Isotherm and Kinetics Studies of Pb (II) Adsorption onto *Pandanus candelabrum* Activated Carbon

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ABSTRACT

Pandanus candelabrum activated carbon was prepared with sodium hydroxide as a chemical agent. The surface morphology, elemental composition, and functional group of adsorbents was analyzed with SEM, EDX, XRD, BET, and FTIR. Batch adsorption of Pb (II) was used to examine the effect of variables like, amount of adsorbent, temperature effect, interaction time, and pH. Freundlich model described best the isotherm parameters and Pseudo-second-order equation fitted the kinetics of the sorption onto NAC. Maximum adsorbent capacity of 13.45 mg/g was recorded at 0.8 g dosage and pH 4. Thermodynamic interaction was spontaneous and exothermic. This has revealed the feasibility and suitability of Pandanus candelabrum an economic and abundant precursor for adsorbent production and treatment of real wastewater.

(Keywords: *Pandanus candelabrum*, activated carbon, chemical activation, adsorption, isotherm, kinetics)

INTRODUCTION

Heavy metals presence in consumable water or wastewater whether in a trace or major quantity can still be perceived as environmental hazard because they are toxic and non-biodegradable (Hina, *et al*., 2017). They are persistent and indestructible and can cause significant risk to both human and animal life when they accumulate in the ecosystem (Goher, *et al*., 2015). This menace is present around us and heavy metals are discharged into water bodies via various industrial process like chemical catalysis, metal surface finishing products, and battery manufacturing has resulted to major environmental challenge (Rao, *et al*., 2007, Goher, *et al*., 2015).

Lead can be introduced into the environmental or water bodies through effluent from industries like textiles, dyes, batteries, refineries, and metal plating. Exposure of human life to these metals can result in vomiting, diarrhea, dermatitis, cancer and this can eventually lead to loss of life (Salem and Awwad, 2014). In 2017, the organization in charge of world health issues reported that drinking water is commonly contaminated by metals like nickel, mercury, cobalt, lead, copper, and cadmium. The organization identified lead among the most toxic and stated that they are source of environmental challenge, even at a very low concentration. This has necessitated environmental organizations like the World Health Organization to pronounce 0.05 mg/L as maximum permissible for Pb in water (WHO, 2017).

One of the major challenges to overcome today is the decontamination of harmful pollutants including heavy metals, from the environment or ecosystem. Therefore, the search for cheap, easy to use, affordable, and accessible methods for treating wastewater is a continuous process. The concepts of any new adsorbent or process must be feasible economical, operation friendly and affordable. Factors such as simplicity and effectiveness must also be considered (Ahmad, *et al*., 2009).

A large area with active surfaces, a well-developed pore network, as well as cost-effectiveness and environmentally friendliness are among the main factors that have continue to make activated carbon the most popular among other adsorbents. Some of the precursors that have being used by several researcher to prepare activated carbons are Kenaf stem (Shamsuddin, *et al*., 2016), Mango kernel (Rai, *et al*., 2016), Corn stalk (Wang, *et al*., 2014), and Tree bark (Dim, 2013) and have produced activated carbons with good adsorption capacities which can be comparable to other popular adsorbents. However, the quest to get low cost and effective adsorbents is still in progress. To the best of our knowledge, *Pandanus candelabrum* stem has never been used to synthesize active carbon via chemical activation. Therefore, the objective is to study the synthesis of active carbon using Pandanus candelabrum stem as a precursor and to test its performance in removing Lead from aqueous media.

MATERIALS AND METHODS

Pretreatment and Adsorbent Preparation

Pandanus candelabrum stem was obtained from Ogbunka, Anambra State, Nigeria. To remove impurities, it was washed repeatedly and was dried for 6 h at 80 °C. 15 g of pulverized Pandanus stem was impregnated with activating agents in a ratio of 1:4 (w/w) NaOH (Kilic, *et al*., 2012) by weight for 24 h. To ensure proper mixing and penetration of the agents into the internal structure of precursor, the doping was done at 85 °C. After impregnation, it was followed by carbonization at 600°C for 1 h in a furnace, and was cooled by open air. The resulting product was neutralized by washing it several times, and for drying for 4 h at 105 °C. The product was pulverized to sizes of 0.1 – 0.2 mm. Then the modified adsorbent labelled as NaOH Activated Carbon (NAC). A Control Activated Carbon (CAC) was produced without chemical impregnation and stored in an air enclosure.

