Comparative Study of Nano-Particles of Magnesium Oxide and Cow Dung for Hydrocarbon Polluted Soil Remediation

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Abstract

A comparative study of Nano particles of Magnesium Oxide and Cow dung for the remediation of hydrocarbon polluted soil was carried out in this research. The cow dung and loamy soil sample were obtained within Agricultural farm in River State University and the Magnesium Oxide particles were produced in Chemical Engineering laboratory, River State University. 300g of Loamy soil was weighed into 14 rectors. Out of 14 samples, 12 samples containing 300g of loamy soil were polluted with 100ml crude, while the remaining 2 served as control unpolluted soil. The 12 polluted sample were stirred for uniform mixing, 50ml of distill-water were added and stirred then left for 3 days. After 3 days, the samples were taken to the lab to analyze the following parameters; TPH, Bacterial, pH, density, moisture content and particle size. 3 days later the Nano-particles of Magnesium Oxide of masses of 5g, 10g, 15g, 20g, 25g and 30g were introduced into 6 separate reactors and also Cow-dung of masses 5g, 10g, 15g, 20g, 25g and 30g were introduced into 6 separate reactors, 2 samples of polluted soil were set as control. Each of the 14 samples were taken to the lab to analysis the TPH, Bacteria and pH for 1st week – 6th week in 45 days. At the end of the analysis, the percentage degradation of TPH in the 30g loamy soil sample obtained by biological method (cow dung) was 97.49%, 98.12%, 98.25%, 98.78%, 99.11% and 99.66% respectively while chemical method (magnesium oxide) was 91.35%, 93.62%, 97.05%, 98.36%, 98.97% and 99.09% respectively. The first order degradation rate constant, obtained across the treatment options ranged from 0.0159– 0.01243day^{-1} for biological method and 0.00161 – 0.1601day^{-1} for chemical method. The half-life analysis showed that chemical method may take the longest time for TPH to degrade half its initial concentration, and biological method is fastest in the soil samples. Also, the first order rate kinetics performed better than Michaelis-Menten equation. The Chemical method (using Nano-particle) slightly outperformed the biological method (using Cow Dung). Finally, the study showed that Nano-particle can be utilized as an alternative bio-stimulant for crude oil remediation. At the end of the analysis, for 30g mass of loamy soil, the percentage reduction of the pollutant was 99.66% when cow dung was used and 99.09% reduction was realized when Magnesium Oxide was used.

KeyWords: Soil, Hydrocarbon, Remediation, Cow Dung, Magnesium Nano-Particle, Kinetic Models.

Introduction

Oil is a major source of energy in Nigeria and the world in general. Oil being the Main stay of the Nigerian economy plays a vital role in shaping the economic and Political destiny of the country. Although Nigeria's oil industry was founded at the beginning of the century, it was not until the end of the Nigeria Civil War (1967 - 1970) that the oil industry began to play a prominent role in the economic life of the country.Nigeria can be categorized as a country that is primarily rural, which depends on primary product exports (especially oil products). Since the attainment of independence in 1960 it has experienced ethnic, regional and religious tensions, magnified by the significant disparities in economic, educational and environmental development in the south and the north. These could be partly attributed to the major discovery of oil in the country which affects and is affected by economic and social components. Crude oil discovery has had certain impacts on the Nigeria economy both positively and adversely. On the negative side, this can be considered with respect to the surrounding communities within which the oil wells are exploited. Some of these communities still suffer environmental degradation, which leads to deprivation of means of livelihood and other economic and social factors. Although large proceeds are obtained from the domestic sales and export of petroleum products, its effect on the growth of the Nigerian economy as regards returns and productivity is still questionable. Petroleum production facilities generate large quantities of oil wastes from drilling, production and processing activities. According to Odiete (1999), "all activities involved in hydrocarbon production and exploration normally have one impact or the other on the environment". The contamination of the environment by hydrocarbon process occurs through exploitation, transport, and leakage of crude oil storage facilities, which are released into the environment, thereby causing environmental pollution (Moro *et al.*, 2015).

