Research Article

Adsorption Batch Studies on the Removal of Pb(II) Using Maize Tassel Based Activated Carbon

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The demand for clean water is on the increase as rapid industrialization is still contributing to pollution. Nowadays, as water is the basic need for mankind, efforts have gathered momentum to decontaminate it in order to address the acute shortage of clean and pure water. Maize tassel was used as the precursor for making activated carbon for the adsorption of Pb(II) ions. The product obtained was characterized and utilized for the removal of Pb(II) from aqueous solutions over a wide range of initial metal ion concentration (10–50 mg/L), contact time (5–300 min), adsorbent dose (0.1–2.5 g), and pH (2–12). The optimum set of conditions for biosorption of Pb(II) ion were found to be initial concentration 10 mg/L, dosage 1.2 g, and pH 5.4. The adsorption data conformed to both the Langmuir and the Freundlich isotherms but fitted best into the Langmuir model. The R^2 for Langmuir equation was 0.9997 and that for Freundlich was 0.9515. The Langmuir monolayer adsorption capacity of the activated carbon was calculated to be 37.31 mg/g. The results indicate that activated carbon might be used to effectively adsorb Pb(II) ions from wastewater treatment plants.

1. Introduction

Nowadays water pollution by heavy metals is fast growing due to natural processes and increasing human activities which include mining, agriculture, and manufacturing industries. These heavy metals are nondegradable and cannot be detoxified biologically [1]. Lead (Pb) is among those contaminants that must be removed from water, due to its high toxicity and tendency to accumulate in tissues of living organisms [2–4]. Pb can occur in different forms such as organic and inorganic forms. Pb is the most significant toxin of the trace metal ions, and human exposure to the inorganic forms is through ingestion of food, water, and inhalation. The aforementioned form affects mainly the peripheral nervous system and haematopoietic, renal, gastrointestinal, cardiovascular, and reproductive systems. Organic Pb mainly affects the central nervous system. According to World Health Organization [5], the permissible level for Pb in drinking water is 0.05 mg/L. Therefore, it is necessary to remove Pb from wastewaters before discharging into the natural water bodies.

One of the ways to address this problem of water shortage is to treat the polluted water through removal of the contaminants. To date, the removal of metals from water has been previously achieved by various methods such as ion exchange, precipitation, oxidation, reduction, and membrane filtration [6, 7]. Compared with these techniques, adsorption technology, using some cheap, easily available agricultural plants, algal biomass, and cyanobacteria, has been reported [8–10]. However, the use of raw sorbents in adsorption has its problems since most plants contain a green pigment known as chlorophyll (sparingly soluble in water), and some organic matter may be leached out, consequently affecting the taste and colour of the treated waters [11, 12]. In spite of the versatility of commercial activated carbon as an adsorbent in wastewater treatment due to its high surface area, microporous characteristics, and high adsorption capacity, it remains costly in developing countries [13–15]. Hence, the derivation of activated carbon from some natural plant based materials might reduce the highlighted problems. In the recent past, activated carbon sorbents derived from locally available materials such as *Ceiba pentandra* hulls [16], *Euphorbia rigida* [17], hazelnut husks [18], wheat bran [19], apple waste [20], coconut shell [21], and *Terminalia arjuna* nuts [22] have received increasing attention for the removal and recovery of pollutants in aqueous media.

Maize is a versatile cereal crop that is grown widely throughout the world in a range of agroecological environments. Maize is a fast growing, vigorous and tall (2-3 m) cereal crop having broad (5–10 cm), and long (50–100 cm) leaves. Maize tassel, an inexhaustible, nonedible, and renewable polymeric material resource, is the male part of the maize plant which is discarded as waste. As a way to address issues related to sustainability, the lignocellulosic material possesses several desirable characteristics to use an adsorbent (a low cost material, mesoporous) and hence can be used as a good precursor of activated carbon [23].

