The Adsorption Studies for the Removal of Lead (Pb^{2+}) from Paint Industrial Wastewater using Avocado Seed Activated Carbon (ASAC)

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ABSTRACT

The rapid increase in industrial activities in Nigeria has led to the high contamination of water bodies with heavy metals such as Lead, Cadmium, Zinc etc from wastewater disposal. Previous techniques adopted for this wastewater treatment such as ion exchange, biosorption, solvent extraction and precipitation etc are highly expensive and capital intensive; hence, the need for cheaper technologies that utilizes locally available biomass as a precursor for the preparation of activated carbon. This study aims to utilize avocado seed for the production of activated carbon to treat paint industrial wastewater containing heavy metals such as Lead (Pb^{2+}) and to evaluate the adsorption isotherm and kinetic models that best fit the equilibrium data obtained from the batch adsorption experiment. The avocado seed activated carbon (ASAC) was prepared by carbonizing the sample at a temperature of 700 °C and activating it with a 30 % v/v Concentration of H_3PO_4 acid at a temperature of 500 °C for 6 hours in a muffle furnace. The batch adsorption studies were done by investigating the varying effect of metal ion concentration (0.5-3.0 mg/L), contact time (15-180 mins) and adsorbent dosage (0.5-3.0 g). The data obtained were analysed using six adsorption isotherms such as Langmuir, Freundlich, Temkin, Toth, Sips and Redlich-Peterson methods. Freundlich isotherm model with an R^2 value of 0.94 and adsorption capacity of 0.1965 mg/g showed the highest percentage removal of Lead (Pb^{2+}) to be > 97 %. The kinetic studies revealed that the experimental data fitted well to the pseudo-first-order model with a high correlation coefficient of 0.980 when compared to other models like pseudo-second-order, intra-particle diffusion and Boyd model, thus; signifying that the adsorption's mechanism was physiosorption. Thus, optimum activation conditions would produce higher activated carbon from avocado seeds that could be used for the removal of heavy metal ions from industrial wastewater, which will aid in eliminating the challenge of agricultural waste accumulation in the environment.

Keywords: Adsorption studies, Avocado seed, Batch adsorption, Isotherm models, Kinetic models, Activated carbon, Wastewater treatment, Chemical activation, Lead (Pb^{2+}) removal, Biomass utilization, waste-to-wealth creation.

INTRODUCTION

Wastewater from numerous industries such as paints and pigments, dye and textile, glass production, mining operations, metal plating, and battery manufacturing processes are known to contain metallic pollutants allied with "Heavy metals". [1,2]. These "Heavy metals" such as Lead (Pb), Cadmium (Cd), Chromium (Cr), Nickel (Ni), Zinc (Zn), Copper (Cu) and Iron (Fe) present in industrial wastewater are non-biodegradable and their existence in receiving lakes and streams cause bioaccumulation in living organisms leading to several health complications in plants, animals and human beings such as cancer, kidney failure, metabolic acidosis, oral ulcer, renal failure and damage in the stomach of the rodent [3,4,5]. As a result of the degree of the problems caused by heavy metals pollution, the removal of heavy metals from wastewater is imperative [6,7,8]. In recent times, investigation into new and cheap methods of metal ion removal has been on the increase.