Characterization of Adsorbents

The physicochemical properties of the *Pandanus candelabrum* stem as performed using the ASTM standard test methods (Yorgun and Yildiz, 2015, Shamsuddin, *et al.*, 2016). SEM – EDX of Model JEOLJSM 7600F, determined the morphology and elemental composition. Thermo Electron Nicolet 4700 FTIR spectrometer recorded the spectra of the adsorbent from 4000 to 500 cm^{-1} resolution. The adsorbent area was determined with NOVA 2200C model surface area analyzer manufactured by Quantachrome.

Batch Adsorption Study

0.7001 mg/L of Pb (II) solution at pH of 6.27 was contacted with 0.6 g of adsorbent, in a flask of 200 ml capacity. The content was agitated at 150 rpm for 100 min at 27°C, At the end after filtration the analysis was performed with Atomic Absorbance Spectroscopy. These parameters were studied with the following experimental conditions, contact time was between 20 -100 min, amount of adsorbent is 0.2, 0.4, 0.6, and 0.8 g/L, pH values were tested from 2 to 10 and temperatures were 30, 40, 50 and 60°C. In each case the sample were filtered and was analyzed. Equation (1) was applied to calculate the amount of heavy metal sorbed, q_e (mg/g),

$$q_e = \frac{(C_0 - C_e)V}{W} \qquad \textbf{(1)}$$

Where, at equilibrium, Pb (II) concentration is represented as Ce (mg L^{-1}), and initially concentration of Pb (II) was represented as C_0 (mg L^{-1}), the volumetric quantity of Pb (II) ion is V (L), and W (g) represent weight of adsorbent. Percentage of amount adsorbed (R) of metal ions as computed with equation (2):

$$\% R = \frac{(C_0 - C_t)}{C_0} \times 100 \qquad \textbf{(2)}$$

Where, at different contact time the concentration of Pb (II) ions is represented as C_t (mg L^{-1})

Desorption of Lead on NAC

Desorption experiment was carried out based on previous method as described (Das, et al., 2014). 0.8 g of adsorbent was contacted with 50 mL of lead (II) solution for 100 min. After the adsorption process, the laden adsorbent was filtered, collected and washed severally to remove excess Pb ions. After which 50 mL of HNO_3 solution at 0.1, 0.3 and 0.5 concentrations were contacted with the saturated sorbent. The content was agitated for 100 min at 800 rpm, afterward it was filtered and analyzed.

Adsorption Isotherm Study

The interaction in any process at equilibrium in aqueous system is fundamental when designing water treatment plants. The two most familiar surface adsorption model, The Langmuir isotherm is a good prediction for monolayer adsorption process (homogenous surface) (Langmuir, 1918) and Freundlich model gives a description of concentration that is suitable for a multilayer adsorption (Freundlich,1906), Equations (3) and (4), in their linear form were applied to experimental data.

Langmuir isotherm:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{1}{q_m} C_e \quad \textbf{(3)}$$

Where, the adsorbed amount of Pb (II) ion is represented as q_e (mg/g), q_m, represents a complete monolayer adsorption of metal ions (mg/g), and K_L (L/mg) is Langmuir associated constant.

Freundlich isotherm:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad \textbf{(4)}$$

Where n and K_F (L/g) represent the constants of Freundlich isotherm.

The value of (R_L) can give information on that will help to assess adsorbent effectiveness, and is defined as represented in Equation (5),

$$R_L = \frac{1}{1 + K_L C_0} \quad \textbf{(5)}$$

The value of R_L can be a guide in the classification of isotherm as, reversible (R_L = 0), favorable (0 < R_L < 1), linear (R_L = 1) and not favorable (R_L > 1).

Kinetic Study

The kinetic study was via batch adsorption method at optimum pH value of 4, 50 mL of Pb (II) solution was contacted with 0.8 g of activated carbon, it was agitated for a time range of 20, 40, 60, 80, and 100 min at 150 rpm. At each interval, some mixtures were sampled for analysis of metal ions remaining at the end (Sebata, *et al*. 2013). The two most common models frequently used in analysis of rate of chemical reaction mechanism, pseudo-first-order Equation (6) and pseudo-second-order Equation (7) were defined as represented:

$$\ln\left(\frac{q_e}{q_t}\right) = \ln q_e - k_1 t \quad \textbf{(6)}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad \textbf{(7)}$$

At any given time (t, min), amount of Pb (II) ion adsorbed is q_t (mg/g), At equilibrium point amount of Pb (II) ion adsorbed is q_e (mg/g), k_1 (mg/g) is first order kinetic constant, and k_2 (g/mg min) is second order kinetic model constant.