The present study aims at assessing the adsorption potential of activated carbon prepared from maize tassel for the removal of Pb(II) ions from aqueous solutions. The interactive effects of process parameters such as adsorbent dosage, agitation time, solution pH, and initial concentration on the adsorption capacity of activated carbon towards Pb(II) are demonstrated.

2. Materials and Methods

2.1. Preparation of Biomass. The maize tassel was collected from Morris farm in Northlea, Gweru, Zimbabwe. This material is a zero-value agricultural waste product. Maize tassel was plucked off the woody parts of the maize plant, thoroughly washed with water, and sun dried for 5 days. The dried biomass was milled and then fractionated using 100–300 μm analytical sieves and washed twice with 0.01 M HCl in order to remove any metals that might be on the biomass.

2.2. Preparation of Activated Carbon. The activate carbon was prepared according to the reported procedures [24, 25]. Briefly, the fractionated maize tassel powder (200 g) was weighed in a clean dry beaker of capacity 1 L containing (200 mL, 97% H_2SO_4 for 24 h) followed by refluxing in a fume hood for 4 h. After cooling, the reaction mixture was filtered, and the filtrate was washed repeatedly with ultrapure water and soaked in 1% $NaHCO_3$ solution to neutralise any remaining acid. The sample was then washed with distilled water until the pH of the activated carbon was between 6 and 7, dried overnight in an oven at 120°C, and kept in a glass bottle until use.

2.3. Characterization of Prepared Activated Carbon. The bulk density was determined by transferring activated carbon to a 10 mL measuring cylinder of about 1.0 cm diameter. Sufficient quantity of carbon powder was added to occupy a volume of 10 mL under the condition, and it was subsequently weighed. The bulk density was expressed as grams per litre (the weight of the activated carbon filling a graduated cylinder on gentle tapping, divided by the volume of the cylinder 10 mL). The simple specific surface area, loss of mass on ignition, pH, and moisture content were also determined [26, 27]. The FTIR spectra of activatedcarbon were obtained with KBr using Shimadzu model *R*-prestige Fourier Transform Infrared Spectrometer. FTIR spectra were recorded in the region of 4000–400 cm^{-1}. X-ray powder diffraction (XRD) analysis of the activated carbon was carried out on a Bruker D8 Advance X-ray diffractometer using nickel filtered Cu K$_\alpha$

radiation (40 kV, 40 mA). The pH of solutions was measured with a proLab 200 (SCHOTT instruments).

2.4. Adsorbate. A stock solution of 1000 mg/L Pb(II) was prepared by dissolving $Pb(NO_3)_2$ (Merck, South Africa) in ultrapure water (resistivity >18 MΩ cm^{-1}) and acidified with 5 mL of concentrated HNO_3 to prevent hydrolysis. The progressive dilution procedure of the stock solution was employed in the preparation of working solutions. The pH of the working solutions was adjusted to the required values with 0.1 M NaOH or 0.1 M HCl. All the chemicals used were of analytical reagent grade.

2.5. Batch Adsorption Studies. A weighed amount of activated carbon was introduced into stoppered reagent bottles containing various concentrations with 100 mL aqueous solutions of Pb(II) ions. The suspensions were shaken at room temperature (25 ± 1°C) using a mechanical shaker for a prescribed time at 160 rpm. Scheme 1 shows the schematic representation of the process involved in the derivation of activated carbon from raw maize tassel up to adsorption. The solutions were filtered through Whatman 42 filter paper, and the residual concentration of metal ion was determined by AAS method at 217 nm. The effects of concentration (10–50 mg/L), contact time (5–300 min), solution pH (2–12), and adsorption dose (0.1–2.5 g) were studied. Blank solutions were treated similarly (without adsorbent), and the recorded concentration at the end of each operation was taken as the initial one.

