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Performance Evaluation of Groundnut Shell Treated Lead Containing Wastewater and Pb-Loaded Adsorbent on the Growth Performance of Beans

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ABSTRACT

Lead (Pb) is the second most toxic metal after As but industrial effluents with Pb are used to irrigate the agricultural lands without treatment, contaminating the soil and affecting plants growth. This study aims at treating water-containing Pb with groundnut shell, studying the influence of the treated water, untreated Pb wastewater and the lead adsorbed groundnut shell adsorbent on beans growth performance. Pb(II) adsorption was studied using the parameters such as contact time, dose of adsorbent, Pb(II) initial concentration, pH and temperature. Untreated Pb(II) wastewater (100, 50, 10, and 1 mg/L), groundnut shell treated Pb(II) wastewater for five concentrations, and the recovered Pb(II) adsorbent were respectively used to irrigate and fertilize the soil and beans growth performance monitored for 50 days. 90% lead was removed in 5 minutes. 100% removal efficiency obtained for 1 mg/L, reducing to 80% for 5 mg/L and increasingly steadily to 97% for 300 mg/L. Maximum of 94% Pb(II) was removed at 20, 35 and 45 °C and the removal process was spontaneous at all the temperatures studied and exothermic. R_L (dimensionless separation factor) of 0.91 shows very favourable Pb(II) removal. Pb(II) adsorption capacity of 117.30 mg/g was obtained, and higher than those of other studies reported for groundnut shell. The adsorption energy from Dubinin–Radushkevich isotherm was 0.669 kJ/mol, – 0.239 kJ/mol from Temkin isotherm and -6.37 KJ/mol enthalpy, indicating physical adsorption process. All beans growth parameters were favourable with treated wastewater for all the concentrations tested compared to the control and raw untreated wastewater. Highest beans growth performance was obtained with used groundnut shell adsorbent. Groundnut shell effectively removes lead from wastewater, producing safer water and lead adsorbed groundnut shell waste adsorbent that both enhance beans growth performance more than the untreated Pb(II) wastewater and the control.

1. Introduction

Water pollution by heavy metals is on the rise worldwide due to steady growth in the industrial sector, improper waste disposal and other anthropogenic activities such as agricultural practices, needed to meet the needs of the growing world population [1]. These activities result in the generation of significant number of wastes in solid and effluent forms, with potential effects on plants and humans. The soil and water sources are generally the recipient of most of these wastes and effluents. These solid or effluent generally contain heavy metals, which are non-biodegradable, bio-accumulate and are toxic [2]. Heavy metals are a group of pollutants, which has gained increasing attention as an environmental pollutant [3]. Pb possess some specific properties such as high malleability, softness, ductility, low melting point and resistance to corrosion [4], resulting in the application of Pb in different domains including automobiles, paint, ceramics, plastics, gasoline, explosives, power batteries, polymers, cosmetic, food cans, radiators, ink, Household products (such as curtain lead weights, coins, costume, jewellery, and stained glass windows), burning of fossil fuels, steel industry, atmospheric deposition, Pb contamination also comes from weathering processes, mining and smelting activities [2,4-6]. Industrial effluents with heavy metals like Pb are used to irrigate the agricultural lands, which in turn contaminate the soil [7]. Pb is the second most toxic metal after As as it has no beneficial role in biological systems and is hazardous for plants, animals and human beings [8].

When the lead level in the body exceeds 5 μg/dL for children and 10 μg/dL for adult, lead poisoning occurs [4]. Threshold level of Pb for plants is around 2 mg kg^{-1}, and 50–300 mg kg^{-1} for agricultural soils [6]. Above these critical limits, it obstructs the germination process and early crop growth causing adverse effects on morphological, physiological and biochemical processes [9]. Pb contamination in soil can lead to impairment in nutrient uptake, alterations in plant water relations and generation of reactive oxygen species (ROS) [4]. This result in reduced photosynthesis, causing cell death with significant reduction in crop yield. Its toxicity retards the emergence of radicle via increased carbohydrates and protein contents by affecting the polyphenol oxidases and peroxidase activity, and resultantly affects oxidizing ability of roots [10]. Lead exposure inhibits the activity of several key enzymes in plants. High concentration of Pb competes with other cations and inhibits the uptake of divalent such as manganese (Mn^{2+}), iron (Fe^{2+}), magnesium (Mg^{2+}) and calcium (Ca^{2+}) [6]. Due to these toxic effects of lead on plants, it is therefore necessary to remove it from wastewater before use for irrigation.

Several methods have been used to remove lead from wastewater such as ultrafiltration, chemical precipitation, ion exchange, electrodialysis, reverse osmosis and flotation [1]. Unfortunately, these methods are expensive for developing countries and even when it exists, lacks people with skills to operate and maintain them. Adsorption is the most promising because it is easy to operate, cheap, can function without electricity and can remove low and high levels of pollutants [11]. It is based on an adsorbent such as biomass, clay, biochar etc. that are generally very available locally as waste or natural material. Many studies have removed Pb from wastewater using groundnut shell due to its abundance [12-18], but studies involving the use of Pb containing wastewater treated with groundnut shell and used for beans germination are scarce in literature. Equally, studies involving the valorization of lead adsorbed groundnut shell adsorbent for beans germination are also scarce in literature. This study is therefore aimed at treating water-containing Pb with groundnut shell, then studying the influence of the treated water, untreated Pb wastewater and the lead adsorbed groundnut shell adsorbent on beans growth performance.

Groundnut shells account for approximately 20% of the dried peanut pod by weight, meaning there is a significant amount of shell residual left

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after groundnut processing [19]. About 65% of of groundnut is produced in Asia, against 26% for Africa. In Cameroon, an annually 414.046 kg/ha groundnut yield has been reported from 2012-2022 [20]. Groundnut shell is carbon rich with significant porosity and many surfaces functional groups such as COOH, OH, NH, C=O, C-O [21], which can be used to bind Pb. The first part of this work involves the influence of different parameters (contact time, mass of adsorbent, Initial concentration of Pb, temperature and pH) on the removal of Pb from wastewater, followed by growing beans on soil irrigated with lead treated wastewater and lastly growing beans on soil fertilized with recycled groundnut shell adsorbent loaded with lead from the wastewater treatment process.

2. Experimental Methods

2.1 Preparation and Characterization of Groundnut Shell Adsorbent

The groundnut shells were collected from a local groundnut-grinding machine. They were thoroughly washed with distilled water to remove dirt particles such as such as sand, soil and till the water coming out was colourless. Then sun dried for 15 days to remove excess moisture and ease crushing. The dried groundnut shells were then crushed into powder, sieved through <2 mm sieve and used for characterization and adsorption studies. Scanning electron microscopy (SEM) coupled with energy dispersive x-ray spectroscopy (EDX) (Thermo Scientific Axia ChemiSEM) were used to determine respectively, the morphology and chemical composition of the groundnut shell.