Thermodynamics Adsorption

The thermodynamic properties like ΔH, ΔS and ΔG were studied. Equations (8 -10) were used to calculate the aforementioned variables in Pb (II) adsorption.

$$\Delta G^o = \Delta H^o - T\Delta S^o \quad \textbf{(8)}$$

$$\ln K_c = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT} \quad \textbf{(9)}$$

$$\Delta G^o = -RT \ln K_c \quad \textbf{(10)}$$

Where, R (8.314 J/mol K) is the universal gas constant, T (K) is the absolute solution temperature. Kc is the equilibrium constant.

RESULTS AND DISCUSSION

Physicochemical properties of NAC and CAC are shown in Table 1 and 2. The elemental analysis (Table 1) show that carbon is the most abundant element (Norouzi, *et al*., 2018). The EDX spectrum indicated that carbon content of CAC is

82.51% and NAC is 84.42%, respectively, above 80% (Table 1). This shows *Pandanus candelabrum* stem can be a suitable precursor in activated carbon preparation (Hina, *et al.,* 2017). For the ultimate analyses results see Table 2.

Ash content of CAC and NAC were found to be 11.66 and 7.62%, respectively, which are comparable to other findings (Mohammed *et al*., 2015). Ash is a measure of incombustible mineral matter content (Yusuff, 2019). The moisture content of CAC and NAC were found to be 7.784 and 4.282%, respectively. The bulk density of any sample to a large extent determines its ability to uptake adsorbate. Generally, the amount of adsorbate uptake per unit volume depends greatly on the density of active carbon (Jibril *et al*., 2007). The bulk density of NAC (0.444 g/cm^3) is larger than CAC (0.354 g/cm^3) and is comparable with other reports (El-Naggar, *et al*., 2016). One of the most vital property of an adsorbent is surface area, higher surface area means plenty active sites for adsorbate adsorption. The value of 1448.9 m^2/g was determined as surface area of NAC, this value indicate chemical activation by NaOH is a suitable method (Norouzi, *et al*., 2018).

Table 1: Elemental Composition of Adsorbents.

Element symbol	Element name	Weight (%)	Weight (%)
		CAC	NAC
C	Carbon	82.51	84.42
O	Oxygen	4.06	3.79
Ca	Calcium	3.22	4.12
Ag	Silver	2.50	0.00
N	Nitrogen	1.85	1.45
K	Potassium	1.38	0.79
Si	Silicon	1.35	1.63
P	Phosphorus	1.16	1.56
Al	Aluminum	0.65	0.60
S	Sulfur	0.63	0.42
Mg	Magnesium	0.57	0.78
Na	Sodium	0.13	0.45

Table 2: The Properties of NAC and CAC

Parameter	NAC	CAC
Ash content (%)	7.622	11.663
Moisture content (%)	4.382	7.784
Bulk density (g/cm^3)	0.444	0.354
Surface area (m^2/g)	112.99	49.225
Pore volume	1.148	1.346

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectra obtained for CAC and NAC are shown in Figures 1 and 2. This analysis was to determine the chemical properties of the sample. In any adsorption process functional groups are main determinant of the mechanism and behavior of the system (Zheng, *et al*., 2014). The wide band at 3813.98 cm^{-1} is frequency stretched by O-H indicating the presence of alcoholic group. A strong and broad adsorption at 2923.39 – 2852 cm^{-1}, corresponds to C-H functional group and methyl group. The peak observed at 2360.52 – 2343.44 cm^{-1}, corresponds to presence of O-H showing the presence of phenol group. A strong bending frequency gives band at 1561.79 cm^{-1} corresponds to presence of C=C showing the presence of aromatic carbon. The stretching frequency of $-CH_3$ is found at the 1462.40 and 1377.01 cm^{-1} and that one at 1154.30 cm^{-1} is due to presence of C-O can be found to be esters. The signal at 721.61cm^{-1} represent –C-H due to H_2O (Liu *et al*., 2010).

