the region a complete devastated wasteland. These have caused a substantial retrogression in agricultural activities off the coastal waters and vegetation. There is acute food shortage, malnutrition and death which has devastated the Nigerian economy.

2.0. Materials and Methods

2.1. Collection and preparation of plantain peel extract

Plantain peel extract, Musa Paradisiaca was obtained from Ogbogono market in Asaba metropolis. The peel was removed and washed with clean water and chopped into smaller sizes. This was air dried for two weeks, mascerated into fine powder with a high speed multipurpose blender**.**

2.2. Biosynthesis of Zinc oxide Nanoparticles (ZnNPs) using Plantain peel extracts

2.2.1 Synthesis of ZnO Nanoparticles

The synthesis was carried out according the method described by [21]. 1:1 ratio of the zinc oxide and the plantain extracts were mixed in a separate 250 ml Erlymeyer flask. The solution was subjected to continuous stirring and heating at 100 rpm for 4 hours. The resultant nanoparticle solution was purified by centrifugation at 10,000 g for 20 minutes. The supernatants were

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discarded and the nanoparticles pellets collected, washed with distilled water dried and stored at -80^{o}C

2. 3. Sampling

Sampling was carried out strategically in collaboration with local inhabitants within the aquatic environment of Ogoni coastal creek in Niger Delta region of Delta State. Surface water samples were collected using the composite sampling method on 2 hour intervals for a 24 hour duration for analysis.

2.4. Analysis of Physicochemical parameters

Physicochemical parameters such as pH, dissolved oxygen, BOD5, COD etc., of the water samples were evaluated to ascertain the extent of contamination prior to and after remediation. The composite water samples were kept in transparent plastic bottles, and thereafter, sent to the laboratory for physicochemical analyses maintaining all laboratory conditions. The pH of the water samples was determined using a pH meter of model, pHS-25; Turbidity of water samples were determined using Waz-B model turbidimeter after initial calibration of the instrument using the manufacturer's certified reference materials (<0.1, 10, 100, 500 and 1000 Nephelogical Turbidity Unit (NTU), following the procedures lay down in the manufacturers' manual guide and the instrument was turned to zero. Dissolved oxygen of the water sample was determined using a Dissolved Oxygen Analyzer, model JPB – 607 Portable meter after initial calibration following the procedures provided in the manufacturers' manual. This was carried out by employing the method of [20]. The Electrical Conductivity of the water sample was measured using a conductivity meter. The instrument was switched on to stabilize and allowed for 10 minutes. The instrument was initially calibrated using the manufacturer's standard. The probe was immersed into the water sample while completely submerging the holes of the sleeve. Air bubbles trapped in the sleeve were removed by gently tapping the bottom of the cup with a probe.

2.5. Characterization of synthesized ZnO nanoparticles

The dried P-ZnNPs were characterized using PAN analytical Xpert Pro θ-2θ powder X-ray diffractometer. The instrument used a Cu Kα radiation of wavelength = 0.1541nm at 45 kV with a monochromatic filter of ^{o}2 in a scan range of 20-80^{o} with a scanning speed of 6o/min. Estimation of particle sizes was performed by Debye-Scherrer's formula. The functional group present in the

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ZnO/plantain peel extract nanoparticles were confirmed using FT-IR spectrometer vector 22, Bruker, Germany. The pellets were scanned at 4 cm^{-1} resolution in the spectra range of 400-400 cm^{-1} at room temperature. The morphological properties were determined prior to analysis. The synthesized ZnO nanoparticles were mounted on aluminium studs and coated with gold film. Visualization of ZnO morphology was performed using a SEM. The size of particles was then analyzed using the ImageJ Program according to the method of [22].

2.6. Application of plantain-MNPs for Oil Spill Contaminated Water Samples

2 g was put in a 500 ml beaker and then 250 ml of distilled water was added. Various ratios of Plantain-MNPs to contaminated oil spilled soil samples, ranging from 1:1 to 1:50, were added and mixed using a glass rod. A permanent Nd-Fe-B magnet (4300 Gauss) was used to collect the dispersed oil spill after every 10 minutes. The remaining oil was extracted from the medium, using ethyl ether. The efficiency of the Plantain-MNPs on the sample was calculated using the equation below:

$$XE\ (\%) = \frac{V0}{V1}\ \mathbf{X\ 100}$$

where V_0 and V_1 are the volume of the removed and original oil, respectively. The used MNPs are collected by an external magnetic field, washed severally with ethyl ether and then recycled.