2.2 Preparation Pb^{2+} Solution

A 1000 mg/L Pb^{2+} stock solution was prepared by dissolving 1.57 g of $(CH_3COO)_2Pb$ (AR grade) in 1 L of distilled water. Working solutions of 1, 10, 50, 100 and 300 mg/L were then prepared by dilution of the stock solution.

2.3 Batch Pb^{2+} Removal Experiments

Prior to removal trials, the calibration curve was obtained by complexing 20 mL of Pb^{2+} concentrations of 0, 1, 5, 10, 100, 200 mg/L, each with 1 mL of 0.05 M KI solution for colour development [22] and absorbance measured on a UV-visible spectrophotometer (PRIM SECOMAM) at a wavelength of 466 nm (pre-determined). The calibration curve was obtained as y = 0.0037x - 0.0029, R^2 = 0.9988. The removal experiments were done by studying the influence of different parameters on lead removal process using 100 mL of synthetic wastewater in 250 mL beakers. The experiments were all carried out without stirring in order to ascertain the efficiency of the process in the absence of electricity, a situation very common in developing countries. The parameters studied were; contact time (5-60 minutes), Pb^{2+} initial concentration (1, 10, 50, 100, and 300 mg/L), mass of groundnut shell (0.05-0.5 g), temperature (20-60°C), and pH (2, 4, 7, 8). All pH modifications were done using 0.1 mol/L HCl and 0.1 mol/L NaOH. After the required time interval, 20 mL of the solution was filtered in to a 250 mL beaker using a Whatman Filter No. 1 filter paper and 1 mL of potassium iodide solution added and shaken manually for proper mixing and colour development. 5 mL of this coloured solution was collected using a syringe in to a quartz cuvette and residual lead concentration determined using UV- visible spectrophotometer. The adsorbed quantities and the percentage of removal were calculated using Eqs.(1) and (2) respectively.

$$\text{Amount removed (qe)} = \frac{(\text{Ci}-\text{Cf})\text{V}}{\text{m}} \tag{1}$$

$$\text{\% removed (Re)} = \frac{(\text{Ci}-\text{Cf})}{\text{Ci}} \times 100 \tag{2}$$

where, the initial and residual concentrations are respectively represented by C_i and C_f (mg/L), V is solution volume in litres (L); m is the amount of adsorbent used in grams; q_e is amount of lead removed (mg/g).

2.4 Equilibrium and Kinetics of Lead on Groundnut Shell Adsorbent

Equilibrium studies will give the distribution of Pb^{2+} molecules between the groundnut shell and wastewater, groundnut shell removal capacity, as well as the type of Pb^{2+}- groundnut shell interactions. Meanwhile kinetic studies provide information on the Pb^{2+} removal rate. The equilibrium isotherm models and the kinetic models tested in this study are given in Tables 1 and 2 respectively.

Table 1 Linear isotherm adsorption models tested [23]

Isotherm	Nonlinear form	Linear form	Plot	Characteristic parameters
Freundlich	$q_e = K_F C^{1/n}$	$\ln q_e = \ln K_F + 1/n 1nC_e$	$\ln q_e$ vs $1nC_e$	K_F = adsorption capacity (mg/g)(L/mg)$^{1/n}$ $1/n$ = adsorption intensity
Langmuir		$\frac{C_e}{q_e} = \frac{1}{bq_m} + \frac{C_e}{q_m}$	$\frac{C_e}{q_e}$ vs C_e	q_m =adsorption capacity(mg/g) b = Langmuir equilibrium constant (L/mg)
Temkin	$q_e = B\ln A_T.C_e$	$q_e = B\ln A_T + B\ln C_e$	q_e vs $\ln C_e$	A_T: equilibrium binding constant (L/mg) $b_T = \frac{RT}{B}$ = adsorption energy (kJ/mol)
Elovich	$\frac{q_e}{q_m} = K_E C_e \exp(-\frac{q_e}{q_m})$	$ln \frac{q_e}{C_e} = \ln K_E q_m - \frac{q_e}{q_m}$	$ln \frac{q_e}{Ce}$ vs q_e	q_m = maximum adsorbate uptake K_E = Elovich equilibrium constant (L/mg)
Dubinin-Radushkevich *(D-R)*	$q_e = X_m exp(-K\varepsilon^2)$	$lnq_e = lnX_m - K\varepsilon^2$	lnq_e vs ε^2	X_m = adsorption capacity (mg/g) K = constant related to the adsorption energy (mol^2 kJ^{-2})
Harkin-Jura		$\frac{1}{qe2} = \frac{B}{A} - (\frac{1}{A})\ln C_e$	$\frac{1}{qe2}$ vs $\ln C_e$	*B* and *A* are Harkin-Jura constants
Halsey	$q_e = (\frac{KH}{Ce})^{1/nH}$	$\ln q_e = \frac{1}{nH}\ln K_H - 1/n_H \ln C_e$	$\ln q_e$ vs $\ln C_e$	K_H and $n_H$are Halsey isotherm constant

Table 2 Linear kinetics adsorption models tested [23]

Model and parameters	Characteristic equations	Linear forms	Straight line equation
Pseudo-First order K_1; qe	$\frac{dq(t)}{dt} = k_1(q_{eq} - q_t)$	$\ln(q_e - q_t) = \ln q_e - k_1 t$	$\ln(q_e - q_t) = f(t)$
Pseudo-second order; K_2, qe	$\frac{dq(t)}{dt} = k_2(q_{eq} - q_t)^2$	$\frac{t}{q_t} = \frac{1}{k_2 {q_e}^2} + \frac{t}{q_e}$	$\frac{t}{q_t} = (\frac{t}{q_e})$
Intra-particular diffusion; K_{id}	$q_t = k_{id} t^{1/2}$	$q_t = k_{id} t^{1/2} + C$	$q_t = f(t^{1/2})$

K_1 is the pseudo-first-order rate constant (min^{-1}), k_2= second-order constant (g mg^{-1} min^{-1}); k_{id} (mg g^{-1} $min^{1/2}$) = rate constant of intra-particle diffusion; (mg g^{-1} min)

2.5 Thermodynamics Parameters of Pb^{2+} Removal on Groundnut Shell

Thermodynamic parameters such enthalpy ΔH°, entropy ΔS°, and Gibbs free energy ΔG° are used to evaluate if the removal process is respectively spontaneous, exothermic or endothermic in behaviour. These parameters were calculated using Eqs.(3) and (4) [23]:

$$\Delta G = -RT\ln K \tag{3}$$

$$\ln K = \left(\frac{\Delta S}{R}\right) - \left(\frac{\Delta H}{RT}\right) \tag{4}$$

where *R* is the ideal gas constant (kJ mol^{-1} K^{-1}) and *T* is the temperature (K). The enthalpy change (ΔH) and the entropy change (ΔS) were calculated from a plot of lnK versus 1/T. K was calculated from Eq.(5),

$$K = C_{ad}/C_f \tag{5}$$

$$C_{ad} = C_i - C_f$$

where K is the equilibrium constant, C_f is the residual concentration at temperature T, C_i is the initial concentration, C_{ad} is the concentration of pollutant in the adsorbent at equilibrium at temperature T.

