



## Equilibrium and Kinetic Studies on Lead (II) Adsorption by Sugarcane Bagasse Derived Activated Carbon

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### ABSTRACT

Municipal and industrial wastewater contain a lot of contaminants. The major contaminants of concern are heavy metals. Heavy metals are known to be toxic, non-biodegradable and have a long half-life. The release of untreated wastewater containing heavy metals can cause serious problems to human, plants and animals. In this study, activated carbon was developed from sugarcane bagasse and its effectiveness in adsorbing lead ions from wastewater was examined. Batch adsorption experiments were carried out to investigate the effects of pH and initial lead concentration on the adsorption process. The batch adsorption test showed that extent of lead adsorption by sugarcane bagasse activated carbon (SCBA) was dependent to pH and initial lead concentrations. The optimum pH for lead adsorption was found to be pH 5.0. Removal of lead decreases with the increase in initial metal concentrations. The adsorption of lead ions onto SCBA follows a pseudo-second-order reaction model. The rate limiting step is a chemisorption or chemical adsorption that involves Van der Waals forces through electrons exchange between the SCBA and lead ions.

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### NOMENCLATURE

$C_i$	Initial lead concentration	$q_t$	The amount of adsorption at time t
$C_e$	Equilibrium lead concentration	$R^2$	Regression coefficient
$k_1$	Rate constant of the pseudo first order adsorption	SCBA	Sugarcane Bagasse Activated Carbon
$k_2$	Rate constant for the pseudo second order adsorption	V	Volume of aqueous solution
$q_{eq}$	The amount of adsorption at equilibrium	W	Mass of adsorbent

### 1. INTRODUCTION

Wastewater containing heavy metals is increasingly released into the environment directly or indirectly, particularly in developing countries [1]. Apart from other contaminants found in wastewater, heavy metals are known to be toxic, non-biodegradable and have long half life [2]. The existence of heavy metals in contaminated water has been a matter of interest to the general public because of their toxic effect on the discharging environment [3]. However, elimination of these heavy metals has become a point of concern in

evaluating the percentage of the heavy metals discharged into the receiving bodies of water, especially in places where recycling of the water is mainly practiced, the elimination of these heavy metals from wastewater is a significant environmental debate in recent time [4]. Treatment of wastewater containing heavy metals can be accomplished through conventional treatment techniques such as ion exchange, floatation, chemical precipitation, membrane filtration, electrochemical process, adsorption and biological systems [5-10]. These techniques were found to have some drawbacks, which include incomplete metal removal, production of excess toxic sludge which requires special disposal method, high initial and

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operation cost, and demand for high energy [11]. Lately, several techniques have been explored for the development of inexpensive and more efficient technologies; this is to reduce the quantity of generated wastewater and also to enhance the quality of the treated effluent. Adsorption method has been found to be one of the most suitable options for wastewater treatment. Commercial activated carbon (AC) is the most effectively and widely used adsorbent in the adsorption process. Application of AC in the adsorption process was found to have some setbacks. AC is found to be very expensive; its cost is related to its quality, the finer it is, the more costly it becomes. Recently, the search for inexpensive adsorbents with the ability for metal binding has been strengthened [12]. The adsorbents can be of agricultural waste, industrial byproducts, biomass, biological and organic origin, polymeric materials and zeolites [13]. In this study, sugarcane bagasse activated carbon (SCBA) was produced as an adsorbent through carbonization process. Adsorption experiments were conducted to investigate the possibility of using SCBA as an alternative adsorbent for removal of lead ions from aqueous solution. Parameters such as initial pH of the solution and SCBA dosage were investigated to find their effects on the adsorption of  $Pb^{2+}$ .

## 2. MATERIALS AND METHODS

### 2. 1. Preparations of Adsorbent Material

Sugarcane bagasse was collected from a local outlet (Pasar Malam) market at Seri Iskandar, Tronoh, Malaysia. The bagasse was manually cut to the average size of 3 cm and thoroughly washed with tap water to remove ligneous and trapped impurities. Bagasse was further washed with deionized water to achieve further purity and for decontamination purpose. The purified bagasse was then dried in an oven at 105 °C for a period of 24 hours until constant weight was achieved. 10 g of the dried bagasse was weighed and placed in a tube furnace; the furnace was then heated to a temperature of 900 °C, at a heating rate of 10 °C/min.