2.7. Kinetics of Nano-remediation of the Water samples

The remediation experiment was carried out according to the methods of [23].

The kinetics of nano-remediation of the surface water samples was monitored at hourly intervals. This is to observe the dynamics and ascertain the efficacy of the method. This was achieved by monitoring the changes in the physicochemical parameters of the samples adsorbent rate of solute.

2.7.1Adsorption Kinetics

In order to determine the potential steps which control the adsorption rate in terms of chemical stoichiometry, mass transport process, pseudo-first order pseudo-second order, Elovich and intraparticle diffusion models [24] were used to test the experimental data. As shown in the following equations:

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. $qt = -\exp(-k_1 t)\, qe + qe$…… 1

$$qt = \frac{k_2 * qe^2}{1 + k_2 * qe * t} \quad \text{……2}$$

$$qt = \frac{1}{\beta} \ln(1 + \alpha * \beta * \quad \text{……3}$$

$qt = K_{ID} \sqrt{t} + 1$……4

where t is the contact time in hour; k1 is the first order rate constant (h-1); k2 is the rate constant of the second order (h/l); qt is the amount of adsorbate in the adsorbentat time t (mg/g); + α is the initial adsorption rate of the Elovich model (mg/g/min) and + β is the adsorption constant of the model (g.mg-1); KID is the intraparticle diffusion rate constant; t is the time of contact (min) and I is the intra-particle diffusion kinetic model intercept.

Statistical treatment was achieved by preparing samples in triplicates. Statistical tool used was Standard Deviation (SD), Coefficient of Variation (CV) and Analysis of Variance (ANOVA).

3.0. Results and Discussion

3.1. Physicochemical parameters of surface water samples

Table 1:

Data for the physicochemical parameters of the surface water samples for the Go khana and Bodo city

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Sampling sites	pH	Temp ^{O}C	EC (µS/cm)	TDS mg/l	COD (mg/l)	Nitrate (mg/l)	Phosphate (mg/l)
SWa_1	7.30 ±0.06	27.40 ± 1.50	356 ± 1.10	142 ± 2.00	235.40 ± 2.50	7.22 ± 0.06	15.50 ± 1.75
SWb_1	7.62 ±0.05	27.80 ± 2.20	366 ± 1.20	210 ± 3.20	232.20 ± 1.00	4.46 ± 0.60	10.80 ± 1.65
SWa_2	7.20 ±0.15	26.50 ± 2.10	385 ± 1.60	115 ± 300	263.50 ± 4.00	3.30 ± 0.05	9.20 ± 2.40
SWb_2	7.28 ±0.02	27.00 ± 1.80	420 ± 1.00	167 ± 4.00	244.20 ± 2.50	4.70 ± 1.30	8.50 ± 0.70
SWc_1	7.50 ±0.03	26.60 ± 1.70	300 ± 1.00	125 ± 2.00	30.70 ± 4.40	2.60 ± 1.50	6.45 ± 1.55
SWc_2	7.10±0.07	25.30 ± 2.40	269 ± 1.40	232 ± 3.50	22.80 ± 2.70	1.85 ± 0.80	6.00 ± 1.35
WHO (2011)	6-5 – 8.5	-	1000	600	200	50	<5

SWa_1: Surface water samples from Eleme SWa_2: Surface water samples from Bodo City
SWb_1: Surface water samples from Tai; SWb_2: Surface water Sample from Gokana
SWc_1: Surface water nanoremediated (Eleme and Bodo City)
SWc_2: Surface water nanoremediated (Tai and Gokana).

Data for the physicochemical parameters of the surface water samples for the Eleme, Bodo city, Tai and Gokana are recorded in table 4. pH values ranged between 7.10± 0.06 - 7.62± 0.05 in all the samples with SWb_2 recording a high pH of 7.62 probably as a result of high concentration of oil spillage and anthropogenic activities emanating from dumping of wastes within that region [25]; [26] had reported that very low or very high pH may be detrimental to aquatic life in general. pH values recorded in this study were within WHO limits of 6.5 - 8.5 and also within the values of 8.60±0.02 reported by [27] in a related research.

Temperature values recorded in this study ranged between 25.30± 2.20 - 27.80± 1.50 across all the samples analyzed. Slight temperature elevation of 27.80 recorded for SWa_1 may be accorded to the extent of oil solubility and dispersants within the water body. A percentage decrease in temperature (8.9%) observed for SWc_1 and SWc_2 could be as a result of effective nanoremediation.