Crude oil pollution in the air occurs via evaporation of volatile components or gas flaring, while on aquatic and land environments it can be via spillage from oil facilities. A study reported that land environment was the most affected of pollution from petroleum industry, mostly from leakage of pipelines, storage tanks and other oil facilities (Nnaji, 2017). Crude oil contains toxic compounds and radioactive elements that are serious health concerns, and also affects plant growth (Oyem & Oyem, 2013). Crude oil pollution, especially on soil, has the ability to bind soil particles together because of low water solubility that could reduce soil nutrients (Wang *et al.*, 2019). Most organic and inorganic chemicals are hazardous to soil, and can cause low yield of agricultural produce (Kuppusamy *et al.*, 2016). Various methods are used for the treatment of petroleum hydrocarbon polluted soil, but the choice of method depends on the cost effectiveness, ability to remove contaminants and availability (Yu *et al.*, 2020). The development of a sustainable method for the removal of petroleum contaminants from the environment is essential. In soil contaminated by petroleum hydrocarbons, methods such as biological, physical and chemical technique can be used to remediate a polluted soil (Yuniati, 2018). Bioremediation is a radiation technique that utilises microorganism or bio-stimulants such as enzymes, spent biomass and fertilizers to remove contaminants from contaminated environment through metabolic process (Kumar *et al.*, 2011). Bioremediation is economical, adaptable, efficient and eco-friendly to the environment (Jeon & Madsen, 2013). Bioremediation can be achieved implemented through bio-stimulation, bio-augmentation and phytoremediation, among others. Bio-stimulation refers to the use of fertilizer, plant and animal wastes, or any other agro based material as amendment agents, to stimulate the growth of microorganisms; while in bio-augmentation bacteria or fungi is directly applied to enhance the degradation rate of pollutant (Varjani & Upasani, 2017). In Phytoremediation, a plant is planted in polluted soil to take up the contaminant as it grows.

Most often, bioremediation process takes place in a system best described as reactor, which can be implemented in batch, or continuous reactors such as the fixed bed, fluidized bed and membrane reactors (Pino-Herrera *et al.*, 2017). The use of bioreactor ensures the growth of microbes necessary for the degradation of contaminants are controlled and monitored, thereby providing the necessary conditions to achieve an optimum process performance (Jeon & Madsen, 2013).

This study adopted the bio-stimulation method, implemented in batch reactor for bioremediation of Total Petroleum Hydrocarbon (TPH) contaminant in polluted loamy soils using Nano Particle and Cow Dung. Soil pollution arising from increasing demand for petroleum and its products has become a common problem in recent years. Soils polluted with petroleum hydrocarbons differ from unpolluted soils and are not able to support adequate crop growth and development. Hence, there is need to treat these soils so as satisfy the food requirement of the ever-growing population and also reduce the impact of crude oil in soil environment. Therefore, capability of Nano Particle and Cow Dung in degrading the (TPH & PAHs) in crude oil polluted loamy soils was investigated. The choice for the use of Nano Particle and Cow Dung as bio-stimulant was based on its essential nutrient, implying that it possesses the ability to stimulate hydrocarbon degrading bacteria necessary for effective bioremediation.

Materials and Methods

The experiments were conducted at the chemical Engineering Laboratory in Rivers State University, Port Harcourt Nigeria.

Materials

The materials used include Volumetric flasks: glass 10 and l00 ml, Agilent 6890 Gas chromatographs with a flame lunation Defector, pH meter with glass electrode, Thermometer, Glass beaker (100ml), Glass rod, Laboratory fume hood, Analytical balance, Glass ware, Mineral salt agar, Distilled water, Petri dishes**,** Scientific Calculator, Cow dung, Nano particle, sieve merge.

Preparation Nano particle

40g sodium hydroxide was dissolved in 250ml distilled water (exothermic reaction, co-precipitate method). 26g of Magnesium (ii) chloride was dissolved in 160ml of distilled water endothermic reaction 'wet chemical method" both were mixed together to give a light yellowish substance.

6g of iron chloride ($FeCl_3$) was dissolved in 25ml of distilled water 14g of sodium hydroxide little by little to give a dark colour precipitation. Both the nanoparticle (magnesia oxide and fenton) reagent were dried into power form local grams produced 69.48 (magnesium oxide) +74.86 (magnesium oxide) = 144.34 grams.