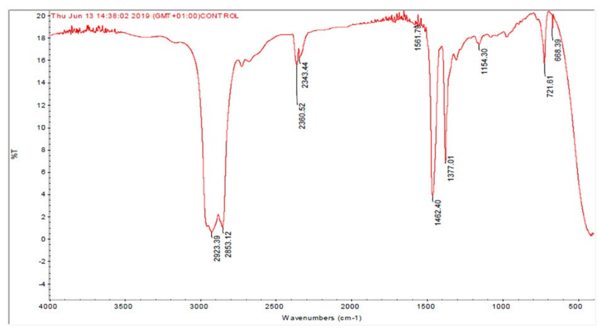

Figure 1: FTIR Spectrum of CAC.

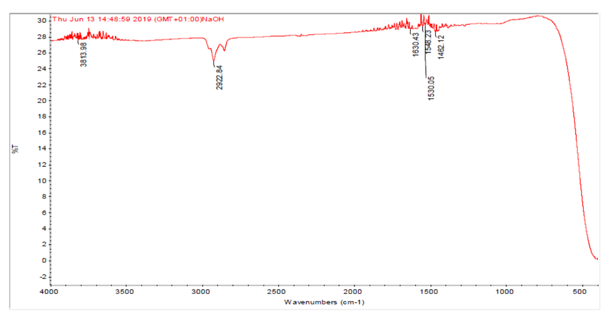

Figure 2: FTIR Spectrum of NAC.

Scanning Electron Microscopy

The SEM micrograph of CAC and NAC are shown in Figure 3. CAC micrograph is shown in Figure 3 with the following features: particles are aggregates of small particles, nonporous, with irregular shape, relatively rough surface, and without cavities. The micrograph for NAC, shows it consists of pores in the rough surfaces, due to extraction of some materials from the surface by the chemical activation (Shamsuddin, *et al*., 2016). The surface of the chemical treated adsorbent by NaOH, showed the presence of more open and connected pores and pathway than unmodified carbon. The mechanisms of activation with NaOH promotes the extraction of water molecules from lignocellulosic materials leading to the generation of porosity (Mohammed, *et al.,* 2015).

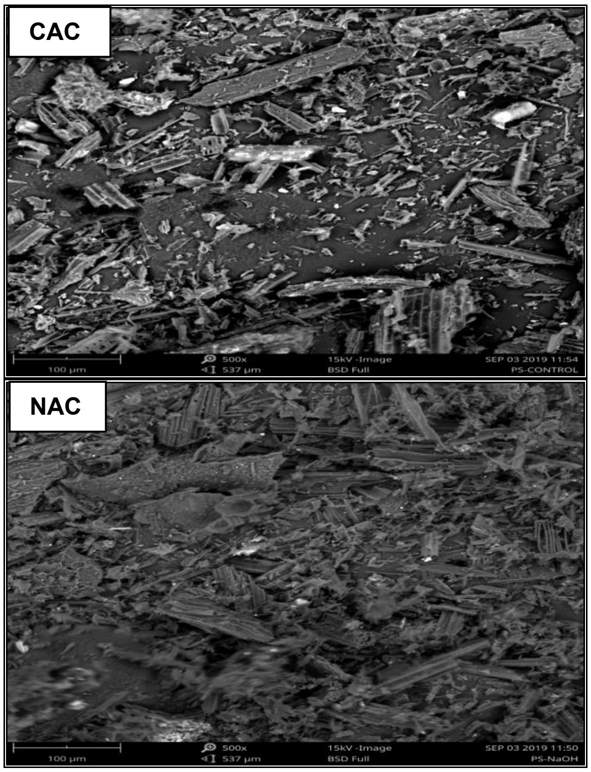

Figure 3: SEM images of CAC and NAC.

Solution pH

Among all the variables that affect adsorption, pH is the vital parameter that has strong impact on nature of charge on adsorbent surface, its speciation, and extent of ionization (Andal and Sakthi, 2010). The pH impact on adsorption of Pb (II) as examined between 2 and 10 (Figure 4).

