

Research Article

Adsorption of Pb(II) Ions from Aqueous Phase using Activated Carbon prepared from Novel Precursor

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Abstract

This research deals with performing a comparative study between prepared and commercial activated carbon to evaluate their performance for removing Pb(II) ions from aqueous solution. The first part of experimental work involves preparing activated carbon from bark of date palm trees (as a novel precursor) using phosphoric acid with concentration of 85% at specific condition of carbonization temperature at 500°C and 45 min as a holding time. The obtained material was submitted to measurements of active surface area and scanning electron microscope. The second part includes the batch adsorption studies to remove Pb(II) ions from aqueous solution by the two different types of activated carbon. The effect of different parameters such as pH of solution, contact time, initial ions concentration, and mass of adsorbent on the removal amount of lead were fully studied. The experimental data shows that the best removal efficiency was obtained at 240 min of adsorbing time with initial metal concentration of 50 ppm, 6 pH solution, and 15 g/L mass of adsorbent, for both prepared and commercial activated carbon. Langmuir and Freundlich models were applied on the achieved data and it was observed that the Freundlich equation was more fitted to our data.

Keywords: Activated carbon; The bark of date palm tree; Lead adsorption; Freundlich isotherm; Langmuir isotherm.

1. Introduction

The pollution of wastewater by heavy metals is presently attracting global attention because of the harmful effects it has on the environment and especially on human health (Saravanan, *et al.*, 2009; Ong, *et al.*, 2007). The high cost of contemporary methods to remove such heavy metals coupled with the fact that some of these methods are usually not environmentally friendly, is motivate the choice of adsorption by activated carbon as cheap technique to remove the metal contaminants from water and soil (Bello, *et al.*, 2008). Among of different contaminants, it considers that lead ions are one of the fundamental wastewater pollutants. Where it could be severely damage the kidney, liver and whole biological system (RSC, 1986). Where it is usually found in industrial effluences such as lead mining, leaded gasoline, smelting and coal combustion, lead base paint, lead-acid batteries ...etc. According to the World Health Organization, the accepted range of Pb(II) in water is less than 0.01 ppm (WHO, 1984).

Adsorption by activated carbon has gained importance over many other conventional process for removal of heavy metals from industrial wastewater

such as precipitation, coagulation, ion exchange ...etc. (Tan, *et al.*, 2008). Activated carbon is probably the most widely used adsorbent material in industrial and environmental applications regarding to its highly micro-porous morphology, high internal surface area, and porosity (Chang, *et al.*, 2000). Also, the low cost of production of activated carbon from any material rich in carbon content or any agricultural waste make it commercially available (Isah, and Yusuf, 2012).

The chemical activation technique used in preparing activated carbon is more favorable over the physical ones regarding to several advantages such as: carbonization and activation is performed in single step, pyrolyzed in lower temperatures, better porous structure are achieved, and chemicals added for activation can be easily be recovered (Chang, *et al.*, 2000).

Numerous researchers have extensively studied the preparation of activated carbon from various types of precursors and evaluate the performance of such developed materials by adsorbing of different heavy researches have been accomplished on converting low cost raw materials and agricultural waste to activated carbon and evaluate their performance by removing different heavy metal ions. Agbele, (2015) prepared activated carbon from plantain peel by three different

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chemical activation agents and use the carbon prepared to remove copper, cadmium and chromium from polluted water. In addition, several raw materials were suggested to produce activated carbon namely oil palm and coconut shells (Rahman, *et al.*, 2014), stem and leavers of water hyacinth plant (El-Wakil, *et al.*, 2014), Calotropis Procera (Dead Sea fruit) (Ramalingam, *et al.*, 2013), rice husk and waste plastic (Thajeel, *et al.*, 2013) and many others.

The present study is mainly focused on preparing the micro-porous activated carbon from the bark of date palm tree (as novel material) by chemical activation method using phosphoric acid (H_3PO_4) as a chemical agent. Also, evaluated the removal efficiency of Pb(II) ions from the aqueous phase using the prepared activated carbon and comparing its performance with the commercial one.