The amount of metal adsorbed by activated carbon was calculated from the difference between metal quantity added to the biomass and metal content of the supernatant using
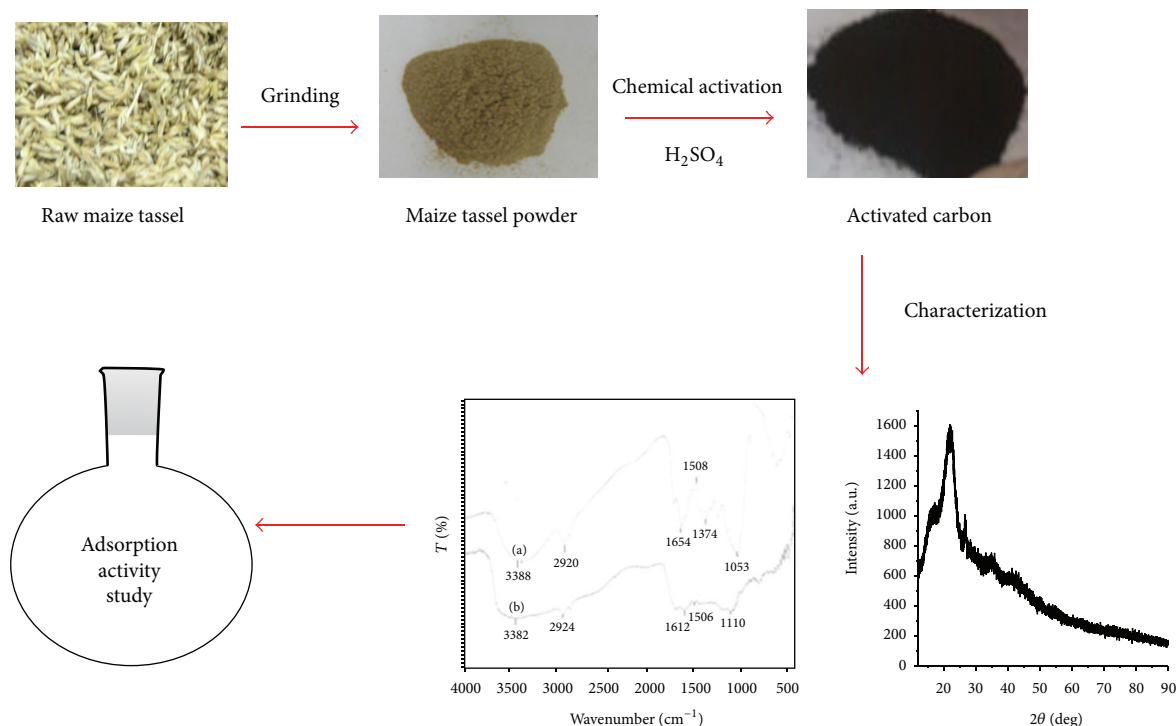
$$q_e = \frac{(C_i - C_e)V}{M}, \tag{1}$$

where q_e is the metal uptake (mg metal adsorbed per g adsorbent), C_i and C_e are the initial and equilibrium metal concentration in solution (mg/L), V is the volume of the solution (mL), and M is the weight of activated carbon (g). The percentage of removed Pb (II) ions (R%) in solution was calculated using

$$R\% = \frac{(C_i - C_e)100}{C_i}. \tag{2}$$

2.6. Desorption Studies. For the desorption studies, contact was made between 1.2 g of activated carbon and a 100 mL Pb(II) solution. After Pb(II) ion sorption, the activated carbon was filtered, washed three times with ultrapure water to remove residual Pb(II) ions on the surface, and kept in contact with the 100 mL HCl solution (0.05–0.3 M desorbent). The mixtures were shaken in a rotary shaker. The filtrates were analyzed to determine the concentration of Pb(II) ions after desorption using AAS.

3. Results and Discussion

3.1. Physicochemical Characteristics of Activated Carbon. The physicochemical characteristics like pH, moisture content,

SCHEME 1: Schematic representation of the process involved in the derivation of activated carbon from raw maize tassel up to adsorption.

TABLE 1: Characteristics of the activated carbon derived from maize tassel.