The highest amount of Pb (II) uptake by NAC happened at optimum pH 4, with corresponding maximum percentage removal of 58%. It can be observed clearly that binding of species onto NAC relied on pH. Increase in Pb removal as the pH increased suggest depletion of positive charges on adsorbent surface as well as reducing the competing rivalry between protons and Pb within available active surface sites (Andal and Sakthi, 2010). This behavior could also suggest decrement in number of protons present in the system. Beyond pH 4, a decrease in Pb ions adsorption was observed, this suggests an increase in the contest between protons and Pb for the same active sites available for binding. And it could be that precipitation of lead ions as lead hydroxide has commenced (Das, et al., 2014, Andal and Sakthi, 2010, Uzaira, *et al*., 2012).

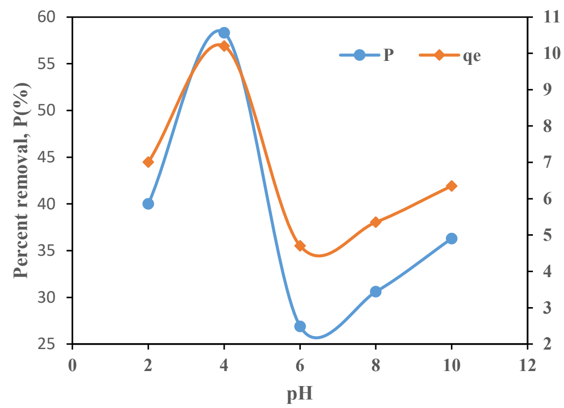

Figure 4: pH Effect on Pb (II) Adsorption by NAC.

Adsorbent Dose

Amount of dose which can remove Pb (II) from wastewater was found through a batch-mode sorption (Figure 5). Pb (II) uptake rise with the amount of adsorbent until 0.8 g was reached. The increment was recorded from 60.70 to 88.89%, that is between 0.2 and 0.8 g; the dose increased the availability of adsorption sites which results to Pb (II) removal percentage to increase. The optimum dose was found as 0.8 g, this is because beyond this dosage amount almost all ions are adsorbed. Hence any additional adsorbent made no significant contribution (Mohammed, *et al.,* 2015). On the other hand, in Figure 5, 13.4 mg/g adsorption capacity was recorded at 0.2 g dosage, while 9.8 mg/g was achieved at 1 g amount of adsorbent. This implies that increment in dose resulted to decrease in amount of metal ion adsorbed.

The high adsorption capacity at 0.2 g, suggest that lead ions present in the solution at that time was enough to saturate the adsorbent surface by occupying most of the vacant active sites available.

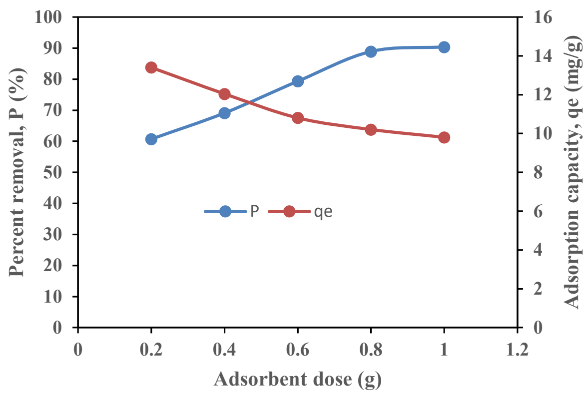

Figure 5: Effect of Adsorbent Dosage on Pb (II) Adsorption by NAC.

But, when the amount of adsorbent is increased the few active sites left are occupied faster since it is now less adsorbate against more adsorbent and this will result to reduction in adsorptive strength of adsorbent (Ahmadi, *et al*., 2016).

Results on amount of dose suggest that activated carbon prepared with NaOH can effectively treat Pb (II) contaminated wastewater. Therefore, the maximum adsorption capacity obtained was compared with other reports that used different adsorbents to remove of lead in solution, and this is presented in Table 3.

Contact Time

Pb (II) adsorption onto adsorbent as carried out within 10 – 100 min, as depicted in Figure 6. A rapid increase in the amount adsorbed with time was recorded from 5.2 to 13.4 mg/g and percent removal increased from 54.5 to 90.35 %., until equilibrium was attained at 80 min (Rao *et al.,* 2007). The rapid display at beginning of the process was attributed to high solute concentration gradient as well as vacant active sites. However, the contact time increased and metal uptake slows down, the gradual reduction of the rate showed that adsorption sites have become saturated and the ease of adsorption becomes more difficult until equilibrium was attained. The percentage metal ions removed at equilibrium time of 80 min was 90.35 % of Pb.