Recently, efforts have been made to use cheap and available agricultural wastes such as coconut shell, coconut coir, orange peel, rice husk, peanut husk and sawdust as adsorbents to remove heavy metals from wastewater [9,10,11,12]. Avocado, also known as *Persea americana*, is a fruit-producing plant predominantly found in Southern Nigeria. Avocado seeds contain starch, reducing sugars, fibre, arabinose, pentose, and protein and starch and cellulose have high molecular mass polysaccharides; consisting of carbon, hydrogen, and oxygen compounds. Avocado seeds have a water content of 12.67%, an ash content of 2.78%, and a mineral content of 0.54% higher than other fruit seeds [13]. Avocado seeds contain high starch, indicating that high carbon content [14]. According to Menendez-Diaz and Martin-Gullon, (2006) [15], activated carbons are extremely versatile adsorbents and have major industrial significance. They have high specific porosity and hence enhanced surface area. Thus, they are used in a wide range of applications, particularly with the removal of species by adsorption from liquid or gas phases. Activated carbons can be considered to be composed of non-graphitic, non-graphitizable carbons with a highly ordered microstructure. Heavy metal is any metallic chemical element that is poisonous at low concentrations and when consumed in permissible quantities causes psychological disorder [16,17]. They are highly toxic for both animal and human beings and can be bio-accumulated through biological chains and are non-biodegradable [16,17,18]. The industrial discharge of these heavy metals which adversely contaminates waterways such as nearby streams and rivers, irrigation water, the application of fertilizer and meta-based pesticides, harvesting process, transportation, storage etc., leads to an increase in the toxicity level [17,19]. Elements, such as Cadmium and Chromium, are considered carcinogenic, while Iron, Copper, Manganese, Zinc, and Nickel are considered essentials [20]. Painting paper, pigment, fuels, photographic materials electroplating, battery manufacturing, explosive manufacturing, and metalworking industries discharge large amounts of heavy metals, including Copper, Zinc, Lead, and Nickel ions into water bodies which causes serious environmental contamination [16].

MATERIALS AND METHOD

Materials:

Avocado fruits were obtained at Ukam Market, Mkpat Enin L. G. A., Akwa Ibom State. The fruits were selectively picked, and the endosperm (copra) was separated from the shell. The shell was soaked for about one hour in the water, well-scrubbed using a sponge and tap water and then sun-dried for three (3) hours and dried seed will then be placed on a clean hard surface and broken into smaller pieces with the use of a hammer these smaller pieces were dried in a Laboratory Oven (N53C-Genlab) at 110^{o}C for 3 hours to constant (dry) weight. The crushed seed samples were carbonized in a muffle furnace (PECVD furnace) to a temperature of 700^{o}C for 1 hour in the absence of oxygen. The sample will then be impregnated by immersing it in a 30% concentration of Phosphoric acid (H_3PO_4) solution using an impregnation ratio (I.R) of (1:2 w/w) for 24 h and thereafter dried in a laboratory drying oven at a temperature of 105^{o}C for 12 h to remove moisture and was activated in a muffle furnace at a temperature of 500^{o}C. The avocado seed activated carbon (ASAC) produced was cooled to room temperature and washed with 0.1 M NaOH solution to remove residual ash content and with deionized water until the pH of the washing solution reached 6-7. The prepared ASAC was then oven-dried at 105^{o}C µm particle sizes and then stored in an air-tight sample container until needed for the adsorption experiment.

Preparation of Aqueous Solution

All reagents used for this study were analytical reagents grade and were procured from Vikam Scientific Supplier, U J Esuene Stadium, Calabar and deionised doubly distilled (DDD) water was used throughout the experimental studies. The stock solution of Lead (Pb) solution was prepared by dissolving 1.0g of $Pb(NO_3)_2 .5H_2O$ salt in 1000 ml of distilled water.

The standard working solutions of 0.5, 1.0, 2.0, 3.0, 4.0 and 5.0 mg/L were prepared by progressive serial dilution of the stock solution of Lead salt using distilled water. The total concentration of each metal ion in the aqueous solution was confirmed by analysis using an Atomic Absorption Spectrometer- AAS (Unicam thermo/solar system 2009 model).

Batch Adsorption Equilibrium and Kinetics Studies

Batch adsorption experiments were carried out to study the varying effect of initial metals ion concentration, adsorbent dosage contact time, pH and temperature on the adsorption of Pb^{2+} on avocado seed activated carbon (ASAC). Each experimental study was carried out by measuring 100 mL of each metal ion solution containing varying initial concentrations into a 250 mL conical flask and 0.5 g of the adsorbent was added to the solution. The mixture was agitated in an orbital shaker (Rotamax 120, Reidolph) at 150 rpm and a temperature of 25 ^{O}C for 1 h to attain equilibrium. The mixture was thereafter filtered using a Whatman No 14-filter paper and the resultant residual metal ion concentration was determined using an Atomic Absorption Spectrometer – AAS (Unicam thermo/solar system 2009 model) and the amount of metal ions adsorbed was calculated, the experimental parameters such as metal ion concentration (0.5-3.0 mg/L), adsorbent dosage (0.5-3.0g) and contact time (15-180 mins) were checked to evaluate the optimum conditions for the maximum adsorption of Pb^{2+} metal ion from 100mL aqueous solution over an applied adsorbent. For each batch experimental run, the amount of Lead (Pb^{2+}) was determined using Equation 1, Equation 2 and Equation 3 respectively.