Electrical conductivity which is the ability of solution to conduct electricity is related to ions concentration and total dissolved solids within the water body [28]. EC observed to be 356.00±

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1.10, 366.00 ± 1.20, 385.00 ±1.60 and 420.00 ± 1.00 µS/cm for SWa_1, SWb_1, SWa_2 and SWb_2 respectively were obviously higher than those of the nanoremediated samples (300 ± 1.00 and 269±1.40 µS/cm for SWc_1 and SWc_2 respectively) indicating effective remediation.

The chemical oxygen demand (COD) values recorded in this study ranged from 232.80 ± 2.70 to 263.5 0 ± 1.40 mg/l for SWa_1, SWb_1, SWa_2 and SWb_2.These were higher than WHO tolerance limit of 200 mg/L. After nanoremediation, the samples recorded a lower COD values of 30.70 ± 4.40 and 22.80 ± 2.70 mg/L which was lower than WHO permissible limits Chemical oxygen demand is a measure of organic contamination in water. It is the amount of dissolved oxygen required to cause chemical oxidation of the organic material in water and is a key indicator of the environmental health of surface water. Chemical oxygen demand is a measure of both organic and inorganic agents competing for DO in water.

Mean nitrate values in this study ranged from 1.85 ± 0.80-7.22 ± 0.06, which was far below the WHO tolerance limits of 50 mg/L after remediation. These values were below that reported by [26] Olayinka *et al.*, (2020) who worked on water samples around Atlas cove, Lagos, Nigeria. Phosphate values ranged from 5.35 ± 0.67 to 12.03 ± 0.08 mg/L.

The phosphate values obtained in this study varied between the sampling sites and were higher than the WHO permissible limit of < 5 mg/l and could probably be due to anthropogenic activities.

Scan Electron Microscope (SEM) of Synthesized Nanomaterials

Figure 2a and 2b shows the images of unripe plantain peels and the modified ZnO nanomaterials. The figure shows that it has an irregular morphology and a porous surface which can enable a large contact area between the absorbent surface for sorption purposes and oil droplets and as well give room for oil retention [29].

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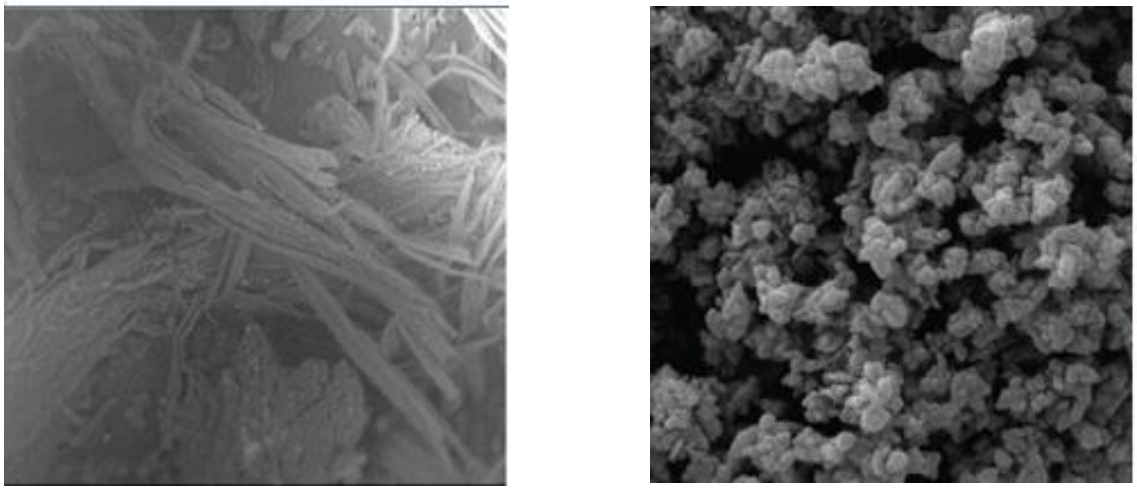
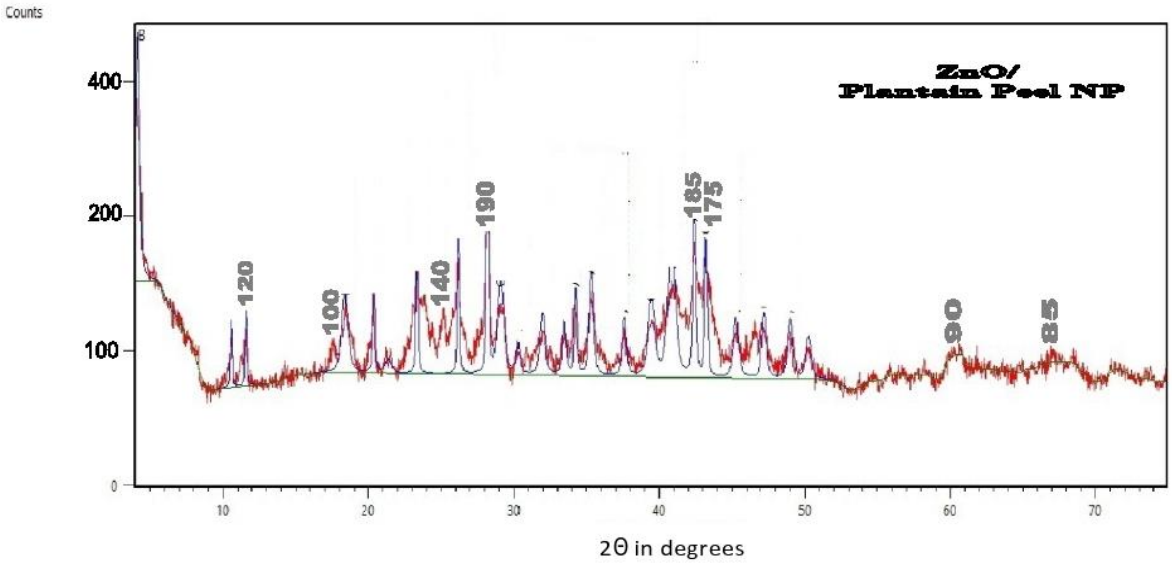