Similar results were also reported in the literature (Kakavandi, *et al.,* 2014).

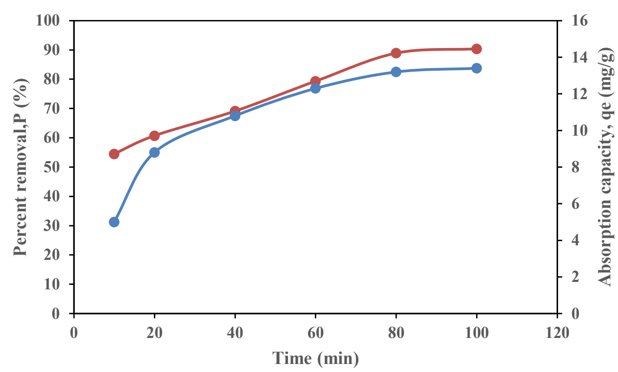

Figure 6: Effect of Contact Time on Pb (II) Adsorption onto NAC.

Table 3: Different Adsorbent and their Adsorption Capacity towards Lead (II) as Compared with Present Study.

Adsorbents	qmax (mg/g)	References
Alluvial soil	1.567	Das et al. (2014)
Phaseolus hulls activated carbon	21.8	Rao et al. (2009)
Bamboo activated carbon	0.67	Lo et al. (2012)
Activated carbon	6.68	Mishra and Patel (2009)
Saraca indica leaf powder	1.19	Goyal et al. (2008)
Acid treated montmorillonite	1.62	Akpomie and Dawodu (2016)
Sewage sludge	1.17	Hu et al. (2015)
Pandanus candelabrum AC	8.07	This study

Effect of Temperature

Temperature effect on Pb (II) adsorption at pH 4 as seen in Figure 7. Maximum adsorptive capacity was achieved at 30°C. Adsorption decreased from 13.31 to 10.92 mg/g with corresponding temperature increment within 30 to 60°C. The sorbent showed gradual decrease in metal ions adsorption as the temperature increased. The characteristics of exothermic process as displayed by adsorbent in aqueous solution could be responsible for this behavior (Gupta, *et al*., 2012). The results show that qe, decreased with temperature rise (Brigante and Avena, 2016, Ebrahimi, *et al*., 2016).

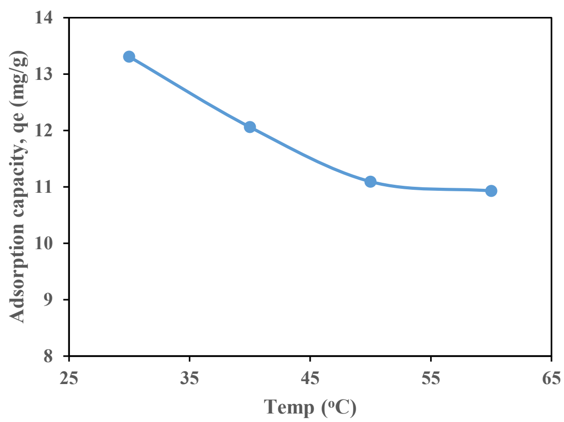

Figure 7: Effect of Temperature on Pb (II) Adsorption by NAC.

Adsorption Isotherms

Langmuir and Freundlich models described isotherm data for removal of Pb (II) as shown in Figures 8 and 9. From Table 4, the regression coefficient (R^2) values showed that the process obeyed Freundlich isotherm more than Langmuir. The adsorption occurred on a heterogenous surface for all sorption sites (Das, *et al*., 2014). The calculated separation factor (n) has a value that is less than zero. This shows that during the process of adsorption, at higher temperature, desorption is taking place (Rani and Sud, 2015, Rao, *et al*., 2007, Ridha, *et al*., 2017).

Table 4: Parameters of Adsorption Isotherms of Pb (II) onto NAC.