$$q\mathrm{e} = \frac{(Co - \mathrm{Ce})V}{M} \qquad \text{Equation 1}$$

$$q\mathrm{t} = \frac{(Co - \mathrm{Ct})V}{M} \qquad \text{Equation 2}$$

$$\%\ \text{removal of crude oil} = \frac{100\ (Co - \mathrm{Ce})}{Co} \qquad \text{Equation 3}$$

where C_o = initial concentration of solution (mg/L), C_e = equilibrium concentration (mg/L), C_t = concentration of solution at time, t, (mg/L), V = volume of the solution (mL) and M = mass of adsorbent used (g).

RESULT AND DISCUSSION

Characterization of the Adsorbent (ASAC)

The physicochemical properties of avocado seed activated carbon (ASAC) were determined using proximate analysis as shown in Table 1. Activated carbon is often produced free of moisture content, but if it contains moisture, it should not be more than 5% because moisture content reduces the adsorption capacity of activated carbon [21]; thus ASAC contained 0.72% moisture content. Ash content negatively influences the overall activity of activated carbon and reduces reactivation effectively. The typical range of values for commercial activated carbon (CAC) is 2-10% [22], and the ash content of ASAC was 1.90% which was well within the acceptable range. Activated carbon having a high value of fixed carbon implies that the adsorbent is having more efficiency and stability. The pH of ASAC was 7.0, and the acceptable pH for most commercial activated carbon is pH 6-7.

Table 1: Result of Physicochemical Analysis.

Parameters	**ASAC**
Moisture Content %	0.72
Volatile Matter %	1.09
Ash Content %	1.90
pH	7.00
Bulk Density (g/cm^3)	0.3146

FTIR Analysis of the Adsorbent

The Fourier Transform-Infrared (FT-IR) spectra analysis of ASAC before and after adsorption revealed the presence of several peaks indicating the presence of different functional groups within the wavelength of 600-400cm^{-1} as shown in Figure 1 and Figure 2. Figure 1 showed the FT-IR spectrum for ASAC before adsorption to have a very strong and broad absorption peak between 2370.6-2105.9 cm^{-1} which indicated the presence of C≡C stretching vibrations of alkyne groups. The absorption peaks between 1994.1cm^{-1} to 1830.1cm^{-1} and 1736.9cm^{-1} revealed the presence of C=O stretching vibration of anhydrides and C=O stretching vibrations of the Ester group respectively. Also, the absorption peaks of 1543.1cm^{-1} and 987.7cm^{-1} revealed the presence of C=C stretching vibrations of the aromatic benzene ring group and P-H stretching vibrations of the Phosphine group respectively.

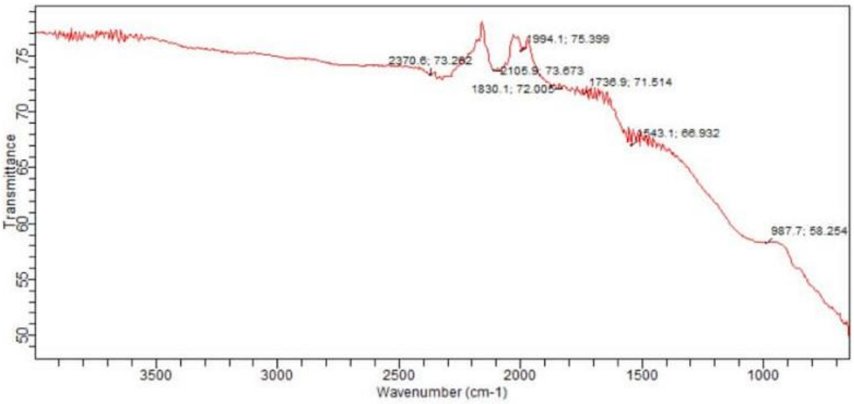

Figure 1: FTIR spectra of ASAC before adsorption.