The furnace temperature was maintained at 900 °C for 3 hours. Nitrogen gas at a flow rate of 100 mL/min was used as inert gas to deplete oxygen within the system. The resulting material (SCBA) was pulverized and immersed in a weak acid (0.5%  $H_2SO_4$ , pH 4.25) at room temperature for 24 hours; this was to ensure the adsorbent is kept within the acidic state. The material was filtered from the solution, washed and dried in an oven at 70 °C. SCBA was stored in an airtight container before use.

**2. 2. Preparation of Adsorbate Solution** A stock solution of lead chloride ( $Pb^{2+}$ ) was prepared by dissolving its calculated weight (salt) in 1 L of distilled

water to obtain a concentration of 1000 mg/L. Further working concentrations were obtained by diluting the stock solution to the required strength. The used chemicals were of analytical grade and were obtained from Merck (Germany).

**2. 3 Experimentations** Series of 250 mL Erlenmeyer flasks containing 100 mL of synthetic  $Pb^{2+}$  solution at predetermined concentrations, and 0.5 g SCBA were used in the adsorption experiments. An orbital shaker (Protech Model 722) was used to agitate the flask at a speed of 150 rpm at room temperature ( $26 \pm 1$  °C), for a given period of time. At the end of scheduled agitation times, the flasks were removed from the orbital shaker. Whatman glass microfiber filters paper (GF/C) was used to separate the mixtures. Atomic absorption spectrometer, AAS (Model AA 6800 Shimadzu) was used to analyze the filtrate for residual  $Pb^{2+}$  concentration in mg/L.

The effect of pH on the adsorption of  $Pb^{2+}$  was studied by varying the initial pH of the solution in the range of 1 to 8. The pH of the solution was adjusted using either 2.0 N HCl or 1.5 N NaOH. Other parameters such as initial  $Pb^{2+}$  concentration, contact time, adsorbent dosage and agitation speed were kept constant at 10 mg/L, 2 h, 0.5 g and 150 rpm, respectively. The pH of the solution was measured using a pH meter (Model EW 53013, Hach Sension 1).

Adsorption experiments were also conducted to study the effects of initial  $Pb^{2+}$  concentration and contact time. 100 mL of synthetic  $Pb^{2+}$  solution with concentrations of 10, 50, 75 and 100 mg/L containing 0.5 g SCBA were agitated at 150 rpm. Prior to the addition of SCBA, samples were initially adjusted to the optimum pH. At predetermined time intervals (5, 10, 15, 20, 30, 60, 90, 120, 150, 180, 210, 240, 270 and 300 minutes) samples were removed from the shaker, filtered and analyzed for residual  $Pb^{2+}$  concentration. Equations (1) and (2) were used to compute metal adsorption efficiency and adsorption capacity of SCBA, respectively.

$$R = \frac{(c_i - c_e)}{c_i} \times 100\% \quad (1)$$

where,  $R$  is the  $Pb^{2+}$  removal efficiency (%),  $C_i$  is the initial  $Pb^{2+}$  concentration and  $C_e$  is the residual  $Pb^{2+}$  concentrations (mg/L) at equilibrium.

$$Q_c = \frac{(c_i - c_e)V}{W} \quad (2)$$

where,  $Q_c$  is the adsorbent adsorption capacity (mg/g),  $C_i$  is the initial  $Pb^{2+}$  concentration and  $C_e$  is the residual  $Pb^{2+}$  concentration (mg/L) at equilibrium,  $V$  is the volume of aqueous  $Pb^{2+}$  solution (L) and  $W$  is the mass of SCBA (g).