Isotherm Model	Parameters	Values
Langmuir Isotherm	K_L (L mg^{-1})	13.921
	q_m (mg.g^{-1})	8.071
	R_L	0.1105
	R^2	0.9947
Freundlich Isotherm	K_F (mg/g)	5.848
	N	0.474
	R^2	0.9986

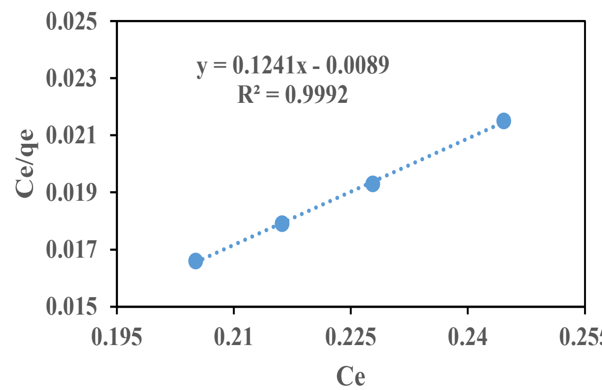

Figure 8: Langmuir Isotherm of Pb (II) onto NAC.

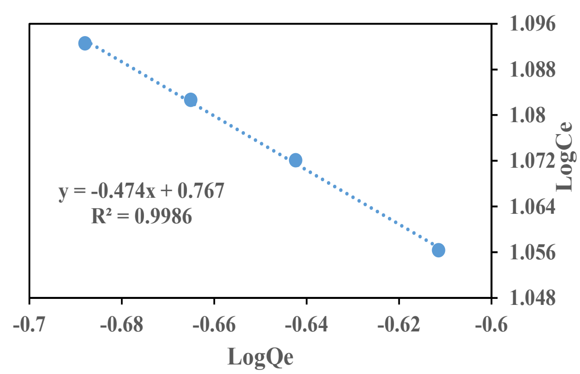

Figure 9: Freundlich Isotherm of Pb (II) onto NAC.

Adsorption Kinetics Study

The parameters of adsorption kinetic of Pb (II) was examined with Pseudo-first order and second order kinetic equation as shown in Figures 10 and 11 and Table 5. The best description of the data was obtained with second kinetic equation having regression coefficient (R^2) 0.9959, which is higher than that of first order which is 0.3474.

The adsorption capacity of first and second equation were compared with the experimental value, it was the second order value that was closer to measured adsorption capacity. This further confirmed it followed the adsorption process correctly. This suggests that the step limiting the reaction rate is chemisorption (Kilic, *et al.*, 2017).

Table 5: Parameters of Adsorption Kinetics of Pb (II) onto NAC.

Kinetic Model	Parameters	Value
Pseudo first Order	q_e (mg/g)	0.994
	K_1 (min^{-1})	0.8883
	R^2	0.3474
Pseudo second Order	q_e (mg/g)	14.728
	K_2 ($g.mg^{-1}.min^{-1}$)	0.043
	R^2	0.9959

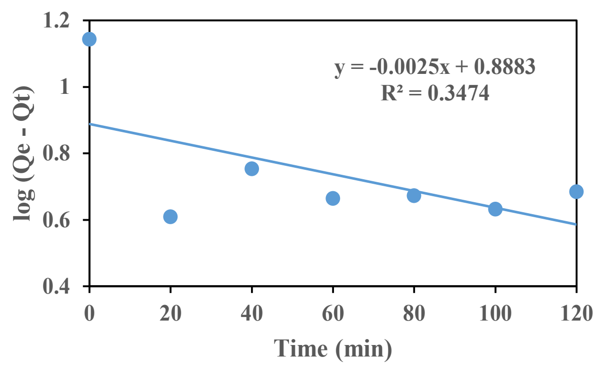

Figure 10: First Order Kinetics of Pb (II) by NAC.

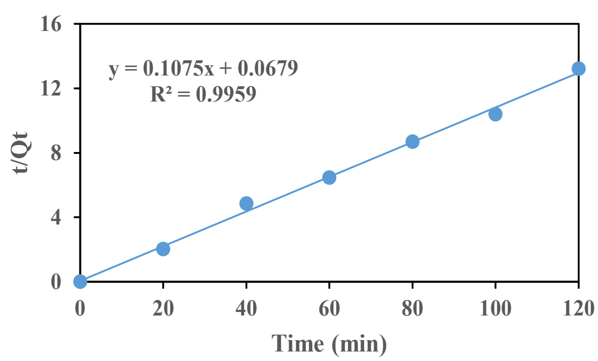

Figure 11: Second Order Kinetics of Pb (II) by NAC.