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2.6 Toxicity Assessment of Treated Wastewater and Use Adsorbent on Beans Growth Performance

Lead containing wastewater was treated with groundnut shell adsorbent and the treated wastewater, untreated wastewater and the recovered adsorbent used respectively to irrigate (treated & untreated wastewater) and fertilize (recovered adsorbent) the soil and beans growth performance on the soil monitored. Accordingly, 5 g of groundnut shell adsorbent was added to 50 L of different Pb^{2+} working solutions of 100 mg/L, 50 mg/L, 10 mg/L and 1 mg/L concentration each and allowed to stand for 10 minutes, then followed by filtration. The filtrate from each concentration was used to contaminate the soil for three days prior to planting. Meanwhile, the adsorbent recovered from filtration of each of the concentrations were mixed together, dried for 10 days and added to the soils where beans was planted on the same day. Ten treatments were used; raw 100 mg/L Pb^{2+} wastewater and treated 100 mg/L Pb^{2+} wastewater, raw 50 mg/L Pb^{2+} wastewater and treated 50 mg/L Pb^{2+} wastewater, raw 10 mg/L Pb^{2+} wastewater and treated 10 mg/L Pb^{2+} wastewater, raw 1 mg/L Pb^{2+} wastewater and treated 1 mg/L Pb^{2+} wastewater, Pb^{2+} adsorbed groundnut shell adsorbent and the control which was soil without lead containing wastewater or recycled adsorbent or any other fertilizer. 3 seeds were planted per treatment, germination percentage, number of flowers and pods development after a period of 50 days monitored. The germination percentage was calculated using Eq.(6).

$$\text{Germination percentage} = \frac{\text{Number of germinated seeds}}{\text{Number of seeds planted}}\, x\, 100 \qquad (6)$$

Prior to planting, a germination test was performed with the beans used to verify the quality of the seeds. 50 beans seeds were planted and the number germinated was used to evaluate the germination percentage (Eq.(16)). 48 seeds germinated giving a percentage of 96, implying the seed were fit for the experiment.

3. Results and Discussion

3.1 SEM-EDX Features of Groundnut Shell

Results of SEM analysis of groundnut shell are presented in Figs. 1(a), (b), (c), and (d) for particle size of 10 µm, 20 µm, 50 µm and 100 µm respectively. While Figs. 1(a) and (b) with particle sizes of 10 µm and 20 µm show a more block and smooth structure with well-structured pores, Figs. 1(c) and (d) with particle sizes of 50 µm and 100 µm present more irregular fiber-like sheets and multi-hole porous structures. Other authors have reported similar SEM structures for raw groundnut shell [24-27].

Fig. 2 and Table 3 illustrate the chemical composition of the groundnut shell. These results indicate the rich carbon content of groundnut shell with 73.08%. The authors Yallappa et al. [24] also concluded that carbon is the major element in raw groundnut shell with a content of 81.84%. Carbon rich adsorbents have unique properties such as high surface area, tunable porosity, and functional groups, which enhance their adsorption capabilities [28].

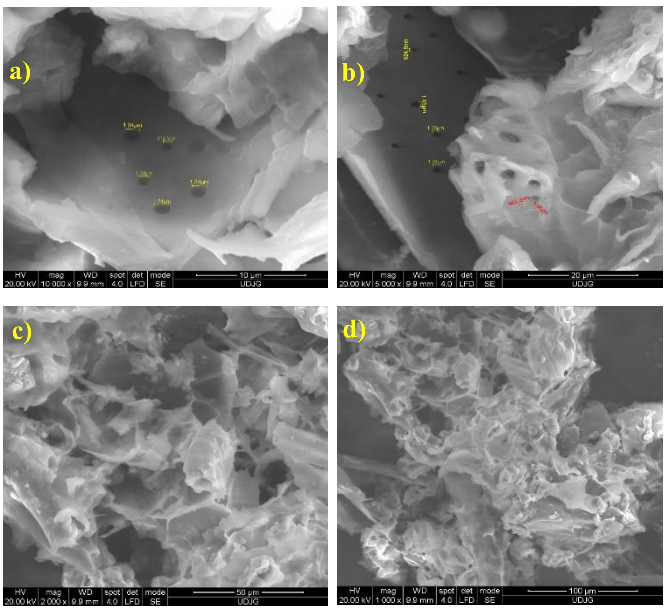

Fig. 1 SEM images of groundnut shell a) 10 µm, b) 20 µm, c) 50 µm and d) 100 µm

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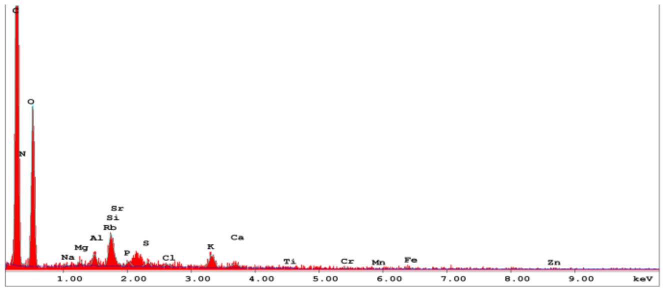

Fig. 2 EDX chemical composition of groundnut shell

Table 3 Major element in groundnut shell

Element	Weight %	Atomic weight %
C	64.74	73.08
O	27.60	23.39
N	1.34	1.29
Si	1.78	0.86

3.2 Contact Time Impact

The effect of contact time on Pb(II) adsorption was studied from 0 to 60 minutes without stirring, with 0.1 g groundnut shell and the results are presented in Fig. 3a for percentage removal and Fig. 3b for residual lead concentration. From Fig. 3a, about 90% of lead was removed in the 5 minutes, which stayed constant up to 20 minutes, then followed by a small decrease to 89% in 40 minutes and 87% in 60 minutes. Though the process was not stirred, the high removal in 5 minutes suggest there is a strong affinity of lead for groundnut shell surface, which became saturated. However, the fact that there was a decrease in removal efficiency from 40 minutes confirming the reduction in the number of vacant sites available for adsorption [29,30]. Results of this study are slightly better than other studies. For example, studies of Bai et al. [25] used groundnut shell heated at 1000 °C to adsorb Pb(II) from wastewater, and obtained maximum removal efficiency of 96.5% in 25 min. Authors Qaiser et al. [12] also reported maximum of 75% of Pb(II) adsorption in 15 min using raw groundnut shell while the studies of OuYung et al. [13] adsorbed Pb(II) from wastewater using raw groundnut shell and reported more than 90% in 30 min.