## 2. 4. Characterization of the Produced Adsorbent (SCBA)

The surface area of SCBA was measured using Micromeritics ASAP 2020 V3.04 H. Weight of the sample before and after carbonization was measured using digital analytical balance Mettler Toledo (ME204) and the carbon yield was calculated according to Equation (3).

$$\text{yield}(\%) = \frac{w_m}{w_b} \times 100 \quad (3)$$

where,  $w_m$  is the dry weight of the SCBA (g), and  $w_b$  is the dry weight of the bagasse (g).

**2. 4. Adsorption Kinetics Studies** Kinetics of  $\text{Pb}^{2+}$  adsorption using SCBA was also studied in the present work. Lagergren first and second order models were employed. The pseudo-first-order kinetic model is expressed generally as Equation (4) [14, 15]:

$$\log(q_{eq} - q_t) = \log q_{eq} - \frac{k_1}{2.303} t \quad (4)$$

where  $q_{eq}$  is the quantity of adsorbate adsorbed at equilibrium (mg/g),  $q_t$  is the amount adsorbate adsorbed at a given time  $t$  and  $k_1$  is the first order constant rate of adsorption ( $\text{min}^{-1}$ ). The plot of  $\log(q_{eq}-q_t)$  against time should be linear with  $k_1/2.303$  and  $\log q_{eq}$  representing the slope and intercept of the line.

Pseudo-second-order kinetic model was also used to analyze the data from the experiments and the mechanism that best fits the data was determined. Linearized form of pseudo-second-order kinetic model is shown in Equation (5). The slope and the intercept of the equation were determined by plotting  $t/q_t$  against time.

$$\frac{t}{q_t} = \frac{1}{k_2 q_{eq}^2} + \frac{1}{q_{eq}} \quad (5)$$

where  $k_2$  is the second order adsorption rate constant (g/mg.min) and  $q_{eq}$  is the adsorbed quantity of pollutant at equilibrium (mg/g). The second order plots of  $t/q_t$  against time will yield a straight line.

**2. 5. Adsorption Isotherm Studies** Freundlich isotherm model is a commonly used model for describing adsorption equilibrium based on the empirical equation. Freundlich isotherm model has the ability to unfold adsorption related to both organic and inorganic composites on a wider range of adsorbents inclusive of biosorbent. Freundlich isotherm model can be written in the following linearized form:

$$\log A_q = \log K_F + \frac{1}{n} \log C_e \quad (6)$$

where  $\log A_q$  is the quantity of the adsorbed pollutant per gram of the adsorbent (mg/g),  $C_e$  is the equilibrium concentration (mg/L),  $K_F$  and  $n$  are Freundlich

constants. The plot of  $\log A_q$  against  $\log C_e$  gives  $1/n$  as slope and  $\log K_F$  as its intercept.

Langmuir model is another frequently used model which describes heavy metals sorption unto biosorbent. Langmuir model operates based on three distinct assumptions viz; that all surface sites are the same and can occupy one adsorbed atom, it is limited to monolayer coverage adsorption and capability of a molecule adsorbed at a given site is not dependent on the occupancy of its adjoining sites. Equation (7) below describes Langmuir isotherm model in its linear form.

$$\frac{C_e}{A_q} = \left( \frac{1}{K_L} \right) \left( \frac{1}{b} \right) + (C_e) \left( \frac{1}{b} \right) \quad (7)$$

where  $A_q$  is the quantity of pollutant adsorbed per gram of adsorbent (mg/g),  $K_L$  is the Langmuir constant for equilibrium,  $b$  is the adsorbate quantity that is required for a monolayer formation and  $C_e$  is the concentration of residual metal at equilibrium (mg/L). Thus, the plot of  $C_e/A_q$  against  $C_e$  will be a straight line with a slope of  $1/b$  and intercept of  $1/K_L$ .