2. Experimental Work

2.1 Precursor selection and pretreated

The selected raw material to synthesis the activated carbon in this investigation is the bark of date palm trees which is considered as agricultural waste during the clipping season of date palms trunk. Such precursor is available locally in form of plentiful. It is preferred to select the waste of date palm bark from the last year to be sure that is completely dry and easier for chopping to small pieces. Previously, the sample was carefully washed with tap water to get rid of the surface dirt and other undesired materials and then dried at sunlight for two to three days. After that, the cleaned and dried raw material is crashed and grinded using Tianjin laboratory grinding machine (Taisite Instrument Co. Ltd) and sieved to get a uniform particle size of almost 1.68 - 2mm (number 12 - 10 mesh size).

The sawdust of the dead palm bark which was obtained at the desired size is then washed with distilled water for several time and dried at $100 \pm 50C$ in laboratory furnace model (M 110, Thermo. Scientific/Germany) over one night to be ready for carbonization process. Figure (1) shows the dry and grinded sample.



Figure 1 The dried and grinded sample of dead palm bark

2.2 Preparation of activated carbon

The previously prepared precursor was soaked in excess amount of (85% w/w) concentration H_3PO_4

supplied by (Romil Pure Chemistry - UK) at room temperature for 24 hours. A genially stirring between to time to achieve good contact between the chemical and the precursor surfaces. Later on, the raw material was washed with de-ionized water for several times to removal all remaining acid and to be sure that the washing water pH equals 6. The wet sample is dried in electrical oven at $110 \pm 2 \text{ }^\circ C$ for 48 hr to be ready for pyrolysis step.

A vacuum tubular furnace, shown in figure (2), model (GSL-1700x/MTI - USA) owned by (Corporation of Research and Industrial Development / Ministry of Industry & Minerals -Iraq) was used for carbonization the sample, where 20 g of raw material is placed in porcelain crucible inside the quartz tube of the furnace. The sample was heated gradually in absence of air at heating rate of $10 \text{ }^\circ C / \text{min}$ to a temperature of $500 \text{ }^\circ C$ and under continuous N_2 flow of 1000 ml/min and then maintained at this temperature for 45min (El-Wakil, *et al.*, 2014; Huang, *et al.*, 2007). Let the sample be cooled to room temperature, subsequently the achieved activated carbon was washed perfectly several times with de-ionized water till the pH of the solid become stable at about (pH=6). Then the sample was dried at $110 \text{ }^\circ C$ for 24 hr by electrical oven. Figure (3) shows the final activated carbon prepared.



Figure 2 The photograph image for vacuum tubular furnace



Figure 3 The sample of dead palm bark after carbonization process

2.3 Commercial Activated Carbon

The commercial activated carbon (CAC) was supplied by (SIGMA-ALDRICH, St. Louis, MO - USA). It firstly was washed with distilled water for removing impurities and after that it was dried at 110 °C for one hour and then it was placed in a desiccator for cooling.

2.4 Adsorption Experiments

The batch adsorption method was selected to conduct the experiments of removing lead ions from aqueous solutions which were previously prepared. The stock solution was diluted as required to obtain different concentrations of Pb(II) solutions from 50 to 150 ppm. Each experimental run was performed by adding certain weight of the synthesis and commercial activated carbon in successive way to 150 ml solution placed in 250 ml flasks. All runs were conducted at constant temperature of 30 °C and specify mixing speed of 150 rpm using hot plate magnetic stirrer type (LMS-HTS-1003, Japan). All samples were collected at regular time intervals from 10 to 300 min. using macro-pipette. Then, the samples were filtered prior to analysis in order to minimize interference of the carbon fines with the analysis.

The concentration of Pb(II) ion solution was determined using the (UV-Visible) spectroscopy measuring device and, the percentage of metal removal (R%) was calculated using equation (1), illustrated below.

$$R\% = \frac{C_i - C_t}{C_i} \quad (1)$$

where:

R% : The percentage metal removal.

C_i : The initial concentration of metal ion (mg/L).

C_t : The concentration of metal ion after adsorption at any time t (mg/L).

The aforementioned procedure was repeated with each set of experiments by varying different parameters such as pH (2.0 - 10.0), initial Pb(II) concentration (50 - 150 mg/L), and adsorbent dose (0.75 - 3.75 g) which is equivalent to (5-25 g/L). It is worth to mention that the pH values of the solution were adjusted by adding hydrochloric acid, 0.1M supplied by (Romil Pure Chemistry - UK) and potassium hydroxide, 0.1M supplied by (Romil Pure Chemistry - UK) solutions where every its necessary, and measured by laboratory pH meter (Microprocessor pH meter, range 0.00 to 14.0, ± 0.01 accuracy - Hunna instruments, pH 211, Romani).