Methods

300g of Loamy soil was weighed into 14 rectors. Out of 14 sample reactors, 12 samples containing 300g of loamy soil were polluted with 100ml crude, while the remaining 2 served as control unpolluted soil. The 12 polluted sample were stirred for uniform mixture, 50ml of distill-water were added and stirred then left for 3 days. After 3 days, the samples were taken to the lab to analyze the following parameters; TPH, Bacterial, pH, density, moisture content and particle size. 3 days later the nano-particles of Magnesium Oxide of masses of 5g, 10g, 15g, 20g, 25g and 30g were introduced into 6 separate reactors and also Cow-dung of masses 5g, 10g, 15g, 20g, 25g and 30g were introduced into 6 separate reactors, 2 samples of polluted soil were set as control. Each of the 14 samples were taken to the lab to analysis the TPH, Bacteria and pH for 1^{st} week – 6^{th} week in 45 days.

Physiochemical Properties of the Soil Sample

Physiochemical properties were analyzed; Total Bacterial Count (TBC), Phosphorus content, electrical conductivity, Total Nitrogen Content, Total Organic Carbon

Electrical Conductivity

The electrical conductivity meter was used to measure the electrical conductivity (EC) of the samples. The same procedure stated for pH measurement was used in the determination of EC. However, the EC electrode was thoroughly washed after each reading to avoid cross-contamination and error.

Total Organic Carbon

Total Organic Carbon (TOC) was determined using a method described by Umeda *et al.* (2017). Thus, 1.0g of soil samples was weighed into 250ml beaker, while 10ml of potassium dichromate solution was pipette into beakers and swirled gently to completely wet the soil sample. Thereafter, 20ml of concentrated H_2SO_4 was added using automatic pipette, and gently swirled for one minute to obtain a uniform suspension, as well as for effective and more complete oxidation before allowed to settle for about 30 minutes on asbestos sheet.

On settling, 100ml of distilled water was added followed by addition of 3-4 drops of 0.5 ml diphenylamine indictor. The solution was titrated with 0.5N ferrous sulphate solution until the colour changes from violet to blue and finally bright green colour. The process was repeated on distilled water (blank titration), but without soil to standardize the dichromate. The TOC was calculated according to the formula.

$$TOC = Blank - \frac{Volume\ of\ soil\ Sample \times 0.195}{weight\ \ of\ soil\ sample} \times 100\% \quad (2.1)$$

Total Nitrogen Content

Total nitrogen content was determined using APHA 4500-NO_3B method (APHA, 1998). Thus, 10g of grinded and sieved soil sample containing 10 mg of nitrogen in a dried 500ml Macro-Kjeldahl flask was weighed. It is swirled for about 2 minutes followed by the addition of 20ml of distilled water, and then, allowed to settle for 30 minutes. A tablet of 1g K_2SO_4-H_2O mixture (catalyst), 10g of K_2SO_4 and 30ml concentrated H_2SO_4 were added to prepared sample in the flask and heated cautiously on digestion stand. Upon the notice of water content and frothing, the heating was increased until a clear digest was obtained. The heating was regulated so that H_2SO_4 is about half way up the neck of the flask. After the heating process, the flask was allowed to cool, while 100ml of water was added slowly. The digest was carefully transferred into another clean 750ml Macro-Kjeldahi flask. All soil particles in the original digestion flask were retained due to the severe bumping soil can cause during the Kjeldahl distillation. Soil residue was washed with 50 ml distilled water four times and the aliquant transferred into same flask. Addition of 50ml H_3BO_3 indicator solution into a 500ml Erlenmeyer flask was followed, which was placed under the condenser of distillation apparatus. The 750ml Kjeldahl flask was also attached to the distillation apparatus. About 150ml of 10N NaOH was added into the distillation flask through the opening funnel, and the distillation was stopped after 150ml of the distillate was collected. The NH_4-N in the distillate was determined by titrating with 0.01N standard H_2SO_4 using 25ml burette graduated at 0.1ml intervals. The colour of the end point changed from green to pink. The percentage of nitrogen in the soil was calculated using equation (2.2).

$$N(\%) = \frac{(T - B) \times N \times 1400}{S} \times 100\% \quad (2.2)$$

Where: T = Sample Titration (ml), B = Blank Titration (ml), N = Normality of H_2SO_4 and S = Sample weight (mg).