Figure 2 shows the FTIR spectra of ASAC after adsorption which exhibited similar characteristics as the FTIR spectra of ASAC before adsorption with few modifications due to the adsorption process for the removal of Lead (Pb^{2+}) ions from aqueous solution. A broad and strong absorption peak of 3652.8cm^{-1} indicated the presence of –OH stretching vibration of the Alcohol group. The strong absorption peak of 3123.5 cm^{-1} revealed the presence of C–H symmetric or asymmetric stretching vibration of the alkene group. Reduced absorption peaks were observed between 2322.1cm^{-1} and 2083.6cm^{-1} indicating the presence of strong C≡C stretching vibrations of the alkyne group. The absorption peak of 1986.7cm^{-1} revealed the presence of a strong C=O stretching vibration of the anhydride group. The absorption peaks between 1774.2cm^{-1} and 1699.7cm^{-1} and 1543.1cm^{-1} indicated the presence of a strong C=O stretching vibration of the Ester group and C=C stretching of aromatic benzene ring groups respectively. Also, the absorption peaks of 972.8cm^{-1} and 670.9cm^{-1} revealed the presence of P-H stretching vibrations of the Phosphine group and –NH and/or –NH_2 stretching vibrations of the amine group respectively. Hence, this observation proves that functional groups such as C=O of Ester, C=C of aromatic benzene and alkyne and -OH of Alcohol groups were involved in the binding of Lead (Pb^{2+}) ion onto ASAC. After the adsorption process, the shifting of the peaks was observed which further affirms that the adsorption process actually took place and also indicated that the ASAC was a good precursor for the removal of Lead (Pb^{2+}) ions from aqueous solution.

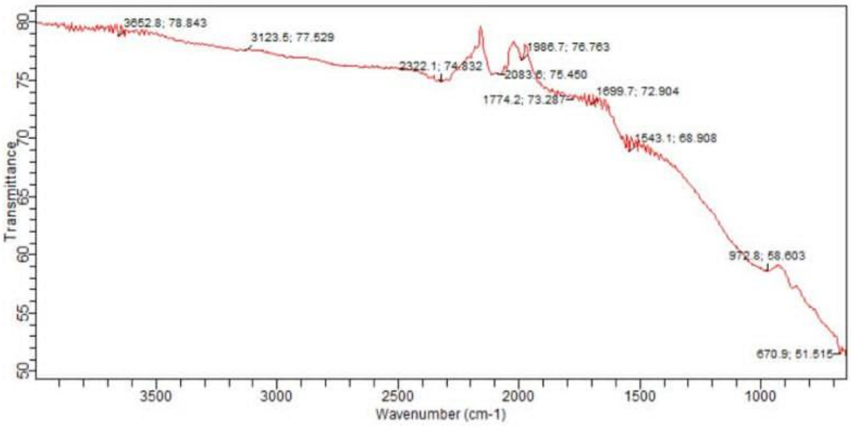

Figure 2: FTIR spectra of ASAC after adsorption.

Surface Morphology Analysis of the Adsorbent (SEM)

Scanning electron microscopy (SEM) has been extensively used to study the surface morphology of activated carbons. The surface morphology of raw/untreated avocado seed, and treated/modified avocado seed treated/modified ASAC after adsorption were observed based on the SEM images presented in Figure 3 and Figure 4. Based on Figure 3, the raw avocado seed was observed to have tiny hole-like pores with uneven porosity and also a rod-like structure with irregular sizes. The sharp edges on the structure indicate that the raw avocado seed has good crystallinity [24]. As seen in Figure 3 and Figure 4, the surface morphology of untreated avocado pear seed material was different from the treated one as the treatment significantly altered the physiochemical properties and porosity of the adsorbent material.

Figure 3: SEM analysis of ASAC before Adsorption.