## 3. RESULTS AND DISCUSSIONS

### 3. 1. Characterization of SCBA Adsorbent

Surface physical parameters obtained from Micromeritics adsorption isotherm for SCBA are shown in Table 1. From Table 1, it can be seen that the BET surface area, Langmuir surface area, total pore volume and BJH average pore diameter of SCBA were found to be 599.0  $\text{m}^2/\text{g}$ , 866.4  $\text{m}^2/\text{g}$ , 0.33  $\text{cm}^3/\text{g}$  and 164.4 Å, respectively. While that of RSCB were found to be 12.3  $\text{m}^2/\text{g}$ , 18.9  $\text{m}^2/\text{g}$ , 0.04  $\text{cm}^3/\text{g}$  and 20.5 Å, for BET surface area, Langmuir surface area, total pore volume and BJH average pore diameter, respectively.

Pore sizes as classified in accordance with International Union of Pure and Applied Chemistry (IUPAC), are clustered into micropores ( $d < 2$  nm), mesopores ( $2 \text{ nm} < d < 50$  nm), and macropores ( $d > 50$  nm) [16]; where  $d$  is the pore diameter of the material. In this study, SCBA falls into the category of mesopore materials. The optimum obtained yield was 23.42%.

**TABLE 1.** Physical properties of SCBA

Properties	Value
BET surface area ( $\text{m}^2/\text{g}$ )	599.02
Langmuir surface area ( $\text{m}^2/\text{g}$ )	866.34
Microspore surface area ( $\text{m}^2/\text{g}$ )	452.26
External surface area ( $\text{m}^2/\text{g}$ )	146.76
Total pore volume ( $\text{cm}^3/\text{g}$ )	0.3383
Microspore volume ( $\text{cm}^3/\text{g}$ )	0.2304
Average pore size (Å)	40.54

**3. 2. Effect of Initial pH** This experiment was conducted to investigate the effect of initial pH on the removal of  $Pb^{2+}$  using SCBA. The initial pH of the solution was varied in the range of 1 to 8. The initial metal concentrations, contact time, adsorbent dosage and agitation speed were fixed at 10 mg/L, 2 h, 5 g/L and 150 rpm, respectively. Figure 1 shows the plot of residual  $Pb^{2+}$  concentration against initial pH of the solution.

From Figure 1, it can be observed that adsorption of  $Pb^{2+}$  using SCBA is less at lower initial pH compared to high adsorption at higher pH level. The less removal of  $Pb^{2+}$  at the low pH value was due to the presence of high concentration of hydrogen ions which compete with the metal ions for the active sites of the adsorbent. As the pH of the solution increased, the number of positively charged sites on the surface of SCBA decreased with increasing number of negatively charged sites. As such, more  $Pb^{2+}$  ions will be absorbed and the hydrogen ions at the surface of SCBA will be released back into the solution. The release of hydrogen ions from the surface of SCBA slightly lowered the final pH of the solution; this was for all tested samples. It has been reported that lead usually presents as  $Pb^{2+}$  at a pH range of 2 to 6 and at pH above 6.0, it will hydrolyze to  $Pb(OH)_2$  and  $PbOH^+$  [16, 17]. In this study, pH 5.0 was considered to be optimum for the removal of  $Pb^{2+}$  from aqueous solution using SCBA as an adsorbent. The residual  $Pb^{2+}$  concentration at the optimum pH value was found to be 1.48 mg/L. This corresponds to removal efficiency of 85.2%.

### 3. 2 Effect of Initial Metal Concentration and Contact Time

This study was conducted to investigate the effects of initial metal concentration and contact time on the adsorption of  $Pb^{2+}$  using SCBA. Adequate contact time is required between adsorbent and adsorbate to enable the adsorbent to proceed towards equilibrium with the adsorbate [18, 19].