Phosphorous Content

Phosphorus content was determined according to APHA method $4500 - PO_4^{3-}$ (APHA, 1998). 1.0g of representative soil sample was weighed into clean extraction flask and 10ml of Bray P-1 extracting solution (0.025N HCl and 0.03N NH_4F) was added and vigorously agitated for 1 minute before being filtered. 5ml of the filtrate was pipette into 25ml volumetric flask and diluted to about 20ml of distilled water, and then, by 4ml of ascorbic acid solution (1.056g ascorbic acid in 200ml molybdate-tartarate solution), which were diluted. The diluted solution was allowed to settle for at least 30 minutes. The recording of data was done after a clear colour has been developed.

Procedure for Total Bacterial Count (TBC) Analysis

Microbiological analysis enumeration of heterotrophic bacteria and fungi was carried out by pour plating technique. This was done by inoculating 0-1ml tenfold seriating diluted sample onto nutrients agar (bacterial), acidified streptomycin (1mg/100ml) (fungal) and mineral salt agar (MSA) (hydrocarbon degraders). The mineral salt media contains the following composition in gram per liter of distilled water, N_aC_L 10g, Mg S0$_4$. 7H_2.0, 9.42g, K_{CL} 0.29g, HP0$_4$ 1.2g, KH_2P0_4 0.83g, $NaN0_2$ 0.42g, Agar – Agar 16g, pH 7.2 and 2 nill at petrol/Diesel. The inoculated nutrient agar plates were incubated at 37^0C for 24 hours while the potato dextrose Agar plates were incubated at room temperature counted and expressed as colony firming units per gram (Cfu/ml).

3. Results and Discussion

Table 4.1: Physicochemical Properties of 300g Soil Sample before and after Pollution

Parameters	Loamy Soil			
	Before	**After**	**Using Cow Dung**	**Using Magnesium Oxide**
pH	6.5	4.1	6.3	5.9
EC (µS/cm)	352.43	843.61	432.13	567.34
TOC (%)	2.56	5.38	2.3	3.10
P (%)	1.46	0.93	1.8	1.5
N (%)	23.02	0.07	33.00	26.12
K (%)	32.84	1.42	43.61	30.23s
TBC (cfu/ml)	4.98×10^3	2.16×10^2	13.81×10^5	9.51×10^5

The physicochemical properties of loamy soils before and after being polluted by crude oil are shown in Table 3.1. The change in the physicochemical properties of the after pollution shows that crude oil has significant impact on soil. From the analysis, it was shown that pH, phosphorus (P), nitrogen (N), potassium (K) and Total Bateria Counts(TBC) in the soils were reduced after the crude oil pollution. On the other hand, the electrical conductivity (EC) and total organic carbon (TOC) in the soils increased after the pollution.

Table 3 Total Heterotrophic Bacteria Count. The samples were analysed to ascertain the bacteria growth so as to determine the treatment option that has the most influence on TPH degradation under the Nano-particle Also, identification of hydrocarbon degrading bacteria and TPH analysis was conducted by the 7 days mark. The following were identified and isolated as hydrocarbon degrading bacteria in the analysis of bacteria isolates: Nano-particle (*Campylobacter sp*. and *Listeria monocytogenes sp).* The growth analysis of TBC at the various days is shown in Figures 3.1 to 3.2.

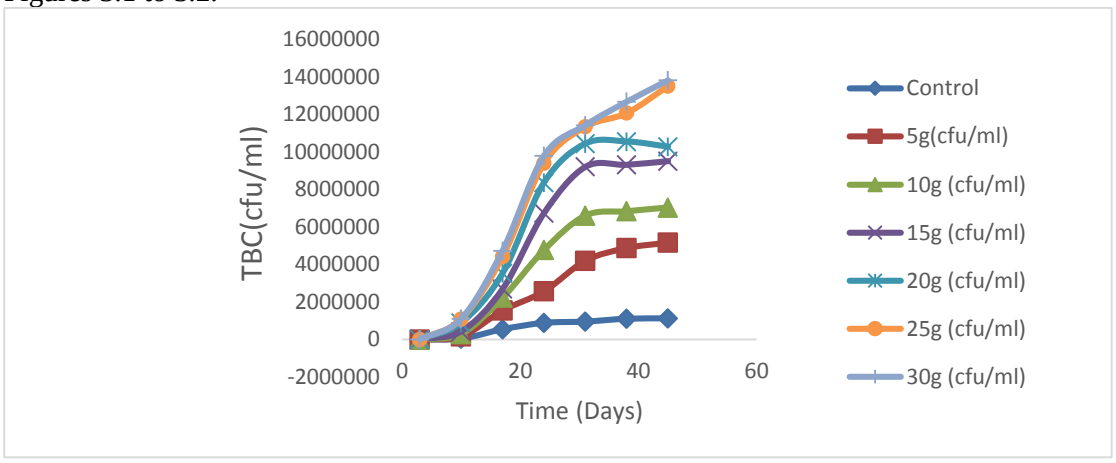


Fig 3:1 TBC Count Variation versus Time in Loamy Soil at Various Weights of Cow Dung Treatment.