Parameter	Value
pH	6.9
Moisture (%)	0.3
Bulk density (gmL^{-1})	0.52
Surface area (m^2/g)	250
Particle size range (μm)	100–300
% Loss of mass on ignition	0.7

FIGURE 1: X-ray diffraction of activated carbon derived from maize tassel.

bulk density, surface area, and loss of mass on ignition are shown in Table 1. The bulk density affects the rate of adsorption of metals ions onto activated carbon. In the present study, the bulk density was less than 1.2 indicating that the activated carbon materials are in fine nature and hence enhanced the adsorption of Pb(II) ions from aqueous solution. The moisture content (0.3%) was determined, even though it does not affect the adsorption power, dilutes the adsorbents, and therefore necessitates the use of additional weight of adsorbents to provide the required weight. The surface area of the prepared activated carbon in the present research study was 250 m^2/g and is higher than a low cost agro-based adsorbent such as palm pith carbon (188 m^2/g) [28] but lower than peanut hull carbon (354 m^2/g) [29].

The X-ray diffraction pattern of the activated carbon is shown in Figure 1. The XRD spectrum of activated carbon is a typical diffraction pattern of an amorphous material which gi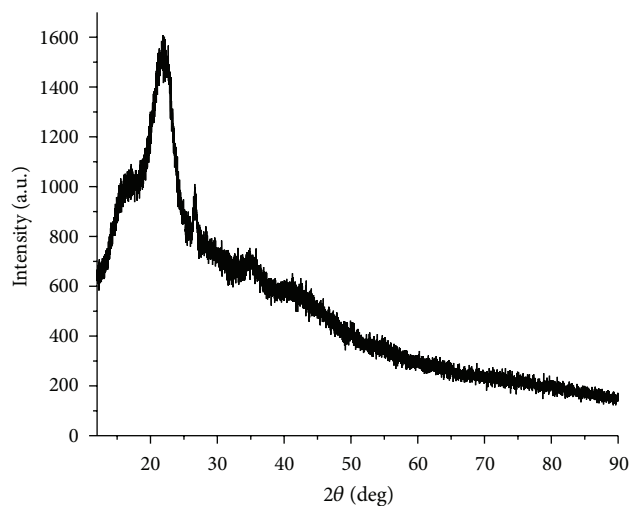ves a continuous function and had two partially, not well defined resolved peaks at about 2θ = angle of 22.6 and 26.4 degrees, respectively.

The FT-IR spectrum was obtained for treated activated carbon before (a) and after Pb(II) ion biosorption process (b) (Figure 2). The results showed us that the activated carbon has different functional groups such as amide, hydroxyl, and carboxyl. Some of these functional groups changed after biosorption process. The comparisons of the FT-IR spectra of
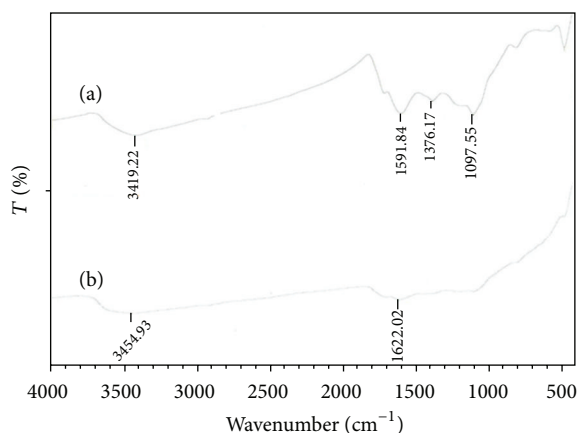
FIGURE 2: FT-IR spectra of treated activated carbon (a) and Pb(II) loaded activated carbon (b).
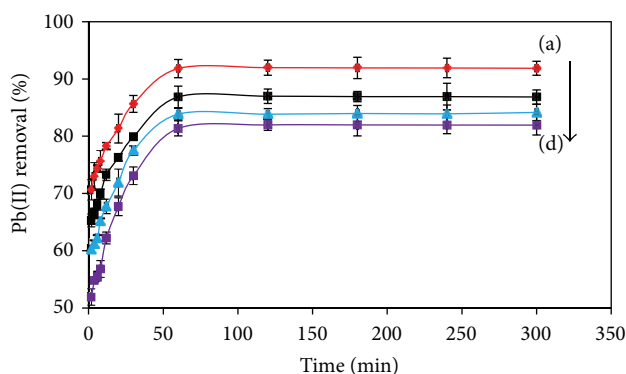