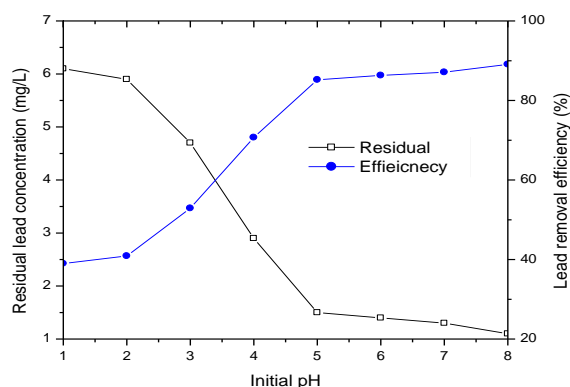


Figure 1. Residual lead concentration against initial pH

The adsorption was investigated using initial  $Pb^{2+}$  concentrations of 10, 50, 75 and 100 mg/L, respectively. The contact time was varied in the range of 5 to 300 min. The adsorbent dosage was fixed at 5 g/L. The pH of the solution was adjusted to the optimum obtained values. Figure 2 shows the plot of residual  $Pb^{2+}$  against contact time for various concentrations of  $Pb^{2+}$ .

From Figure 2, it can be observed that residual  $Pb^{2+}$  concentration decreased with the increase in contact time for all the initial metal concentrations. Rapid removal of  $Pb^{2+}$  was observed during the first 20 minutes of contact time. This was followed by a slow and gradual decrease in the  $Pb^{2+}$  removal as the contact time was increased. The rapid removal at the first 20 minutes of contact time was due to the presence of more vacant active sites at the surface of SCBA. However, as the contact time was increased, the available active sites on the surface of SCBA may have been occupied by the  $Pb^{2+}$  ions. At this stage, the adsorption will be controlled by the rate of transport of the pollutant from the outer to the inner site of the adsorbent. It was also observed that the removal was high at low concentration of  $Pb^{2+}$  in the solution. This was because at low initial metal concentrations, there were few  $Pb^{2+}$  in the solution compared to the available number of active sites on the SCBA surface. On the other hand, at high initial  $Pb^{2+}$  concentration, there were more  $Pb^{2+}$  than the available active sites on the SCBA surface.

Significant  $Pb^{2+}$  removal was not observed as the contact time was increased to beyond 180 minutes. The equilibrium contact time of 180 minutes was considered to be optimum. The residual  $Pb^{2+}$  concentrations at the equilibrium time of 180 minutes were found to be 0.86, 9.92, 24.5 and 42.6 mg/L for the initial lead concentrations of 10, 50, 75 and 100 mg/L, respectively. Their corresponding removal efficiencies at the equilibrium time were found to be 91.4, 80.2, 67.3 and 57.4%, respectively.

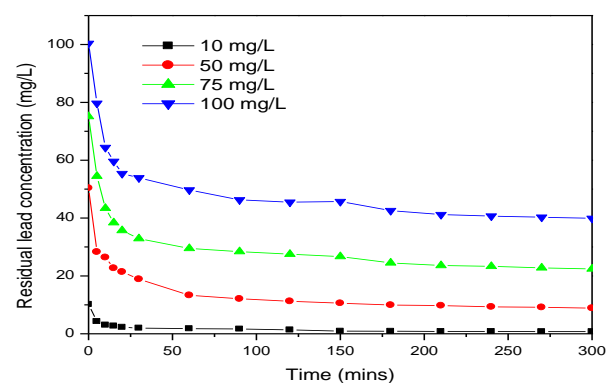


Figure 2. Residual lead concentrations against contact time

**3. 3 Adsorption Kinetics Studies**

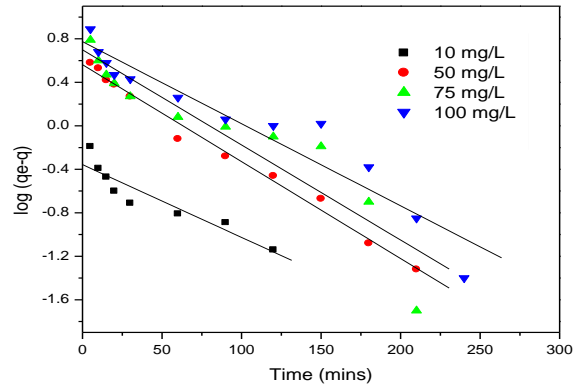
The adsorption kinetic studies for Pb<sup>2+</sup> was investigated at various initial lead concentrations in the range of 10 to 100 mg/L. The contact time was varied in the range of 5 to 300 minutes. The removal percentage of Pb<sup>2+</sup> by SCBA adsorbent was monitored. The data were regressed against pseudo-first-order and pseudo-second-order kinetic models. The linear plots of pseudo-first-order and pseudo-second-order models for Pb<sup>2+</sup> removal using SCBA are plotted in Figures 3 and 4, respectively. The values of *k<sub>1</sub>*, *k<sub>2</sub>*, *q<sub>e,exp</sub>* and *q<sub>e,cal</sub>* are obtained from the slopes and intercepts of the plots and are presented in Table 2.