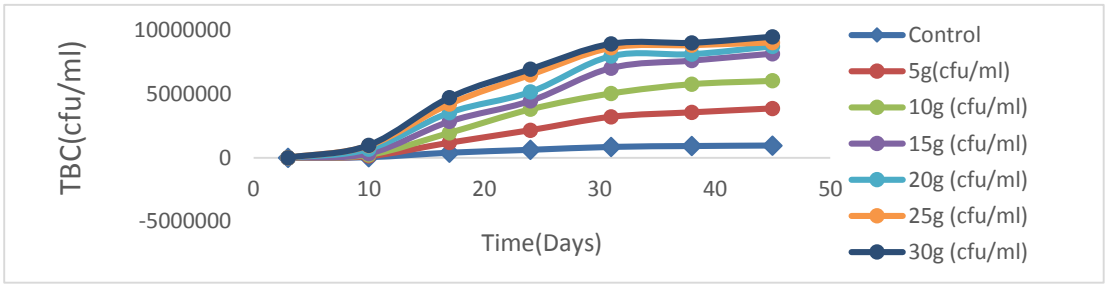
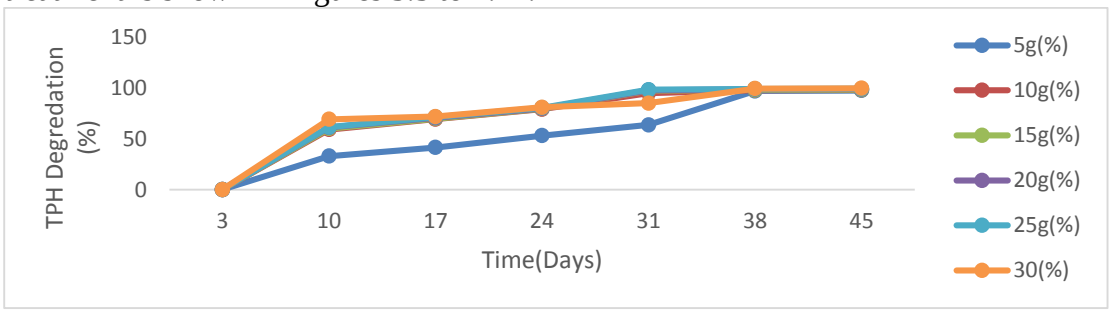


Fig 3:2 TBC Count Variation versus Time in Loamy Soil at Various Weights of Nano-Particle Treatment

TPH Degradation in Soils under the Influence of Treatment.

This study showed that the weight of treatment applied in crude oil contaminated soils has effect on TPH degradation as time of bioremediation increases. Therefore, this section presents the results of the TPH recorded during the investigation periods. Thus, the degradation pattern of TPH in loamy soil under nanoparticle (treatment) compared with control samples as investigated with time at various weights of treatment is shown in Figures 3.3 to 4.12.



TPH Degradation in Loamy Soil versus Time at Various Weights of Nano-Particle Treatment

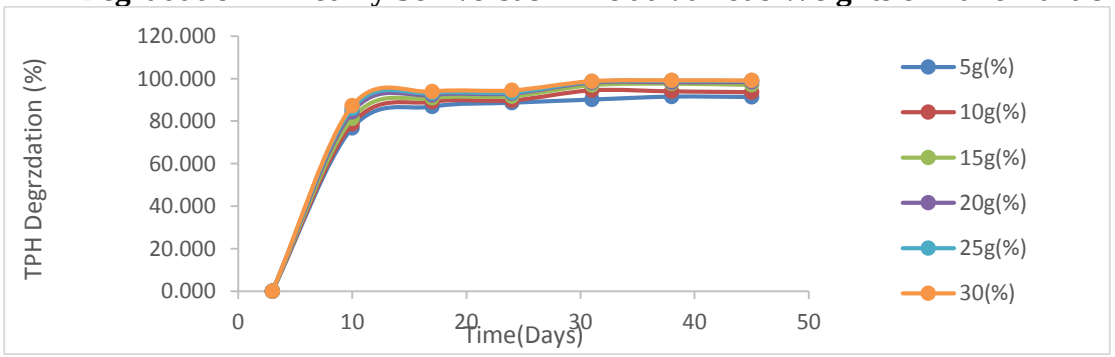


Figure 3.4: TPH Degradation versus Time in Loamy Soil at Various Weights of Cow Dung Treatment

Comparison of Treatment Performance in the Soil.