Fig 2: (a) Raw plantain peelings (b) Modified plantain peelings with ZnO

X-ray Diffractogram of synthesized zinc oxide nanoparticles

The X-ray diffratogram (XRD) is shown in figure 3. This is recorded at the range of 2θ with values at 120^{O}, 175^{O}, 185^{O}, 190^{O}. These peaks are indexed at the Zinc oxide wurtzite structure crystallizing in two main forms as cubic zinc blend and hexagonal wurtzite. Other peaks are indexed at 100^{O}, 140^{O}, 85^{O} and 90^{O} for the PPE. All peaks recorded reveals that the synthesized nanopowder was free of interfering impurities devoid of any other characteristic XRD peaks other than that of zinc oxide and plantain peel extract peaks.



Fourier transforms infrared spectroscopy (FTIR)

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Figure 4 shows the FTIR of the PPE and the ZnONPs samples reveals bands at different ranges. The peaks for PPE are at 3,350, 2,890, 2,430, 1620, 1,362 and 1,011 cm^{-1}. The peaks for ZnONPS are at 3330, 2,942, 1594, 1,366 and 1,030 cm^{-1}. The band stretching below 500 cm-1 (432 cm-1) correspond to the Zn-O stretching mode, The band at 1,594 cm-1 correspond to the C-O stretching for esters and alcohols recorded within a range of 1000 cm^{-1}-1300 cm^{-1}. The absorption peak at 2,942 is ascribed to the C-H stretch/bending vibrations in alkanes and the peak at 1594 is the C=C stretch of aromatic rings. These data are in line with the results observed by [30].