2.5 Analysis of Activated Carbon

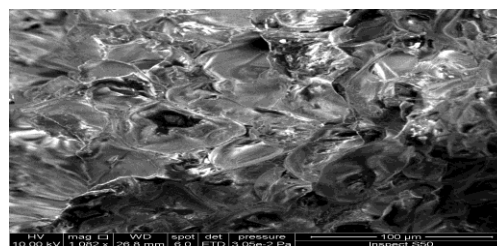
2.5.1 Surface Area Analyzer

The surface area of prepared activated carbon was determined using Surface Area Analyzer instrument, (model SA-9600 Qsurf series -thermo electron corporation - BET test method) . The measurement

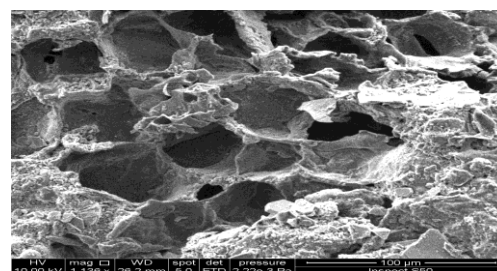
was carried out in laboratories of Ministry of Oil/ Petroleum Research and Development Center - Iraq. The surface area of the prepared activated carbon was 509.2948 m²/g.

2.5.2 Scanning Electron Microscope (SEM)

The prepared activated carbon was examined by Scanning Electron Microscope (SEM) (Inspect S50 model, FEI / Netherlands). Where, SEM micrographs of the raw material, and final activated carbon by H₃PO₄ were presented in Figure (4). These images show pores and cavities for activated carbon samples. The pores observed in SEM images are having diameter in micrometer (µm) range. The increase in porosity and cavities can lead to enhance the ability of these materials to adsorb lead and other adsorbents with high efficiency .



(a)



(b)

Figure 4 The SEM images of dead palm bark sample at 1136 times magnification of enlargement: (a) the original sample before activation, and (b) chemically activated carbon by H₃PO₄ and carbonized.

3. Results and Discussion

3.1 Effect of initial concentration of Pb(II) ions

The initial concentration of metal ions in the solution is an important parameter since the metal ions concentration change over a broad range in industrial effluents.

The batch adsorption experiments were carried out with different initial concentrations(C_i) (50, 75, 100, 125, 150).The variation of percentage removal of Pb(II) ions with different initial concentration for both prepared activated carbon (PAC) and commercial activated carbon (CAC) is properly clarified in Figure (5). This figure shows a comparative performance between prepared and commercial activated carbon at

equilibrium state and clarify the optimum Pb(II) ions initial concentration used at confined experimental conditions. It is also evidently observed that the percentage removal of the Pb(II) ions is sufficiently high at low concentration and gradually decreases as the concentration increases (Venkateswarlu, *et al.*, 2007). This could be attributed to the fact that after the formation of mono-ionic layer at low concentration over the activated carbon surface, further formation of the layer is highly hindered at higher concentration and is due to interaction between Pb(II) ions on the surface and in the solution (Ananthakumar, *et al.*, 2009).

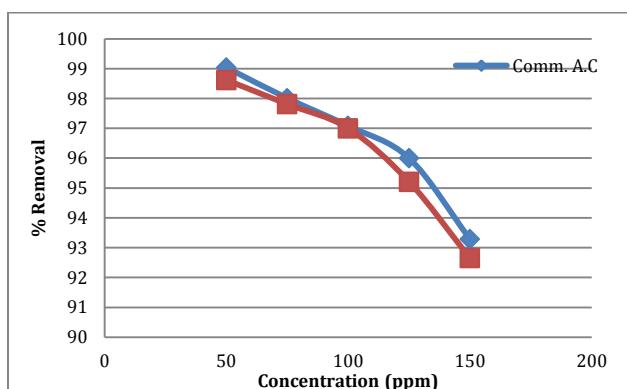


Figure 5: Variation of removal percentage of Pb(II) ions versus initial concentration for both CAC and PAC at equilibrium condition