The performances of nanoparticles and cow dung as bio-stimulant for the degradation of TPH from crude oil polluted loamy soil was compared. Figure 4.13 compared all the treatment options with time at 30g, while Figures 4.14 and 4.15 compared the results obtained at 45th day for the various treatment weights for Cow dung and nanoparticle treatment in the soil samples.

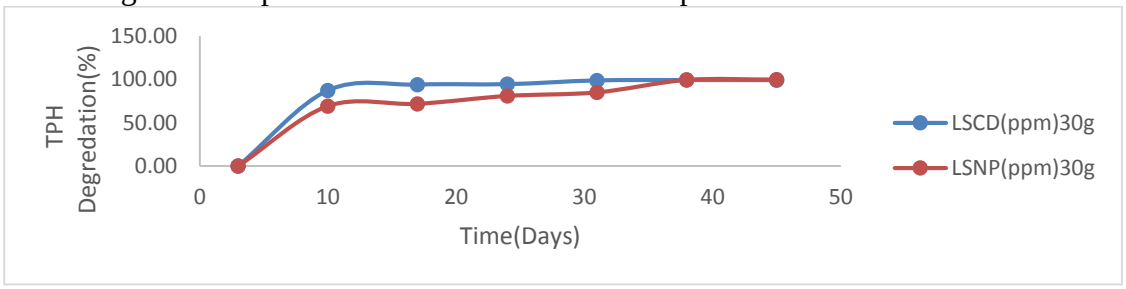


Figure 4.5: Comparison of TPH Removal in the Different Treatment at 30g.

4.5.1 Evaluation of First Order Rate Constant and Half Life

The degradation rate constant in the first order rate kinetic model was determined by comparing Equation (3.16) with the regression equations in Figures 4.19 to 4.24 for the different treatment options. From the determined rate constant, the time taken for the TPH concentration to reduce to half its initial concentration (half-life) was then evaluated.

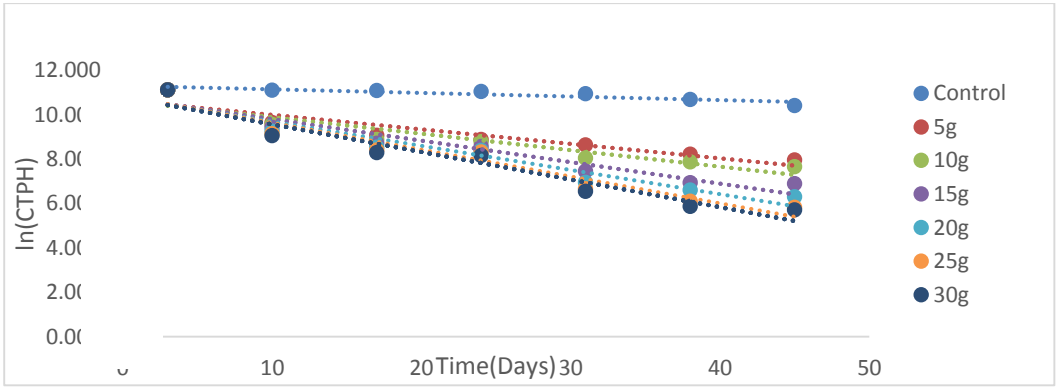


Figure 4.6: Plots for Evaluation of Rate Constant for Cow Dung Treatment in Loamy Soil

Figure 4.6 shows various linear regression equations for the different treatment options in loamy soil under cow dung treatment. From the linear equations on the plot, the degradation rate constant, k_d was determined.