Despite the high removal efficiency obtained in this study, about 8 mg/L in 5 minutes to about 12 mg/L of Pb(II) in 60 minutes still remained in the treated water (Fig. 3b). This can be resolved by adding further steps to eliminate the pollutant depending on the end use. If it is for consumption, it should be noted that when the lead level in the body exceeds 5 µg/dl for children and 10µg/dl for adult, lead poisoning occurs [4]. Meanwhile the threshold level of Pb for plants is around 2 $mgkg^{-1}$, and 50–300 $mgkg^{-1}$ for agricultural soils [6]. Author Elkhatib et al. [31] studied the impact of lead accumulation on seed germination, plant growth and yield of radish plants (*Raphanus sativus* L.) treated with 0, 100, 200 and 300 ppm of Pb. He concluded that higher concentration of lead (300 ppm) caused a significant decrease in seed germination, plant height, fresh leaves and dry weights, leaves area and yield. Authors Aicha et al. [1], also reported that the effects of Pb on seed of *Cleome amblyocarpa* Barr. & Murb germination show alteration depending on concentration, duration of exposure and seedling organs. This means the low residual concentrations obtained in this study are not likely harmful to plants.

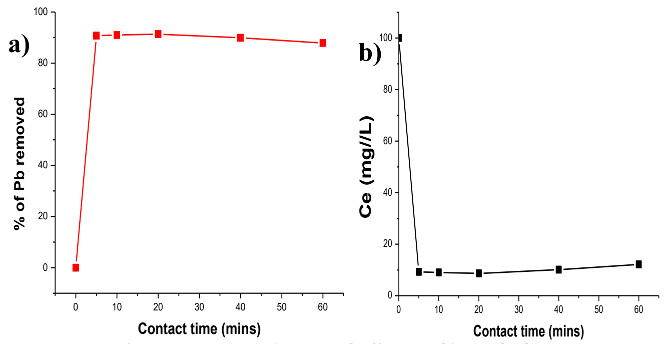

Fig. 3 Impact of contact time on a) removal efficiency b) residual concentration of Pb(II) adsorption colour and using groundnut shell

Results of the effect of contacted time were fitted to three kinetic models, and the calculated kinetic parameters are summarized on Table 3 where the following observations can be made. Firstly, the pseudo-first-order model has R^2 > 99% but there is a strong disagreement between calculated (qe, c (mg/g) and experimental (qe, ex (mg/g)) amounts adsorbed, hence this mode does not describe Pb(II) removal on groundnut shell adsorbent. This is also valid for Intraparticle diffusion model with R^2 < 99%. However, pseudo-second-order shows a good fit with R^2 of 0.9997 and an agreement between calculated (qe, c (mg/g) and experimental (qe, ex (mg/g)) amounts adsorbed, suggesting Pb(II) removal occurs through a two-step process; migration of adsorbate to adsorbent surface followed by attachment [32]. Other studies adsorbing Pb(II) with groundnut shell have also reported similar findings with Pseudo-second-order fitting the removal process [12,13].

Table 3 Kinetic parameters for the removal of Pb(II) from wastewater using groundnut shell

Models	Parameters	Value
Pseudo-first-order	k_1(min)	0.0441
	qe, c (mg/g)	0.2079
	*q*e, ex (mg/g)	91.02
	R^2	0.9933
Pseudo-second-order	k_2 (g/mg/min)	0.024
	qe, c (mg/g)	87.72
	qe, ex (mg/g)	91.02
	R^2	0.9997
Intraparticle diffusion (Weber–Morris)	k_{id} (mg/g/min$^{1/2}$)	0.5112
	C (mg/g)	92.637
	R^2	0.6755

qe, c and qe, ex represent calculated and experimental equilibrium amounts adsorbed respectively.

3.3 Quantity of Groundnut Shell Impact

Increase in adsorbent dose led to a decrease in efficiency of Pb(II) removal process from about 94% with 0.05 g adsorbent to 80% with 0.5 g (Fig. 4a) and an increase in residual concentration to about 20 mg/L with 0.5 g adsorbent (Fig. 4b). These results are contrary to those of Qaiser et al. [12], where Pb(II) removal increased from about 10% at 1g/L dose of groundnut shell adsorbent to about 85% at 50 g/L dosage. However, these results are similar to those of Maki and Qasim [18] who activated groundnut shell with KOH at 750 °C, and used it to adsorb Pb(II) from wastewater. Removal efficiency increased to about 98% with 2 g adsorbent, and then dropped to less than 95% with 4 g adsorbent. According to them, the decrease of the removal efficiency with higher dose higher can be attributed to the increasing of the covered active sites by metal ions, which subsequently cause a loading in adsorbent capacity. The decrease in this study may de resulting from the fact that the process was not stirred, as increase in mass of adsorbent reduced the surface area of adsorbent available for adsorption with no stirring.

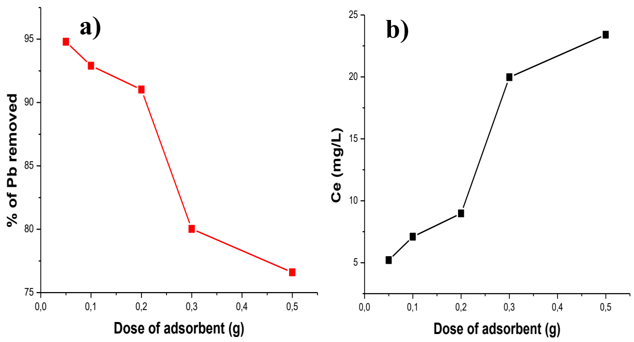

Fig. 4 Impact of groundnut shell quantity on a) removal efficiency b) residual concentration of Pb(II) adsorption colour and using groundnut shell

3.4 Pb(II) Initial Concentration Impact

Results of the effect of Pb(II) concentration on removal efficiency by groundnut shell is shown in Fig. 5a and Fig. 5b for residual concentration. From Fig. 5a, removal efficiency of 100% is obtained for 1 mg/L initial concentration, then reducing to about 80% for 5 mg/L concentration and increasingly steadily to about 97% for 300 mg/L concentration. Correspondingly, the residual concentration (Fig. 5b) is very low with about 6 mg/L for 300 mg/L. Thus, removal efficiency increases with increasing concentration. This is due to the high driving force, which causes huge quantity of the molecules to penetrate through the compacted shell of the groundnut shell, as this compacted shell prevents the spread of molecules in solution [32]. These results are contrary to those of Maki and Qasim [18] who activated groundnut shell with KOH at 750 °C. They reported maximum efficiency of 97% at Pb(II) concentration of about 30 mg/L, and the efficiency reduced to about 90% at 100 mg/L.