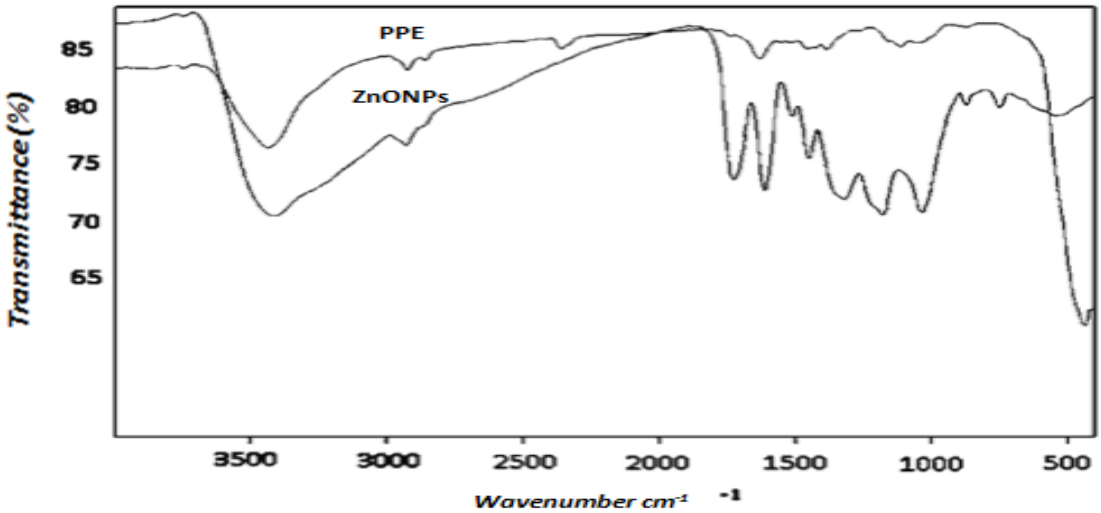

Fig 4: FTIR Spectrum of plantain peel and ZnONPs

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Total petroleum hydrocarbon

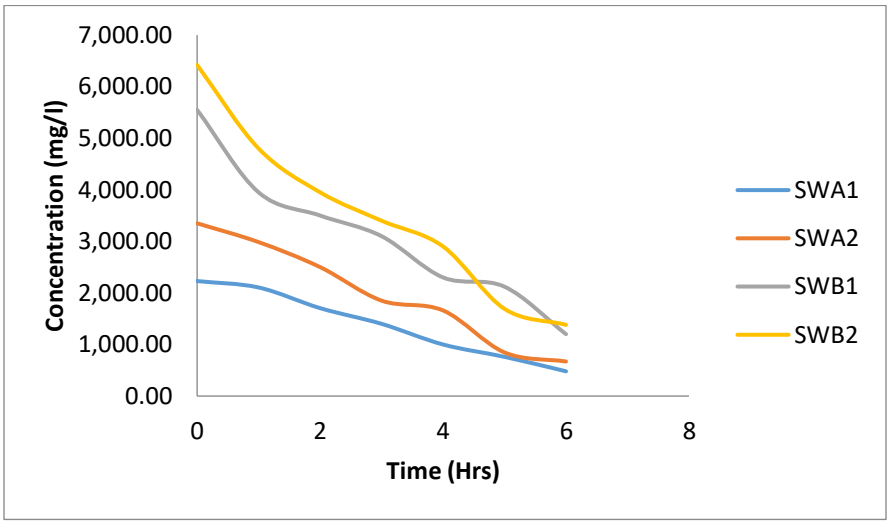

Fig 5: Progressive diminishing trend in TPH removal of SWa_1, SWa_2, SWb_1, and SWb_2 Samples.

Mean concentration of TPH in the surface water samples ranged between 2,230.25± 6.40 and 6,420.00±6.10 mg/l at the commencement of analysis indicating the level of oil contamination in these sites (Figure 5). However, a progressive trend in the removal of TPH was observed across the samples on application of the green synthesis nanoparticles. SWb_2 had the highest mean TPH concentration (6,420.00±6.10 mg/l) from commencement and also retained the highest mean concentration after analysis 1,380. 5±4.50 mg/l. The site is dominated with thick emulsified oil giving total resistance to degradation. The mean levels of TPH observed in this study were slightly higher than those of earlier researchers. [31] reported TPH variations from 90 to 250 µg/L in surface water and sedimments of Qua-Iboe River in Akwa-Ibom.

Extensive oil contamination in Ogoniland, Nigeria has been carried out by [5] and reported Extractable petroleum hydrocarbon of 17,900 mg kg^{-1} in sediments and 8.000 mg kg^{-1} in surface waters within the coast and creeks of the contaminated sites. Department of Petroleum Resources (DPR) (2002) sets its TPH permissible limit in water at 10 mg/L. TPH limit for both drinking water and wastewater discharge is 0.5 mg/L [32] while the EPA has the TPH limit as 0.5 mg/L in water. The concentrations obtained in this study were higher than the limits. This may be because of the nature of the aromatic and aliphatic hydrocarbon present in the water.