Table 3.2: Rate Constant and Model for Loamy Soil under Cow Dung Treatment

Weight (g)	k (day^{-1})	Predictive Model	$t^{1/2}$ (days)
Control	0.0159	$C_{TPH} = 81570.80e^{-0.0159t}$	43.591
5	0.0651	$C_{TPH} = 42066.21e^{-0.0651t}$	10.646
10	0.0744	$C_{TPH} = 41772.77e^{-0.0744t}$	9.316
15	0.0967	$C_{TPH} = 47667.26e^{-0.0967t}$	7.167
20	0.1091	$C_{TPH} = 48533.04e^{-0.1091t}$	6.352
25	0.121	$C_{TPH} = 51688.99e^{-0.121t}$	5.728
30	0.1243	$C_{TPH} = 49563.01e^{-0.1243t}$	5.576

Table 3.3: Rate Constant and Model for Loamy Soil under Nano-Particle Treatment

Weight (g)	k (day^{-1})	Predictive Model	$t^{1/2}$ (days)
Control	0.0161	$C_{TPH} = 81470.80e^{-0.0161t}$	43.049
5	0.1089	$C_{TPH} = 180412.29e^{-0.1089t}$	6.364
10	0.1197	$C_{TPH} = 121905.32e^{-0.1197t}$	5.790
15	0.1248	$C_{TPH} = 122271.59e^{-0.1248t}$	5.553
20	0.1351	$C_{TPH} = 134995.94e^{-0.1351t}$	5.130
25	0.1446	$C_{TPH} = 147856.87e^{-0.1446t}$	4.793
30	0.1601	$C_{TPH} = 170586.88e^{-0.1601t}$	4.329

Table 3.3. and 3.4 shows the first order kinetic rate model, after inserting the degradation rate constant for the respective treatment option, is also shown in Table the evaluated degradation rate constant, the estimated time at which the TPH concentration would degrade to half its initial concentration is given in Table 4.2 for the respective treatment option. Based on the evaluated time, it will take about 43 days for the TPH concentration to reduce to half its initial concentration if it were allowed to degrade naturally (control sample) in loamy soil, but the addition of 5g cow dung treatment caused the time to reduce to about 6 days to attain 50% degradation, which even reduced further to just about 21 days when the treatment weight increased to 30g. This implied that increase in treatment weight reduces the time at which the TPH degraded to half of its initial concentration. This observation agreed with the work of Ofeogbu *et al*. (2015), for soil amended by cow dung and NPK fertilizer. This reduction in half life was attributed to increase in degradation rate. This is evident in the value of the degradation rate constant, which was lowest in the control sample, but increased as the treatment weight was increased.

Conclusion

The performance of Cow dung and Nano-particle treatment has been investigated for bioremediation of crude oil polluted loamy soil.

The characterized loamy soil before and after pollution showed that the physicochemical properties of the soils changed immediately after polluted by crude oil, and this implied that crude oil has significant impact on soils. Thus pH, phosphorus, nitrogen, potassium and Total Bateria Counts in the soils were reduced after pollution, while electrical conductivity and total organic carbon were increased.

Analysis of bacteria growth for the various treatment options shows that Total Bacteria Count (TBC) in the soil amended with different weights of *cow dung* (treatment) reduced immediately after pollution, but the TBC population increased significantly as time and treatment weight increased. The microbial growth rate attained a stationary state at some point in time, but the bacteria growth in control samples was very slow, indicating that *cow dung* stimulated the growth rate of bacteria in the soil.

The percentage degradation of Total Petroleum Hydrocarbon (TPH) in loamy soils under cow dung and nanoparticle treatment increased with time and treatment weight. There was rapid increase in percentage degradation in the treated soil compared to the gradual increase recorded in the control samples for the various soils. The highest TPH degradation was recorded on the 45^{th} day across the various treatment weights, implying that duration of remediation influenced the degradation efficiency. Also, Treatment improved the degradation rate of TPH in the soils.

Comparison of the different treatment options in remediation of the polluted soils showed that the all the treatment options performed better on the 45^{th} day, but the 30g weight samples performed much better than the other treatment options across the soil types. TPH percentage degradation across the treatment options was highest in Nano-particle treatment soil compared with Cow dung treatment soil. At the end of the analysis, for 30g Nano-particle treatment, the TPH degradation percentages were recorded as 97.48% to 99.66% for Nano-particle, while 91.35% to 99.10% were recorded for Cow dung treatment, respectively. Similarly, the Nano-particle samples slightly edged the Cow dung samples soils, indicating that treatment for bioremediation Nano-particle perform better than Cow dung.

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