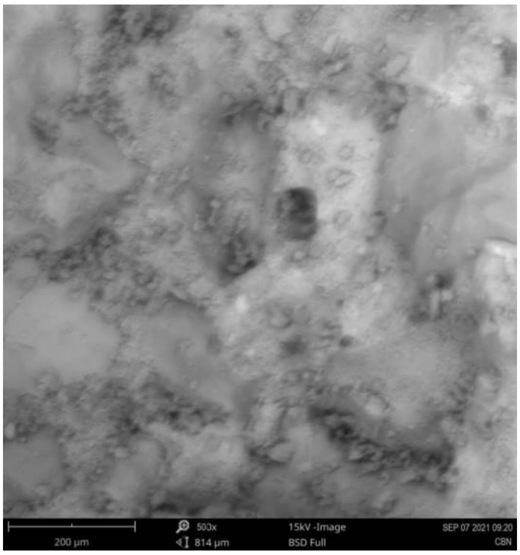

Figure 4: SEM analysis of ASAC after adsorption.

Factors Influencing the Batch Adsorption Equilibrium Studies

Effect of Contact Time: The effect of contact time on the adsorption process was studied by adding 2.0 g of the adsorbent to 100 mL of a solution having an initial concentration of 100 mg/L and pH of 7 into different 250 mL conical flasks and was then agitated for a predetermined time interval of 15-180 mins until equilibrium was attained. As illustrated in Figure 5, the rate of adsorption was rapid at the initial stages and became slow in the later stages until saturation was gradually attained. At the initial stage of 15-30 mins, the rate of adsorption was observed to increase progressively until equilibrium was attained at 135 mins. This is as a result of a large number of active surface sites available for adsorption [23,24] and all the binding sites on the adsorbent were vacant and the solute concentration gradient was high [12]. After 45 mins, all the vacant active sites were gradually filled and not much adsorption took place as the adsorbents were completely saturated. This decline was due to a decrease in total adsorbent surface area and fewer available binding sites [23,25]. It was also observed that an additional increase in the contact time did not enhance the adsorption due to the active surface sites of the adsorbent being completely occupied by the Lead (Pb^{2+}) ions.

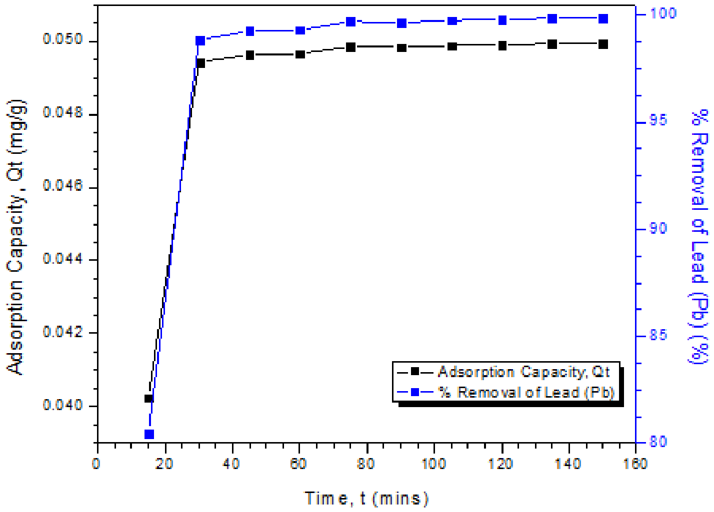

Figure 5: The effect of contact time on the adsorption capacity and % removal of Lead (Pb^{2+}).

Effect of Adsorbent Dosage: The percentage removal and adsorption capacity were both influenced by the amount of the adsorbent dosage used in the adsorption process. As shown in Figure 6, it was observed that the percentage removal of Lead (Pb^{2+}) increased with an increase in adsorbent dosage but adsorption capacity decreased with an increase in adsorbent dosage. This is as a result of the availability of more unoccupied vacant active sites for adsorption and the significant increase in the adsorbent surface area. Several other investigators have also reported a similar trend on the effect of adsorbent dosage effect in batch adsorption process [24,26].