FIGURE 4: Effect of pH on lead adsorption by activated maize tassel (contact time: 60 min; dosage 1.2 g; initial concentration 10 mg/L; error bar = ±S.D.; and n = 3).

FIGURE 3: Effects of agitation time on lead ions removal (concentrations: a to d: 10 mg/L to 50 mg/L; adsorbent dosage: 1.2 g; pH: 5.4; error bar = ±S.D.; and n = 3).

FIGURE 5: Effect of adsorbent dosage on lead adsorption by activated maize tassel (contact time: 60 min; dosage 1.2 g; initial concentration 10 mg/L; error bar = ±S.D.; and n = 3).

activated carbon and after Pb(II) biosorption give the following bands and peaks. The bands at 3419.22 and 3454.93 cm^{-1} are the OH stretching of polymeric compounds. The bands located at 1376.17 and 1591.84 cm^{-1} are possibly due to the functional groups like C=O in carboxylic groups and carboxylate moieties. The additional small peaks at 1090.55 cm^{-1} indicate the S=O. The band at 1622.02 cm^{-1} corresponds to carbonyl stretching vibration of amide considered to be due to the combined effect of double-bond stretching vibration and –NH deformation band for Pb(II) loaded activated carbon. The C=O and S=O functional groups have been reported to show very high coordination with trace metals; hence it disappeared in the Pb(II) loaded [14]. For this reason, the presence of these functional groups may be responsible for good adsorptive behavior of the activated carbon towards the Pb(II) ions.

3.2. Effect of Agitation Time and Initial Concentration on Adsorption. The effect of agitation time is one of the important factors when designing batch sorption systems for economical wastewater treatment plant application. As shown in Figure 3, the relationship between agitation time and Pb(II)
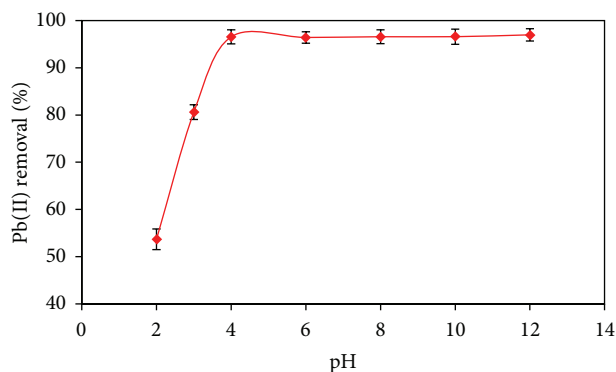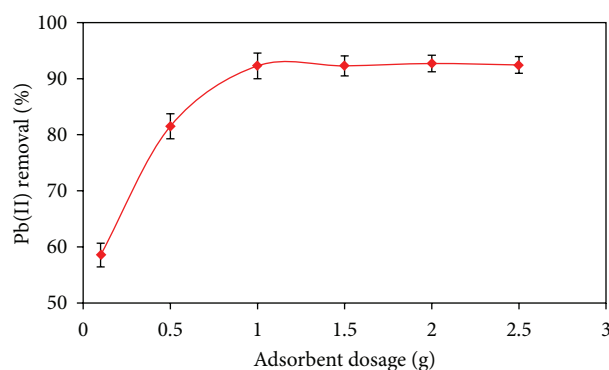
ion sorption onto activated carbon at different initial Pb(II) ion concentrations is displayed.