As can be seen from Table 2, the pseudo-second-order kinetic model presented high values of coefficient of determination (*R*<sup>2</sup>). All the values were close to unity. There was a deviation between the experimental *q<sub>e,exp</sub>* values and the calculated *q<sub>e,cal</sub>* values for the pseudo-first-order kinetic model. This indicates a poor fit of the pseudo-first-order model to the experimental data. On the other hand, the calculated *q<sub>e,cal</sub>* values obtained from pseudo-second-order model agreed effectively with the experimental *q<sub>e,exp</sub>* values. Therefore, it can be concluded that the adsorption of Pb<sup>2+</sup> ions onto SCBA is a pseudo-second-order reaction model. This suggests that the rate limiting step is a chemisorption or chemical adsorption that involves valence forces through electrons exchange between the adsorbent (SCBA) and the adsorbate (Pb<sup>2+</sup>).

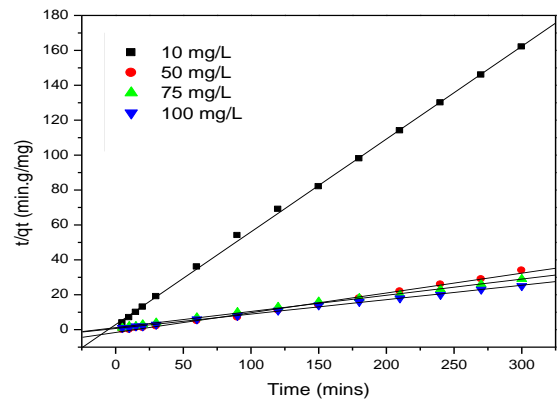
**3. 4. Adsorption Isotherm Studies (Performance of SCBA)**

This study was carried out to establish the relationship between the concentration of Pb<sup>2+</sup> ions and its degree of adsorption onto the surface of the SCBA adsorbent at a fixed temperature. The adsorption isotherm constants obtained from the plots of the linearized Langmuir and Freundlich models are presented in Table 3.

From Table 3, it can be seen that coefficients of determination (*R*<sup>2</sup>) for SCBA from the Langmuir isotherm model was found to be 0.9508. This indicates that Langmuir isotherm model satisfactorily described the adsorption of Pb<sup>2+</sup> onto SCBA.



**Figure 3.** Pseudo-first-order kinetic plots for lead adsorption



**Figure 4.** Pseudo-second-order kinetic plots for lead adsorption

**TABLE 2.** Parameter values of pseudo-first-order and pseudo-second-order models

Pb <sup>2+</sup> (mg/L)	Pseudo-first-order model				Pseudo-second-order model		
	<i>q<sub>e,exp</sub></i>	<i>k<sub>1</sub></i>	<i>q<sub>e,cal</sub></i>	<i>R</i> <sup>2</sup>	<i>k<sub>2</sub></i>	<i>q<sub>e,cal</sub></i>	<i>R</i> <sup>2</sup>
10	<b>1.8</b>	0.27	<b>1.9</b>	<b>0.9942</b>	0.0912	<b>1.8</b>	<b>0.9994</b>
50	<b>8.1</b>	0.4843	<b>8.8</b>	<b>0.9032</b>	0.0107	<b>8.4</b>	<b>0.9942</b>
75	<b>10.3</b>	0.608	<b>10.7</b>	<b>0.9875</b>	0.0097	<b>10.3</b>	<b>0.9999</b>
100	<b>11.9</b>	0.4138	<b>12.3</b>	<b>0.9595</b>	0.0075	<b>11.7</b>	<b>0.9987</b>