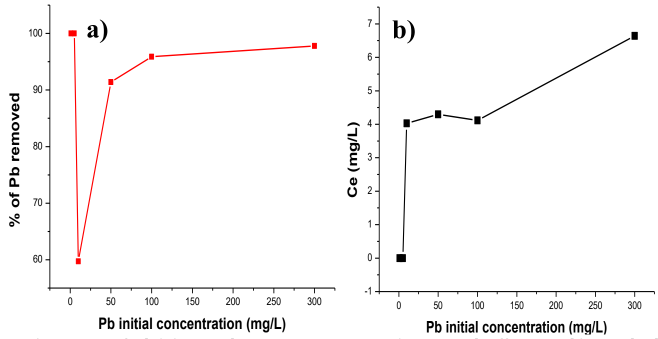

Fig. 5 Impact of Pb(II) initial concentration on a) removal efficiency b) residual concentration of Pb(II) adsorption colour and using groundnut shell

Langmuir, Freundlich, Dubinin- Radushkevich, Elovich, Temkin, Harkin-Jura and Halsey isotherm models were used to evaluate the equilibrium adsorption parameters of Pb(II) by groundnut shell and the results of the calculated parameters are presented on Table 4. It can be observed that all the presented models have R^2 >95%, with Langmuir model having the highest value of 0.9999, indicating it best described the process of Pb(II) removal by groundnut shell adsorbent. The fitness of this model shows that adsorption occurred on a monolayer surface dominated with homogenous sites [33]. The dimensionless separation factor, R_L, a good indicator of the adsorption is defined by Eq.(7),

$$R_L = \frac{1}{1+bC_i} \quad (7)$$

where, b is the Langmuir constant and C_i the initial Pb(II) concentration. For favorable adsorption, $0 < R_L < 1$; while $R_L > 1$, $R_L = 1$ and $R_L = 0$, respectively, describe unfavorable, linear and irreversible adsorption [33]. R_L of 0.91 was obtained, proving that Pb(II) removal from wastewater by groundnut shell is very favourable. This is further confirmed by high Langmuir equilibrium constant (b) value of 0.933 L/g. Other authors also obtained the best fit of Langmuir isotherm for removal of Pb(II) using groundnut shell. Accordingly, authors Maki and Qasim [18] obtained Langmuir equilibrium constant (b) value of 0.085 L/g, 1.48 L/g by OuYang [13], and 0.012 L/g by Qaiser et al. [12]. These studies also obtained respectively, maximum Pb(II) adsorption capacities of 87.11, 7.13, and 30.21 mg/g compared to 117.30 mg/g in this study (Table 4).

The Dubinin–Radushkevich isotherm model helps in determination of the apparent adsorption energy, *E* (kJ mol^{-1}) and is given by Eq.(8),

$$E = 1/\sqrt{2K} \quad (8)$$

K is a constant related to the adsorption energy (mol^2 k/J^2)

The variation of the energy between 8 and 16 kJ/mol, implies ion exchange controls the removal process, less than 8 kJ/mol, implies physical sorption mechanism, and above 16 kJ/mol, shows chemisorption may be the main removal mechanism [33]. From Table 4, the calculated energy is 0.669 kJ/mol, indicating physical adsorption.

The Elovich isotherm model assumes that the adsorption sites increase exponentially with adsorption, implying multilayer adsorption [34]. Though this model has a well-fitted R^2 value of 0.9984 (Table 4), the calculated adsorption capacity of 9.29 mg/g is so low. This is likely due to migration of Pb(II) molecules from one layer to another since Elovich isotherm model assumes multilayer adsorption.

Temkin isotherm model [35] is based on the assumption that the heat of adsorption of the adsorbates in the layer decreases linearly rather than logarithmically with coverage due to adsorbent-adsorbate interactions and that adsorption is characterized by a uniform distribution of the binding energies, up to some maximum binding energy[36]. Temkin isotherm describes Pb(II) adsorption on groundnut shell with R^2 value 0.998 (Table 4). The b_T (adsorption energy (kJ/mol)) value of – 0.239 was obtained, indicating Pb(II) is adsorbed on groundnut shell by physical mechanism [23]. These results are similar to those of Osobamiro et al. [21] who reported a good fit of Temkin isotherm model (R^2 = 0.9661) when they adsorbed lead (II) from aqueous solution using groundnut shell.

Harkin–Jura isotherm model assumes multilayer adsorption on absorbents surface with the surface having heterogeneous pore distribution [36]. Results in Table 4 shows that this model fit Pb(II) adsorption on groundnut shell with with R^2 value of 0.9965, hence indicating the heterogeneous pore distribution on the groundnut surface and the possibility of multilayer adsorption of Pb(II) on its surface.

The Halsey isotherm model shows a good fit of Pb(II) adsorption on groundnut shell with R^2 of 0.9975 (Table 4). Because this model like Harkin–Jura is suitable for multilayer adsorption as well as adsorption on a heterogeneous surface [23], fitness of this model indicate that the surface

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of groundnut shell is heterogeneous in nature, hence there is possibility of multilayer adsorption of Pb(II) on groundnut shell surface.

The results of Elovich and Harkin–Jura isotherms obtained in this study are contrary to Osobamiro et at [21] who reported R^2 values 0.6630 for Elovich model and 0.6901 for Harkin–Jura, hence these models don't fit adsorption of lead (II) from aqueous solution using groundnut shell.