It was observed that 52, 60, 65, and 70% of Pb(II) ions were removed in the first two minutes for the different concentrations (10, 20, 30, and 50 mg/L, resp.), and the process was rapid up to 60 min attaining 91.8% removal for 10 mg/L. Beyond 60 min, the percentage of biosorption is almost constant indicating the attainment of equilibrium conditions. The presence of adequate external surface area on the activated carbon may have boosted the rate of adsorption in the initial stages which was followed by a slower internal diffusion process, which may be the rate determining step [30]. Therefore, all other experiments were conducted at an agitation time of 60 min.

The effect of initial concentration on the percentage removal is also shown in Figure 3. The removal of Pb(II) ions by activated carbon was found to decrease with increase in initial Pb(II) concentration. The observed behavior can be attributed to the increase in the amount of Pb(II) ions to the unchanging number of available active sites on the activated carbon. Hence, more metal ions were left in solution. Thus, it

can be said that removal of Pb(II) ion is highly concentration dependent.

3.3. Effect of pH on Adsorption.

The effect of initial pH of a solution is a major factor used to determine the adsorption property of an adsorbent from waste water. The possible reasons are its effects on the chemistry of the ions and the activity of functional groups (carboxylate, phosphate, and amino groups) on the cell walls [19, 31]. As revealed in Figure 4, there is a change in the quantity of adsorbed Pb(II) ions on the solid phase of activated carbon by increasing pH of the medium up to a maximum value of pH 5.4. At low pH values, the adsorbent is positively charged since the pH is lower than the isoelectric point or point of zero charge (PZC) [13]. Hence the removal yield of Pb(II) ions is very low due to the electrostatic repulsion forces between positively charged H_3O^+ and Pb^{2+} ions. Thereafter the adsorption percentage decreased in alkaline medium perhaps due to the formation of $Pb(OH)_2$ and soluble hydroxyl complexes such as $PbOH^+$, aqueous $Pb(OH)_2$, and $Pb(OH)^{3-}$, and the activated carbon was deteriorated with the accumulation of Pb(II) ions making true adsorption studies impossible [32–34]. Similar trends were reported for adsorption of Pb(II) ions on activated carbon prepared from coconut shell [35], *Euphorbia rigida* [17], sawdust [36], and mature pods of *M. Oleifera* containing seeds [37]. The results obtained in this study indicated that the activated carbon is more effective in removing Pb(II) ions from an aqueous system with 96.6% removal as compared to raw maize tassel (75%) [38], tobacco stems (94.37%) [30], and physically prepared AC (78%) [39]. Therefore, pH 5.4 was selected to be the optimum pH for all further studies.

3.4. Effects of Dosage on Adsorption.

The effect of biomass dosage on the biosorption of Pb(II) ions was investigated (Figure 5). As shown in Figure 5, the percentage removal increases sharply from 58.5% to 92.2% with increasing dosage from 0.1 to 1.2 g, respectively. Beyond 1.2 g, the percentage was not increased significantly and reached the maximum of 92.3% at dosage of 1.2 g. This phenomenon can be due to the greater availability of active sites or surface area making easier penetration of the Pb(II) ions to the adsorption sites of activated carbon, and increasing beyond dosage of 1.2 g had no much effect after equilibrium is reached. These results are in agreement with other work reported in the literature [2, 34].

3.5. Adsorption Isotherms.

Adsorption isotherms are very useful for analyzing the nature of adsorbate-adsorbent interaction. So, in order to optimize the design of a successful adsorption unit to remove trace metal ions from industrial effluents, it is significant to establish the best correlation for the equilibrium isotherms. In this study, the equilibrium data for the adsorbed Pb(II) ions onto activated carbon was expressed using two of the most popular isotherms, namely, Langmuir and Freundlich isotherms.

The Freundlich isotherm [40] is given by

$$q_e = K_F C_e^{1/n}. \tag{3}$$

TABLE 2: Langmuir and Freundlich constants for Pb(II) adsorption using activated carbon.