*q<sub>e,exp</sub>* = Experimental value, *q<sub>e,cal</sub>* = Calculated value (from model)

**TABLE 3.** Langmuir and Freundlich isotherms for Pb<sup>2+</sup>

Langmuir				Freundlich		
<i>Q<sub>o</sub></i>	<i>b</i>	<i>R</i> <sup>2</sup>	<i>R<sub>L</sub></i>	<i>K<sub>F</sub></i>	<i>R</i> <sup>2</sup>	<i>1/n</i>
23.4	0.29	0.9508	0.26	1.73	0.8629	0.52

This indicates the monolayer coverage of Pb<sup>2+</sup> ions on the homogenous surface of SCBA. The Langmuir equilibrium parameter *R<sub>L</sub>* value for SCBA was found to be 0.26. This is within the range for favorable adsorption (0 < *R<sub>L</sub>* < 1) [21, 15]. The maximum uptake

capacity for SCBA was found to be 23.4 mg/g. This is better than the adsorption capacities reported for some agricultural by-products in the literature. The Freundlich  $R^2$  value was found to be 0.8629; this indicates that the adsorption data was not adequately described by Freundlich isotherm model. The adsorption intensity  $1/n$  value for  $Pb^{2+}$  adsorption was found to be 0.52. This indicates a favorable adsorption process [22].

#### 4. CONCLUSIONS

The present study shows that activated carbon produced from the carbonization of sugarcane bagasse can be used as an environmentally friendly adsorbent for the removal of  $Pb^{2+}$  from synthetic wastewater. It was found that the initial pH of the solution has an effect on the adsorption of the studied heavy metals, especially at lower pH level. The adsorption was favored at a pH level of 5.0. The contact time as a parameter was also found to have an effect on the adsorption of  $Pb^{2+}$ . It was found that as the contact time was increased, the residual concentration of the heavy metal in the solution continued to decrease until equilibrium removal was achieved. The optimum contact time for the equilibrium removal of  $Pb^{2+}$  was found to be 180 min. The kinetics analysis results obtained from pseudo-first-order and pseudo-second-order showed that the experimental data fitted well with pseudo-second-order kinetic model. The obtained experimental uptake capacities were almost the same as the calculated values from pseudo-second-order kinetic models.

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# Equilibrium and Kinetic Studies on Lead (II) Adsorption by Sugarcane Bagasse Derived Activated Carbon

TECHNICAL  
NOTE

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فاضلاب های شهری و صنعتی حاوی مقادیر بالایی از آلاینده ها هستند. فلزات سنگین به عنوان عمده ترین آلاینده ها محسوب می شوند. فلزات سنگین به عنوان مواد سمی، غیر قابل تجزیه با نیمه عمر طولانی شناخته می شوند. فاضلاب های تصفیه نشده حاوی فلزات سنگین است که ورود آنها به محیط می تواند مشکلات جدی به انسان، گیاهان و حیوانات وارد کند. در این مطالعه، کربن فعال از باگاس نیشکر تهیه شد و موثریش در جذب یونهای سرب از فاضلاب مورد بررسی قرار گرفت. آزمایش های جذب هم دمایی به منظور بررسی اثر pH و غلظت اولیه سرب روی فرایند جذب انجام شد. آزمون جذب هم دمایی نشان داد که میزان جذب سرب توسط کربن فعال باگاس نیشکر وابسته به غلظت اولیه سرب و pH بود. pH بهینه برای جذب سرب 5 بود. حذف سرب با افزایش غلظت اولیه فلز کاهش یافت. جذب یونهای سرب توسط کربن فعال باگاس نیشکر یک مدل واکنش شبه درجه دو است. مرحله محدود کردن روند جذب یک واکنش جذب شیمیایی است که شامل نیروهای والانس می شود که از تبادل الکترون بین کربن فعال باگاس نیشکر و یونهای سرب ایجاد می شود.

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