3.2 Effect of Adsorbent Dosage

The variation of removal percentage of Pb(II) ions versus adsorbent dosage for both CAC and PAC at different adsorbent doses ranging from (5 – 25) g/L which equal to (0.75 – 3.75)g were collaborated and other working parameters will consider to be constant (pH at 6 and initial ions concentration at 100ppm) is suitably illustrated in Figure (6). It is clearly noted from this graph that the maximum adsorption removal achieved by using 15g/L of prepared activated carbon was 96% compared with commercial activated carbon which recorded 97%. On the other hand, the removal percentage of Pb(II) ions are increased as the amount of adsorbent is increased gradually from 5 g to 15 g/L and it is found that any further addition over the above mentioned weight (15g/L) will not make any enhancement in the adsorption process, on the contrary it is recorded that the percentage removal of Pb(II) ions decreases almost 3.5% for CAC and 3% for PAC with increasing dose. It is due to the fact that at specific initial concentration, the active free sites on solid surface which available to capture metal ions at low solid weight are insufficient. So, the maximum removal will be low at low solid loading and increased with increasing adsorbent dosage. While almost negligible decrease of removal efficiency over the specific adsorbent weight i.e beyond optimum weight. These results were confirmed by previous researchers

(Tahiruddin, and Abd Rahman, 2013; Mengistie, *et al.*, 2008). The maximum adsorption removal achieved by using 15g/L of prepared activated carbon was 96% compared with commercial activated carbon which recorded 97%.

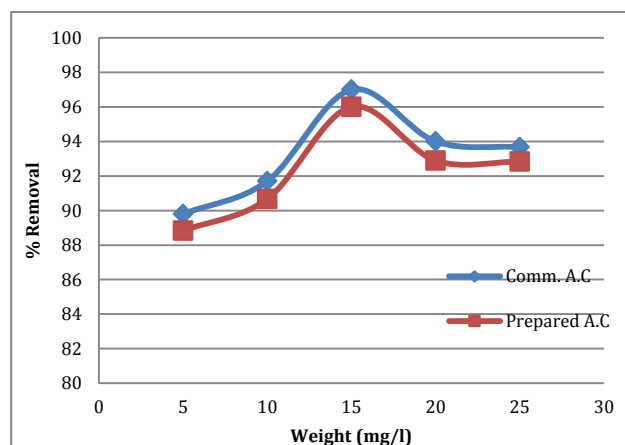


Figure 6: Variation of removal percentage of Pb(II) ions versus adsorbent dosage for both CAC and PAC at equilibrium condition

3.3 Effect of the Solution pH

The pH of solution is one of most significant variables which directly influenced on the amount of ions removal from aqueous phase (Tang, *et al.*, 2009; Karthikeyan, *et al.*, 2005; Zhang, *et al.*, 2010). In this study, it has been focused on pH values from 2 to 10 to evaluate the removal percentage of metal ions under wide range of this variable. It is evident from figure(7) that the lowest adsorption values of Pb(II) ions are achieved at pH equals 2 for both commercial and prepared activated carbon and increase with increasing the pH of solution till reached maximum adsorption at pH equals 6. While, the removal efficiency of activated carbon is progressively decreases in basic media where it achieved minimum value when the solution pH equals 10. This could be explained as, in acidic media i.e. when the pH of solution less than 4, the elevated hydrogen ions (H^+) will rival the Pb(II) ions from reaching the free available sites on the adsorbent surface and prevent the Pb(II) ions from bounding with activated carbon surface due to the repulsive forces. Besides that, it is also noted when the pH values reaches 6, the adsorption process enhanced and removal percentage get maximum quantities regarding the lower hydrogen ions (H^+) exist, and on the sense the Pb(II) ions will get better chance to occupy the free sites of the active surface. Later on, when the pH precede toward basic region and elevated above 6, the recorded values of removal ions decrease significantly specially at 8 and 10 pH to record values in the med of seventy percent for both commercial and prepared activated carbon. This is attributed to Pb(II) ions precipitated regarding to the formation of lead hydroxide anions due to the

dissolution of $Pb(OH)_2$ as a result, the adsorption rate decreases. This contribution is agreed by numerous researchers (El-Wakil, et al.,2014; Gottipati, 2012).

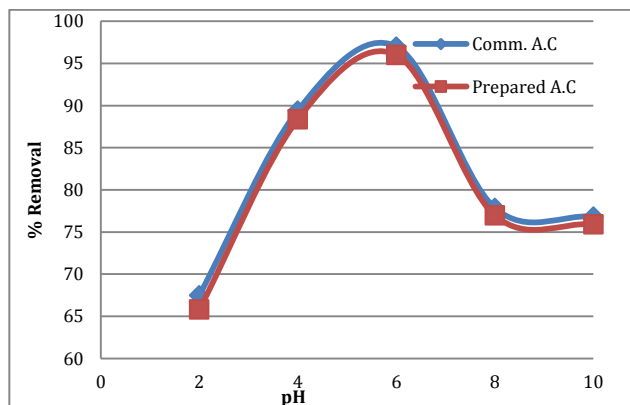


Figure 7: Variation of removal percentage of Pb(II) ions versus pH values for both CAC and PAC at equilibrium condition.