Adsorption isotherm	Parameter	Value
Langmuir	q_{max} (mg/g)	37.31
	b (L/mg)	0.062
	R^2	0.9997
Freundlich	K_F	0.077
	n	0.482
	R^2	0.9515

The parameters can be linearized by taking logarithms to determine the parameters K_F and n:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e. \tag{4}$$

A plot of $\ln q_e$ versus $\ln C_e$ gives a straight line, and K_F and n can be calculated from the intercept and slope, respectively. The linear form of the Langmuir isotherm model [41] can be represented by

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \left(\frac{1}{bq_{max}}\right)\frac{1}{C_e}. \tag{5}$$

A plot of $1/q_e$ versus $1/C_e$ was found to be a straight line with $1/q_{max}$ as intercept and $1/bq_{max}$ as slope, and hence q_{max} and b can be calculated. Figures 6(a) and 6(b) show Freundlich adsorption isotherm and the Langmuir adsorption isotherm, respectively. The Freundlich constants n and K_F, Langmuir constants b and q_{max}, and the correlation coefficient R^2 are given in Table 2. The calculated value of Freundlich constant n is within the range ($0.1 < n < 1$), reported in the literature [42] showing that adsorption is favorable. However, the linearized equation did not give a good correlation for the removal of Pb(II) ions onto activated carbon, indicating that Pb(II) adsorption by activated carbon fits better to the Langmuir model than to the Freundlich model.

In addition, a dimensionless constant called separation factor, R_L, can be used to express an essential feature of Langmuir isotherm [43]:

$$R_L = \frac{1}{1 + aC_m}, \tag{6}$$

where C_m is the initial concentration (10 mg/L, in this case) of Pb(II). The value of R_L indicates the type of the isotherm to be either unfavorable when $R_L > 1$, linear if $R_L = 1$, and favorable if $0 < R_L < 1$ or $R_L = 0$. The calculated R_L was 0.62 indicating that the adsorption of the Pb(II) was a favorable process. The comparison of maximum monolayer adsorption capacity of Pb(II) ions onto various activated carbons derived from different precursors is given in Table 3. It should be noted that adsorption capacity varies and depends on the characteristics of an individual adsorbent, the extent of surface modification, the analytical method used for decontamination (batch method, column, reactors, etc.), heat treatment temperatures, and the initial concentration of
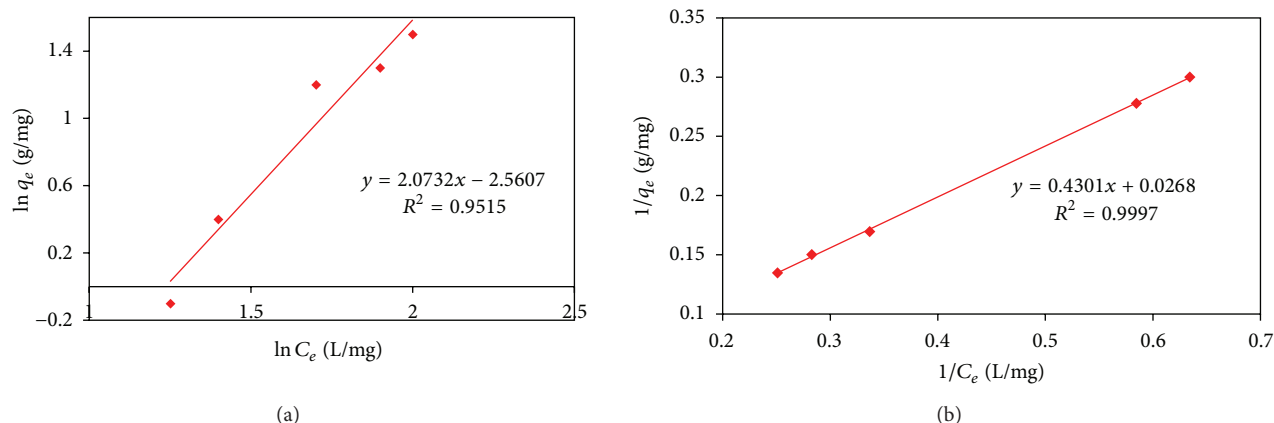
(a)

(b)

FIGURE 6: (a) Freundlich isotherm for Pb(II) ion adsorption using activated carbon (b) Langmuir isotherm for Pb(II) adsorption using activated carbon (10 mg/L, pH = 5.4, and dosage = 1.2 g).