Thermodynamics Studies

The plot of InKc against 1/T and the parameters of the thermodynamic interaction are shown in Figure 12 and Table 6. ΔH°, the change in enthalpy was negative (-10.93 kJ/mol), which signifies the interaction between Pb and activated carbon surface was an exothermic process**.** The values of Gibbs free energy (ΔG°) measured at different temperatures are -9.62, -10.45, -11.73 and -12.911kJ/mol, this parameter which measures the spontaneity of a reaction process, showed increase in disorderness.

From Table 6, for the rise in temperature from 303 - 333 K, the free energy increased. This indicates a favorable spontaneous exothermic reaction**.** The entropy change (ΔS°) is negative, it was measured at 303K as - 2.32 J/molK. This signifies a strong affinity of ions for adsorbent and a high degree of randomness.

Table 6: Thermodynamic Parameters at Different Temperature.

T(K)	InKc	ΔGo	ΔSo	ΔHo
303	3.8206	-9.62	-2.32	-10.93
313	4.0144	-10.45		
323	4.3679	-11.73		
333	3.2189	-12.911		

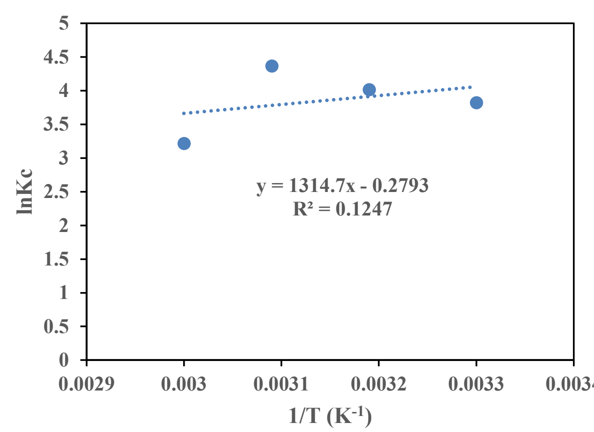

Figure 12: Plot of InKc versus 1/T for Thermodynamic Parameters of Pb (II) Adsorption onto NAC.

Desorption of NAC

Desorption process performed on NAC by HNO_3 is very important economically because it provide opportunity of resource renewability of NAC and Pb adsorbate. The results of desorption at desorbing agent solution concentrations of 0.1, 0.3 and 0.5 is presented in Figure 13**.**

Basically, in this process the arrangement of lead ions will be disorganized and liberated from NAC surface to desorption solution. It can be observed that desorption efficiency increases with increase in HNO_3 solution concentrations.

The desorption efficiency recoded at 0.1, 0.3 and 0.5 mol/L concentrations are 75.2, 88.4 and 98.8%, respectively. The maximum desorption efficiency of 98.8% of lead ions was recorded with 0.5 mol/L solution, and this is recovery rate of more than 95%.

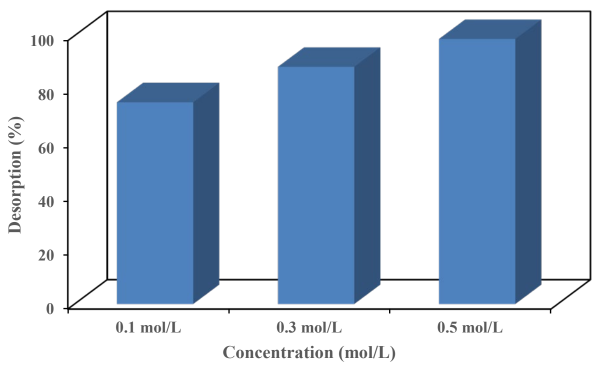

Figure 13: Desorption of NAC by HNO_3 as Desorbing Agent.

CONCLUSION

Adsorption batch tests for Pb (II) removal from sewage wastewater by *Pandanus candelabrum* stem based activated carbon was studied. The chemically treated adsorbent had surface area of 1448.9 m^2/g which was larger than untreated activated carbon. Adsorption efficiency was obtained at pH 4. Langmuir and Freundlich isotherm described adsorption process for Pb (II) removal. Regression coefficient (R^2) values showed that Freundlich isotherm followed the process adequately, on a heterogenous surface layer.

The best description of adsorption kinetics was obtained with pseudo-second order equation. Thermodynamic interaction indicated a favorable and spontaneous exothermic process. The adsorption capacity and percentage removal at equilibrium time of 80 min was 13.4 mg/g and 90.35%, respectively. This finding suggest that activated carbon prepared with NaOH can effectively treat Pb (II) contaminated wastewater.

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