Table 4 Isotherm parameters for Pb(II) removal using groundnut shell

Models	Parameters	Value
Langmuir	q_m (mg/g)	80
	b (L/g)	0.933
	R_L	0.91
	R^2	0.9999
Freundlich	K_F (mg/g)(L/g)$^{1/n}$	117.30
	n_F	-8.66
	R^2	0.9975
Dubinin- Radushkevich (D-R)	q_m (mg/g)	84.32
	K_{D-R} (mol^2/KJ^2)	-1.1173
	E (kJ/mol)	0.669
	R^2	0.9814
Elovich	q_m(mg/g)	9.29
	K_E (L/mg)	0.005
	R^2	0.9984
Temkin	A_T (L/mg)	1.6807E-05
	b_T (kJ/mol)	-0.239
	R^2	0.998
Harkin-Jura	A	-3.33E+04
	B	-1.998
	R^2	0.9965
Halsey	n_H	-8.66
	K_H	1.20E-18
	R^2	0.9975

3.5 Impact of Wastewater pH

pH has a very important impact during adsorption as it responsible for charge distribution on the adsorbent surface. Results of the effect of pH on Pb(II) adsorption using groundnut shell is presented in Fig. 6a for removal efficiency and Fig. 6b for residual concentration. Maximum removal efficiency of about 92% occurs and pH 2 and pH of 10 (Fig. 6a), with lowest removal of about 62% occurring at pH of 7.5. The highest residual Pb(II) concentration 37.5 mg/L was also obtained at pH of 7.5 (Fig. 6b). Authors Usifoh et al. [37] studied the adsorption of Pb (II) ions from aqueous solution using functionalized and carbonized groundnut shell and reported that maximum lead adsorption occurred at pH 11 for all adsorbents tested. According to them, at higher pH values, increase in adsorption may be attributed to the abundance of OH^-, which causes increase in diffusion of the Pb^{2+} ions. Authors OuYang et al. [13] studied the adsorption of Pb(II) from solution using peanut shell and reported that adsorption efficiency increased gradually in the pH range from 2 to 5 and reached to a maximum value around pH 5, then followed by a decreased. Decreasing trend in uptake at of 7.5 and pH 8 is due to formation of soluble hydroxyl complexes. Authors Duwiejuah et al. [38] reported 100% lead removal at pH of 5.48 to 7.1 using groundnut and sheanut shells biochars.

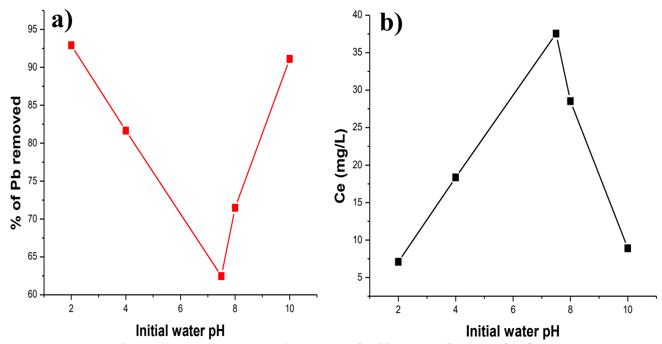

Fig. 6 Impact of wastewater pH on a) removal efficiency b) residual concentration of Pb(II) adsorption colour and using groundnut shell

3.6 Impact of Wastewater Temperature

Results of the effect of temperature on Pb(II) adsorption from wastewater are presented in Fig.7a for removal efficiency and Fig.7b for residual concentration. 94% of Pb(II) was removed at 20 °C, then decreased slightly to 91% ant 25 °C before rising to 94% at 35 and 45 °C. The removal efficiency reduced again to about 92% at 60 °C. The highest residual concentration of about 9 mg/L occurred at 25 °C (Fig. 7b). These results contradict those of Duwiejuah et al. [38] who reported that temperature ranging from 20 °C to 60 °C had a negligible effect on the adsorption of Pb(II) on functionalized and carbonized groundnut shell. However, authors Qaiser et al. [12] also obtained maximum Pb(II) removal at 20 °C on groundnut shell but removal efficiency decreased with increasing temperature from 20-45 °C. The decrease at 25 °C is due a change in the texture of the groundnut shell biomass due increasing temperature and hence a reduction in surface area available for adsorption. However, the increase in removal efficiency at 35 and 45 °C is likely due to the i) creation of new active sites resulting from heating and ii) acceleration of some originally slow adsorption steps and enhancement of the mobility of adsorbate from the bulk solution towards the adsorbent surface [39]. The reduction in removal efficiency at 60 °C is likely due to the destruction of active sites as carbon rich materials are very sensitive to heating.

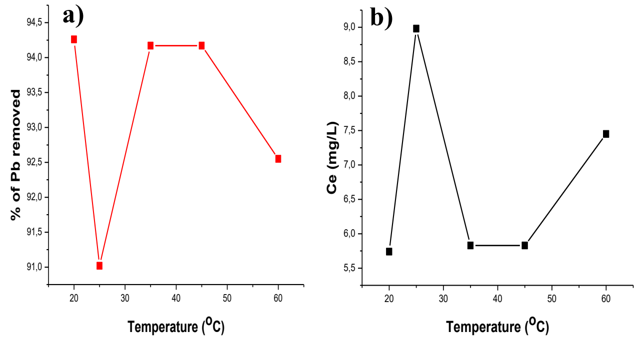

Fig. 7 Impact of wastewater temperature on a) removal efficiency b) residual concentration of Pb(II) adsorption colour and using groundnut shell

The thermodynamic parameters calculated are presented on Table 5, where it can be observed that Pb(II) adsorption on groundnut shell is very spontaneous with negative Free energy (ΔG°) at all the temperatures studied. Negative enthalpy value of -6.37 KJ/mol indicate an exothermic process of Pb(II) adsorption on groundnut shell adsorbent. The works of Qaiser et al. [12] also reported that Pb(II) adsorption on groundnut shell is exothermic. The negative value of entropy obtained as 0.0021 kJ/molK also shows that there is decrease randomness of Pb(II) molecules around the groundnut shell surface. Generally, the change of ΔH° for physisorption is between −20 and 0 kJ/mol, the physisorption together with chemisorption is from 20 to −80 kJ/mol and chemisorptions is in a range of −80 to −400 kJ/mol [32]. The enthalpy values obtained in this study is in the −20 and 0 kJ/mol range, indicating Pb(II) adsorption on groundnut shell occurs by physisorption mechanism. This confirms the conclusions drawn from apparent energies calculated from Dubinin- Radushkevich and Temkin isotherms.

Table 5 Thermodynamic parameters for Pb(II) removal using groundnut shell

T(K)	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (kJ/molK)
298	-5.56		
303	-5.74	-6.37	-0.0021
308	-6.05		
318	-5.79		
333	-5.48		

3.7 Toxicity Assessment of Treated Wastewater and Use Adsorbent on Beans Growth Performance

The germination of beans was monitored for 11 days in all the ten treatments and results are presented in Fig. 8a for 100 mg/L Pb(II) concentration (raw & treated), Fig. 8b for 50 mg/L Pb(II) concentration (raw & treated), Fig. 8c for10 mg/L Pb(II) concentration (raw & treated), Fig. 8d for 1 mg/L Pb(II) concentration (raw & treated), and Fig. 9 for used groundnut shell adsorbent.