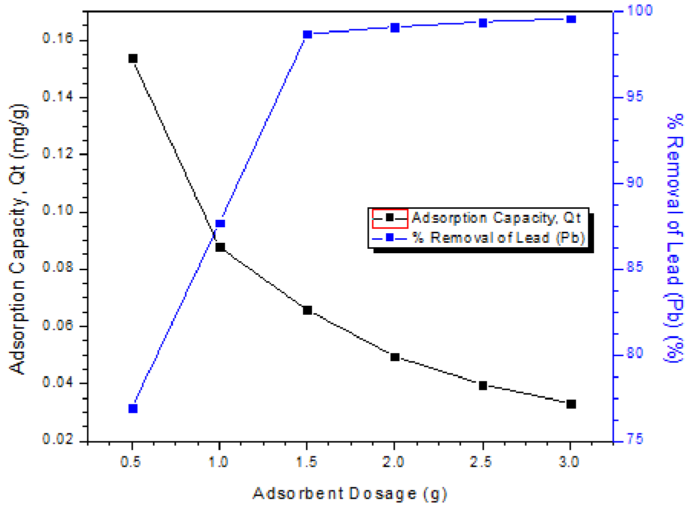

Figure 6: The effect of adsorbent dosage on the adsorption capacity and % removal of Lead (Pb^{2+}).

Effect of Initial Concentration: The effect of the initial concentration of Lead (Pb^{2+}) on the adsorption efficiency by ASAC has been systematically investigated by varying the initial concentration of Lead (Pb^{2+}) between 0.5mg/L-3mg/L. Figure 7 showed that adsorption capacity increased with a corresponding increase in the initial concentration of Lead (Pb^{2+}). This is due to the decrease in the number of active binding sites of the absorbents as a result of saturation at high concentration [12].

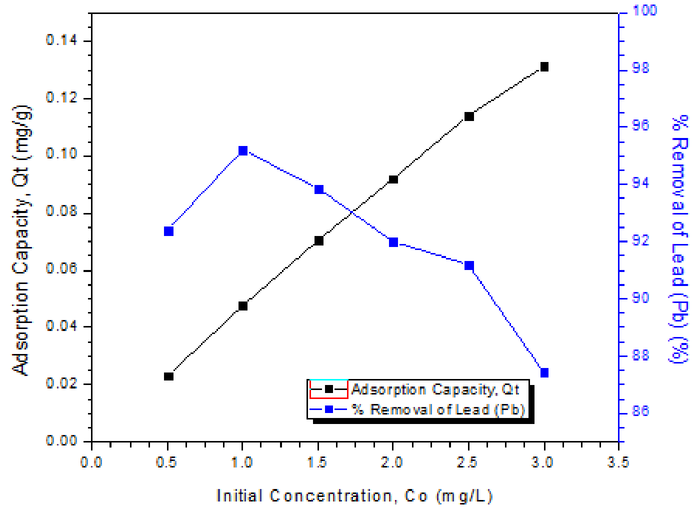

Figure 7: The effect of initial concentration on the adsorption capacity and % removal of Lead (Pb^{2+}).

Adsorption Isotherm Studies:

The adsorption isotherm study was analysed using a plot of the adsorption capacity at equilibrium, qe versus the equilibrium concentration, C_e for Lead (Pb^{2+}) adsorption onto ASAC and as illustrated in Figure 8. Six adsorption isotherm models namely; Langmuir, Freundlich, Temkin, Toth, Sips and Redlich-Peterson models were used and the experimental data obtained were analysed using nonlinear regression method. The applicability of these isotherm equations used to describe the adsorption process was determined using the correlation coefficients. R^2 value.