TABLE 3: Performance of activated carbons from various precursors for adsorption of Pb(II) from aqueous solutions.

Precursor	Activation	Conditions: pH; agitation time	Maximum adsorbent (mg/g)	Reference
Hazelnut husks	Chemical activation by $ZnCl_2$	6.7; 60 min	13.05	[18]
Coffee residue	Chemical activation by $ZnCl_2$	5.8; 2 h	63	[23]
Rice husk	Chemical activation by H_3PO_4	—	138.89	[27]
Coconut shell	Chemical activation by H_2SO_4	4.5; 2 h	26.50	[35]
Tamarind wood	Chemical activation by $ZnCl_2$	6.5; 50 min	43.85	[45]
Tamarind wood	Chemical activation by H_2SO_4	6.5; 40 min	134.22	[45]
Cashew nut shell	Chemical activation by H_2SO_4	5.0; 30 min	408.6	[46]
Sugarcane bagasse	Chemical activation by H_2SO_4	5; not given	7.297	[47]
Maize tassel	Chemical activation by H_2SO_4	5.4; 60 min	37.31	This study

TABLE 4: Desorption of Pb(II) ions from activated carbon using HCl.

Initial Pb(II) concentration (mg/L)	% Pb(II) ion removal	% Recovery of Pb(II) ions			
		0.05 M	0.1 M	0.2 M	0.3 M
10	91.8	74.25	80.50	90.60	87.65
20	86.7	70.21	77.54	83.29	80.50
30	83.9	66.25	74.58	79.32	75.65
50	81.3	58.2	65.65	70.52	68.56

the adsorbate used [26, 44]. As can be seen from Table 3, activated carbon from maize tassel precursor in this work has reasonable adsorption capacity (37.31 mg/g) for Pb(II) ions, closer or higher than some agro-based derived activated carbons but lower than other highly lignocellulosic plants such as *Tamarind wood* (134.22 mg/g) and cashew nut shell (138.89 mg/g) just to mention a few.

3.6. Desorption Studies. For wastewater treatment, regeneration of the adsorbent material is of importance in economic development, since it helps to protect the environment and recycling of the adsorbate (Pb(II) ions) and adsorbent (activated carbon). The successful application of this technique also reduces the dependence on thermal activation, incineration, and land disposal, which directly or indirectly increases environmental pollution [1]. Desorption studies help in process design systems by giving information on the mechanism and recovery of adsorbates from industrial wastewater and the adsorbent. In this study, desorption results are presented in Table 4. It can be observed that desorption rate increases with increase in HCl concentration but attained a constant at 0.2 M HCl.

4. Conclusions

Maize tassel, a residue released in a substantial amount in the agricultural sector, was converted into activated carbon. The material under consideration is not only economical but is a waste product. Hence, its use as an adsorbent would on one hand solve the problem of its disposal and on other hand provide an effective adsorbent for the treatment of metal bearing wastewaters. Results from adsorption studies revealed that the percentage removal decreased with increase in the initial concentration of Pb(II) ions suggesting the applicability of the activated carbon at lower concentrations. Adsorption equilibrium for the metal was reached in about 60 min. It was also observed that the adsorption was pH dependent and the maximum Pb removal of 96.6% occurred at pH of 5.4. An increase in the activated carbon dosage leads to an increase in Pb(II) ions removal due to a corresponding increase in the number of active sites.

Acknowledgment

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