The number of flowers produced in each treatment are given in Fig. 10a for 100 mg/L Pb(II) concentration (raw & treated), Fig. 10b for 50 mg/L Pb(II) concentration (raw & treated), Fig. 10c for10 mg/L Pb(II) concentration (raw & treated), Fig. 10d for 1 mg/L Pb(II) concentration (raw & treated), and Fig. 11 for used groundnut shell adsorbent.

The number of pods produced in each treatment are given in Fig. 12a for 100 mg/L Pb(II) concentration (raw & treated), Fig. 12b for 50 mg/L Pb(II) concentration (raw & treated), Fig. 12c for10 mg/L Pb(II) concentration (raw & treated), Fig. 12d for 1 mg/L Pb(II) concentration (raw & treated), and Fig. 13 for used groundnut shell adsorbent. Results of each treatment were compared with the control.

For treatments in Fig. 8, the germination was delayed in the treatments with raw untreated wastewater and the control while treatments with

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treated wastewater showed enhanced germination over the control and raw untreated wastewater throughout the study period. From Fig. 9, the used groundnut shell adsorbent had a very significant germination over the control. In all the treatments, 100% germination was observed on day 11 and data collection on germination was stopped on this day.

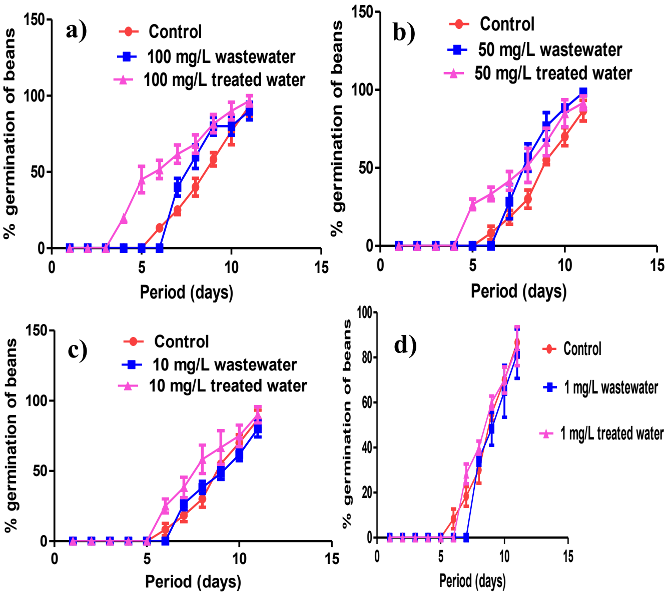

Fig. 8 Effect of raw and treated wastewater containing lead concentration in a) 100 mg/L b) 50 mg/L c) 10 mg/L d) 1 mg/L on beans germination percentage

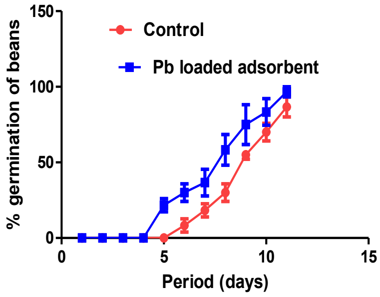

Fig. 9 Impact of used groundnut shell adsorbent from the treatment of wastewater containing lead on germination percentage

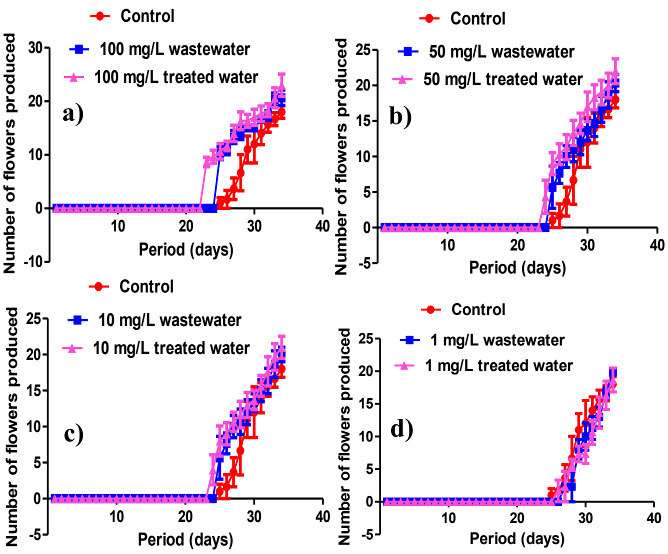

Fig. 10 Effect of raw and treated wastewater containing lead concentration in a) 100 mg/L b) 50 mg/L c) 10 mg/L d) 1 mg/L on number of flowers produced by beans

For treatments in Fig. 10, flowers production was enhanced in treatments with raw and treated wastewater compared with the control except for 1 mg/L concentration treatments where the control had a better flowers performance initially. From Fig. 11, the used groundnut shell adsorbent had a very significant flower production performance over the control.

For treatments in Fig. 12, pods production which is the yield of the beans was in the order: treated wastewater>control>untreated wastewater for all the studied Pb(II) concentrations. From Fig. 13, the used groundnut shell adsorbent had a very high pod production compared to the control. The highest pod production was obtained in the used groundnut shell adsorbent treatment.

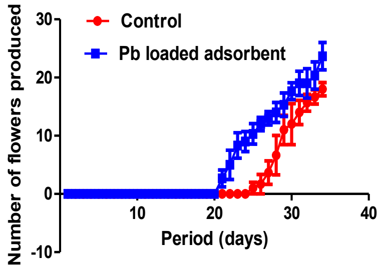

Fig. 11 Impact of used groundnut shell adsorbent from the treatment of wastewater containing lead on number of flowers produced

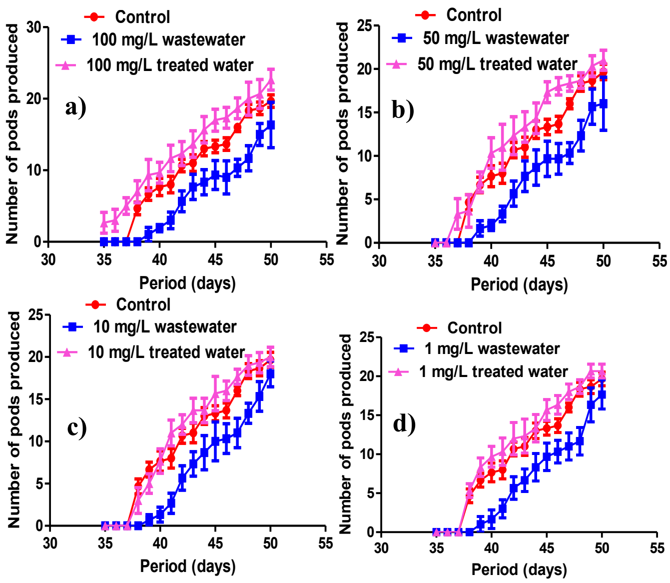

Fig. 12 Effect of raw and treated wastewater containing lead concentration in a) 100 mg/L b) 50 mg/L c) 10 mg/L d) 1 mg/L on number of pods produced by beans