The reaction pathways of the contact between the oil samples and plantain peelings synthesized ZnO nanoparticles took a pseudo-first order which is based on the assumption that the rate limiting step is a chemical sorption. The adsorption rate is dependent on the adsorption

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capacity and not on the concentration of the adsorbate as symbolized in the following linear equations

The expression given by Lagergren kinetic model for the Pseudo- first order is given as:
Log10qe-qt = log10qe-kt/2.303……(1)
lnqe/qt = ln (qe – qt)……(2)
ln (qe – qt) = ln (qe – k1t)……(3)

Where qe and qt (mg/kg) are oil spill retention capacity at equilibrium and time t (min), k1 (min^{-1}) is the constant rate parameters of the Lagergren pseudo-first order model based on the adsorption that the rate of change of solute uptake with time is directly proportional to the difference in saturation concentration and the amount of solid uptake with time which is generally applicable over the initial stage of adsorption process [33]. The values of k_1 as well as the adsorption capacity (qt) were determined from the slope and intercept of the plotted curves (Figures 7a-d). Correlation coefficient R^2 were close to 1 in all samples. This observation is constant with findings of [34] who studied the kinetics and thermodynamics for the sorptive removal of crude oil spills using a low-cost chitosan-poly (butyl acrylate) grafted copolymer.

The Elovich kinetic model employed in their study revealed a linear graph with R^2 close to 1.

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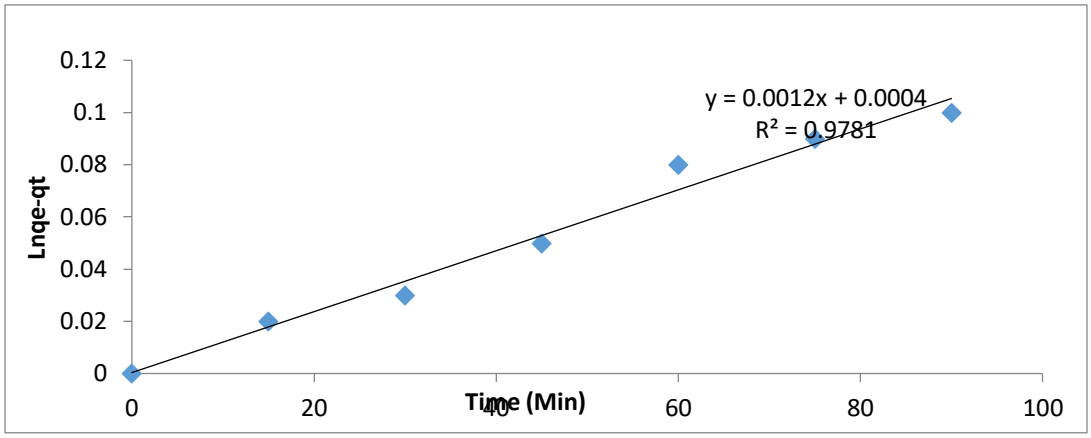

Fig 5a: Pseudo – first order plots for the diminishing trend of TPH in water samples on application of synthesized Plantain peelings extract/ZnO nanoparticles for SWa_1

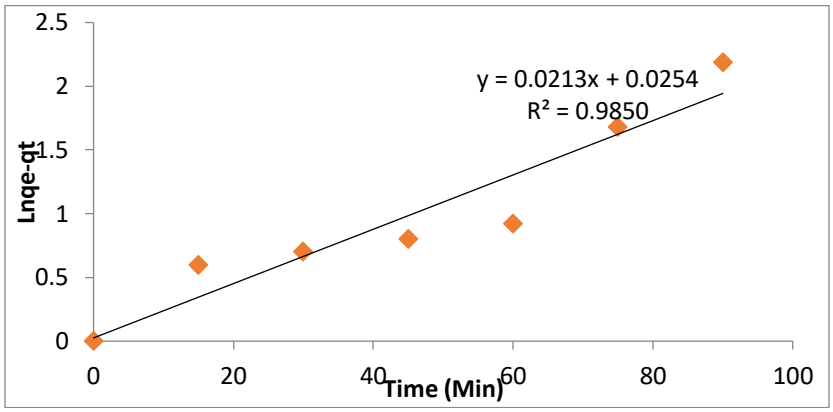

Fig 5b: Pseudo – first order plots for the diminishing trend of TPH in water samples on application of synthesized Plantain peelings extract/ZnO nanoparticles for SWb_1