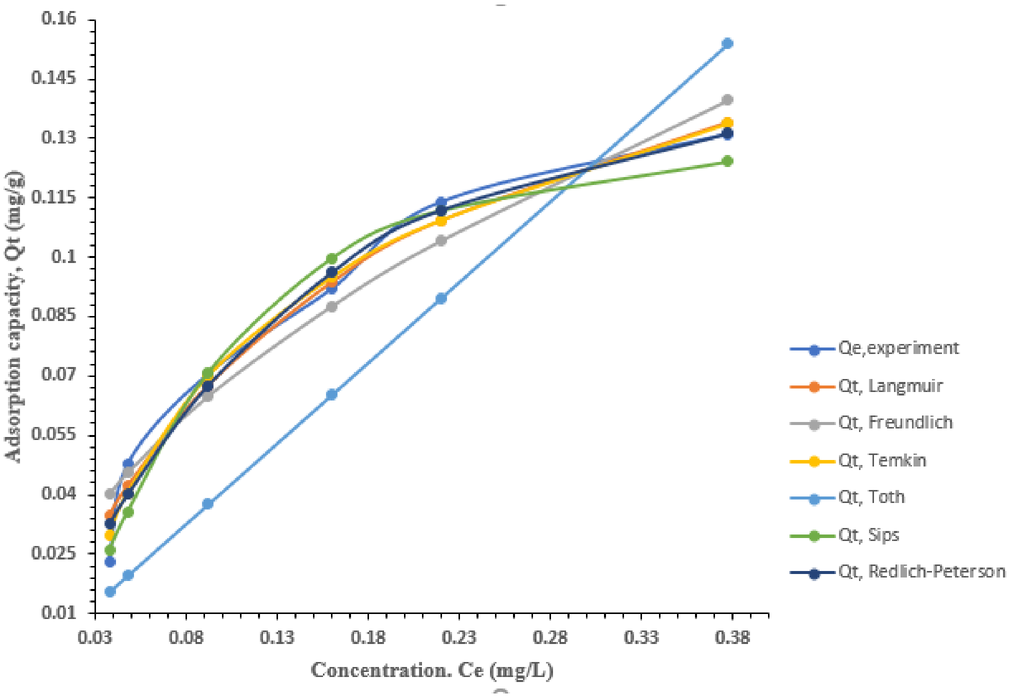

Figure 8: The Isotherm model plots for the removal of Lead (Pb^{2+}) onto ASAC.

As shown in Table 2, the adsorption data best fitted into Temkin, Redlich-Peterson isotherm models and followed by Langmuir compared to the other (Toth, Slips and Freundlich) isotherm models based on R^2 values of 0.984, 0.979 and 0.942 respectively. From Temkin and Redlich-Peterson isotherm models, the value of n is greater than 1 which means that the adsorption is a favourable normal physical process. This suggested the formation of a monolayer coverage of Lead (Pb^{2+}) adsorption onto the homogeneous distribution of the active sites on the surface of the avocado seed activated carbon (ASAC).

Table 2: Adsorption Isotherm constant for the removal of Lead (Pb^{2+}).

Adsorbent	**Adsorption Isotherm Models**											
ASAC	**Langmuir**		**Freundlich**		**Temkin**		**Toth**		**Sip**		**Redlich-Peterson**	
	q_m (mg/g)	0.1965	**K_f (mg/g)**	0.2369	**A (L/g)**	50.861	**q_m (mg/g)**	0.3621	**q_{ms} (mg/g)**	0.13373	**A**	0.9277
	K_L (L/mg)		**N**	1.8433	**B**	0.0453	**K_T (L/mg)**	1235112	**a_s (L/g)**	70.879	**B**	5.9566
							T	117.6	**B_s**	1.7371	**β**	1.307
	R^2	0.97512	**R^2**	0.9419	**R^2**	0.9841	**R^2**	0.725	**R^2**	0.969	**R^2**	0.9791

Adsorption Kinetic Studies

The mechanism of adsorption and potential rate-controlling steps such as mass transport and chemical reaction processes involved in the Lead (Pb^{2+}) adsorption from the aqueous solution was investigated using several kinetic models such as the pseudo-first order, pseudo-second order, intra-particle diffusion and Boyd models and the experimental data obtained were analysed using nonlinear regression method. Table 3 showed that the correlation coefficients for the pseudo- first order kinetic model for ASAC ($R^2 = 0.979$) was much higher and closer to unity than that of pseudo-second order kinetic model and intra-particle diffusion model, thus; concluded that the adsorption behaviour of Lead (Pb^{2+}) on ASAC predominantly followed pseudo-first order kinetic model.

Table 3: Adsorption kinetic model constants for the removal of Lead (Pb^{2+}).

Adsorbents	**$q_{e,exp}$ (mg/g)**	**Pseudo-first order**			**Pseudo-second order**			**Intra-particle Diffusion**			
		K_f (g/mg.min)	**$q_{e,cal}$ (mg/g)**	**R^2**	**K_s (g/mg.min)**	**$q_{e,cal}$ (mg/g)**	**R^2**	**K_{ip} (mg/g.min$^{1/2}$)**	**C (mg/g)**	**$q_{e,cal}$ (mg/g)**	**R^2**
ASAC	0.0499	0.11216	0.04997	0.9797	5.609794	0.0512	0.834	0.000723	0.0425	0.0514	0.6379