Results of germination obtained in this study are better than those of Tsamo et al. [40], who irrigated beans with 40, 200 and 600 µmol/L of Pb^{2+} concentrations and only 33% germination was obtained for each concentration against 66% for the control. The study of Elkhatib [31] investigated the effects of lead on radish plants (*Raphanus sativus* L.) seed and plant germination, growth and yield. Seeds and plants were treated with different concentrations of Pb: 0, 100, 200 and 300 ppm for 120 hours. 97.47% germination was obtained in the control, 93.33% with 100 mg/L Pb concentration, 86.76% with 200 mg/L Pb concentration and 63.92 with 300 mg/L Pb concentration. More lead accumulated in root tissues than in shoot tissues. They concluded that higher concentration of lead (300 ppm) caused a significant decrease in seed germination, plant height, leaves fresh and dry weights, leaves area and yield. Meanwhile, the studies of Ekinci et al. [41] investigated beans grown in soil contaminated with lead at different concentrations (0, 1000, 1500, 2000 mg/kg) and concluded that concluded that here was a significant decrease in growth and chlorophyll contents of bean seedlings in parallel with the increased lead dose. Authors Aicha et al. [1] studied the effect of six concentrations of lead nitrate (0.50, 100, 150, 200 and 250 ppm) at five repetitions on seeds of *Cleome amblyocarpa* Barr. & Murb stressed after 15 days of germination. Like in this study for untreated wastewater, they concluded that the seed germination rate decreases insignificantly relative to the control. This also justifies why treated water had early (day 3 or 4) and sustainable germination. Other studies presented clearly shows that low level of lead in water cannot significantly affect plants particularly beans growth. This means the low residual concentrations obtained in this study are not likely harmful to plants. This justify why number of pods were highest in the treatments with treated wastewater where residual lead concentration was very low.

The recycled Pb-loaded groundnut shell adsorbent in this study showed high performance in beans germination performance (Figs. 9, 11 & 13). Treatments with recycled adsorbent had the highest number of flowers and pods produced compared to beans grown on soil with raw and treated

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water at all the concentrations tested as well as the control. For example, it had about 30 flowers and pods each produced against <25 each for all other treatments. This is likely because the lead on the adsorbent was not at toxic levels or the adsorbed lead may be embedded in the groundnut shell structure that makes it not available for plant uptake.

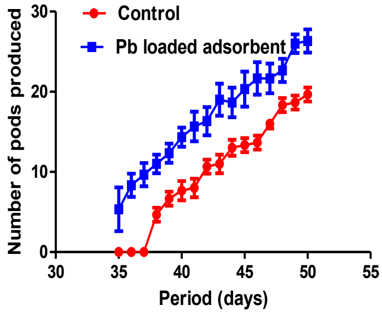

Fig. 13 Impact of used groundnut shell adsorbent from the treatment of wastewater containing lead on number of pods produced

However, the addition of peanut shells to the soil as organic amendment may increase nutrient levels such as carbon, nitrogen, phosphorus, and calcium structure, reduce soil salinity and enhance growth performance [42]. Bako et al. [42] studied the effects of groundnut shells on soil properties, growth and yield of Maize using 0.0, 2.5, 5.0 and 7.5 t/ha doses. Their results showed that groundnut shells application increased soil porosity, water holding capacity, soil pH, organic matter, nitrogen, phosphorus, calcium, magnesium, potassium, sodium and total exchangeable bases and decreased soil bulk density to favorable levels.

4. Conclusion

The study investigated the efficiency of groundnut shell in removing Pb(II) from synthetic wastewater, the effect of the treated and raw wastewater at four initial lead concentrations and the groundnut shell loaded with lead after adsorption on the growth performance of beans. The effect of contact time, adsorbent dose, initial lead concentration, pH and temperature were used to evaluate the lead adsorption process. Untreated Pb(II) containing wastewater (Pb(II) (concentrations: 100 mg/L, 50 mg/L, 10 mg/L and 1 mg/L), groundnut shell treated Pb(II) containing wastewater Pb(II) (concentrations: 100 mg/L, 50 mg/L, 10 mg/L and 1 mg/L), and the recovered Pb(II) adsorbent were respectively used to irrigate and fertilize the soil and beans growth performance on the soil monitored for 50 days. Groundnut shell with smaller particle sizes presents a more block and smooth structure with well-structured pores, while those with large particle sizes show a more irregular fiber-like sheets with multi-hole porous structures. Even though, the process was not stirred, 90% of lead was adsorbed in 5 minutes. Removal efficiency decreases with increase in adsorbent dose and an increase in residual concentration. Removal efficiency increases with increasing concentration, hence generating a very low residual concentration. Maximum removal efficiency of about 92% occurred and pH 2 and pH of 10, with lowest removal of about 62% occurring at pH of 7.5. A maximum of 94% of Pb(II) was removed at 20, 35 and 45 °C and the removal process was very spontaneous at all the temperatures studied and exothermic. Pseudo-second-order showed a good fit with R^2 of 0.9997 and an agreement between calculated and experimental amounts adsorbed. Langmuir model best described the process of Pb(II) removal by groundnut shell adsorbent. The adsorption energy (kJ/mol)) value of –0.239 was obtained from Temkin isotherm, indicating Pb(II) is adsorbed on groundnut shell by physical mechanism. Harkin–Jura isotherm model showed that groundnut shell surface has a heterogeneous pore distribution and the possibility of multilayer adsorption of Pb(II) on it. The fitness of Hasley isotherm, indicate also that the surface of groundnut shell is heterogeneous in nature.

Germination of beans was delayed in the treatments with raw untreated wastewater and the control while treatments with treated wastewater showed enhanced germination. Flowers and pod production by beans was higher with treated wastewater compared to the control and raw untreated wastewater. However, highest growth performance was obtained in the used groundnut shell adsorbent treatment. Results of this study shows that groundnut shell is very effective in removing lead from wastewater, producing treated water and lead adsorbed groundnut shell waste adsorbent that enhances beans growth performance more than the untreated Pb(II) wastewater and the control. However, it is recommended that lead accumulation in the beans grains after harvest be analyzed to prevent lead contamination after consumption.

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