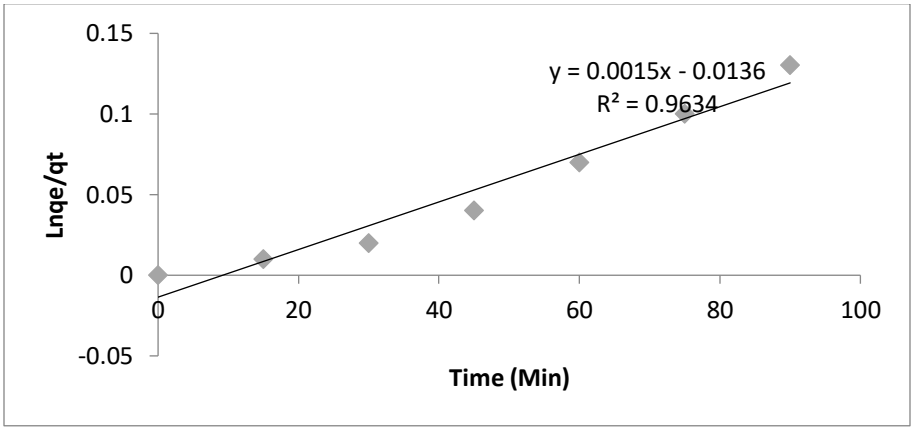

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Fig 5c: Pseudo – first order plots for the diminishing trend of TPH in water samples on application of synthesized Plantain peelings extract/ZnO nanoparticles for SWc_1

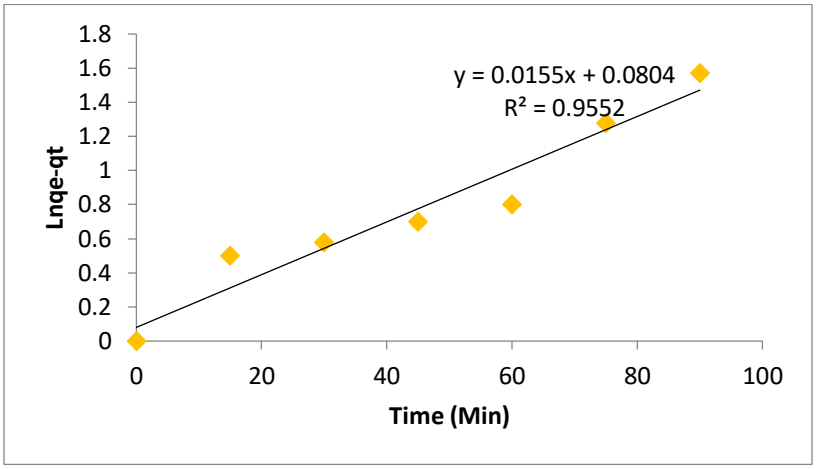

Fig 5d: Pseudo – first order plots for the diminishing trend of TPH in water samples on application of synthesized Plantain peelings extract/ZnO nanoparticles for SWc_2

Conclusion

Difficulties and environmental hazards often attained in the attempt to remediate oil contaminated soils and other affected media has kept the remediation industry in a dilemma of recent. In other to overcome this challenge, green synthesis using plantain peelings *(Musa Paradisiaca)* extract, a cost effective waste material synthesized with ZnO for the remediation of oil spill contaminated water medium has proven to be efficient amongst other remediation technologies. The synthesized plantain peelings/ZnONPs employed in this study exhibited adsorption properties when used on the oil spill contaminated surface water samples. A progressive trend in the removal of TPH was observed across the samples on application of the green synthesis nanoparticles indicating that the synthesized MPE/ZnONPs had good efficacy for oil spill removal in the contaminated surface water samples. SWb_2 had the highest mean TPH concentration (6,420.00±6.10 mg/l) from commencement and also retained the highest mean concentration after analysis 1,380.35±4.50 mg/l as the site is dominated with thick emulsified oil giving total resistance to degradation. (Figure 5).

Recommendation

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Based on the findings from this study, it is therefore recommended that for optimum remediation efficacy, most especially in oil saturated water medium where there are increase in oil and emulsion densities low cost effective and environmentally-friendly modified bio-based waste materials, may be employed for optimum degradability of bio recalcitrant pollutants.

Efforts should also be made by concerned bodies to adopt a collaborative approach which may involve combined remedial techniques as most of the sites under study have been heavily impacted with oil contaminants including the mangroves, creeks and rivers and as such, effective remediation would take some time to be achieved.

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