This elucidated the fact that the overall rate of adsorption process was controlled by physiosorption which involved co-valent bonding forces through the sharing or exchange of electrons between the adsorbents and adsorbate Lead (Pb^{2+}) and also that the rates of surface reaction was more prominent than chemical reaction (chemisorption). The possibility of intra-particle diffusion resistance influencing the rate-determining step in the adsorption process was also investigated and the result is as shown in Table 3. Eba *et al.* (2010) [27] stated that the larger the intercept (boundary layer effect; C) for intra-particle diffusion model, the greater the contribution of the surface sorption in the rate- controlling step (Auta and Hameed, 2011) [28]. This implied that the correlation coefficient and thickness of the boundary layer for ASAC ($R^2 = 0.6374$; C = 0.0425 mg/g) validated the existence of some degree of boundary layer control in the adsorption process which also indicated that the intra-particle diffusion was not the only rate-limiting step, but other processes might as well have controlled the rate of adsorption. The Boyd model which is widely used for studying the mechanism of adsorption was also used to determine whether the main resistance to mass transfer was in the thin film (boundary layer) surrounding the adsorbent particle or in the resistance to diffusion inside the pores. The linearity test of the plot of B_t against time was used to distinguish between the film and particle-diffusion controlled adsorption mechanism as shown in Figure 9.

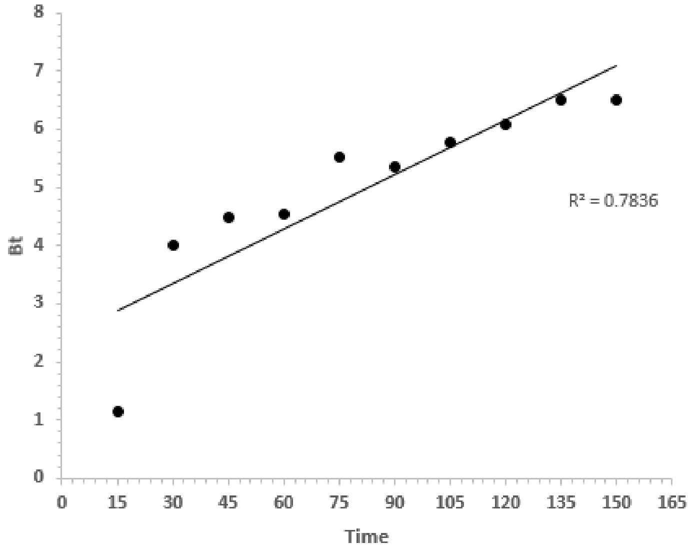

Figure 9: The Boyd model plot for the removal of Lead (Pb^{2+}) onto ASAC.

CONCLUSION

This study focused on the adsorption of Lead (Pb^{2+}) from an aqueous solution using ASAC as a low-cost adsorbent and the conclusions drawn from the study are as follows:

i. (Avocado seed can be utilized as a precursor to produce activated carbon for treatment of industrial wastewater containing hazardous metals which also minimizes the environmental degradation caused by the incessant dumping of these agricultural waste to achieve a cleaner and sustainable environment as well as provide an avenue for waste-to-wealth creation.
ii. The kinetic data fitted well into the pseudo-first order model equation indicating that surface diffusion mechanism was more prominent.
iii. The experimental data were analysed using Langmuir, Freundlich, Temkin, Toth, Sips and Redlich-Peterson models and the Langmuir model provided the best correlation for the experimental equilibrium data which suggested the formation of a monolayer coverage of Lead (Pb^{2+}) adsorption onto the homogeneous distribution of the active sites on the surface of the avocado seed activated carbon (ASAC).
iv. The energy of adsorption of Lead (Pb^{2+}) using ASAC indicated that it was a physiosorption process.
v. The chemical activation of ASAC with 30% v/v concentration of H_3PO_4 acid effectively enhanced its surface morphology; thus increasing the adsorption capacity and % removal of Lead (Pb^{2+}).

Activated carbon usage is widely accepted in several industries; hence, it is recommended that more research works should be done to explore and focus on the use of natural biomass as precursors applying varying activation methods and the use of optimization techniques be employed in the production and regeneration process of the spent adsorbent.

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