3.4 Adsorption Isotherms

The adsorption capacity of any adsorbent is concenter a specific function of concentration and medium temperature of adsorbate. The two semi-empirical equations for example the Langmuir and Ferundlich isotherm equations could be used to determine the amount of adsorbed material (Banat, and Mohai, 2000). Experimental analysis is usually used to calculate the coefficient of these isotherms for various systems. In this study the coefficient of Langmuir and Ferundlich isotherms of adsorbents in existence of lead are determined from experimental data.

3.4.1 Ferundlich Isotherm

It is the earliest known relationship describing the sorption equation derived empirical in 1912. This fairly satisfactory empirical isotherm can be used for non-ideal sorption that involves heterogeneous surface energy systems and is expressed by the following equation (Weber, 1972; Metcalf, and Eddy, 2003):

$$Q_e = \frac{x}{m} = K_f C_e^{\frac{1}{n}} \quad (2)$$

where:

Q_e : The amount of adsorbate adsorbed per unit mass of the adsorbent.

x : The mass of the adsorbate (mg).

m : The mass of the adsorbent (mg).

C_e : The equilibrium concentration of the adsorbate (mg/l).

K_f : Is roughly an indicator of the adsorption capacity of the adsorbent which can be defined as the adsorption or distribution coefficient (mg/g)

$1/n$: Is the adsorption intensity or surface heterogeneity.

In general, as the K_f value increases the adsorption capacity of adsorbent for a given adsorbate increases. The magnitude of the exponent, $1/n$, gives an indication of the favorability of adsorption, the slope $1/n$ ranges between (0 and 1), becoming more heterogeneous as its value gets closer to zero. Values of $n > 1$ represent favorable adsorption condition. Eq. (2) may be linearized by taking logarithms (Metcalf, and Eddy, 2003):

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e \quad (3)$$

A plot of $\log Q_e$ vs. $\log C_e$ results in a straight line with a slope of $(1/n)$ and an intercept (k).

Experimental data are often plotted in this manner as a convenient way of determining whether removal of material from solution is accomplished by adsorption and as means of evaluating the constants (k) and (n).

3.4.2 Langmuir Isotherm

Langmuir isotherm is also one of the well-known isotherms, which can be defined through following equation,

$$Q_e = \frac{x}{m} = \frac{abC_e}{1+abC_e} \quad (4)$$

In this equation (x/m) and C_e are the same as variables of Ferundlich isotherm; a and b are constant of Langmuir isotherm data. Eq. (4) represents the inversion of Eq.(5).

$$\frac{1}{Q_e} = \frac{1}{ab} * \frac{1}{C_e} + \frac{1}{a} \quad (5)$$

It is clear that the plot of $1/Q_e$ versus $1/C_e$ is a straight line with slope of $1/ab$ and intercept of $1/a$. Therefore the values of Langmuir isotherm coefficient (a and b) for various adsorbents-heavy metals systems can be estimated from the slop and intercept of the plot of $1/Q_e$ versus $1/C_e$. The value of constant (a) equals to the inverse of intercept while the value of constant (b) equals to the inverse of slope per (a).

The Langmuir, and Freundlich isothermal adsorption models were applied for Pb(II) ions adsorption utilized by commercial and prepared activated carbon which having the same particle size (1.68-2 mm) equivalent to (12 -10 mesh number) at pH equals 6 and constant temperature. Figures (8) to (11) depicted the application of the data obtained on these models. The parameters for each model were obtained and presented in table (1) and (2).

Figures (8), (9), (10), and (11) show the linear plot of Langmuir and Freundlich isotherms for adsorption of Pb(II) on commercial and prepared activated carbon. The model parameters obtained by applying Langmuir and Freundlich models to the experimental data are given in table (1) and (2). It can be found that the regression coefficient R^2 obtained from Freundlich

model are closer to 1 than that of the Langmuir model, suggesting that the Freundlich isotherm fits better with the adsorption of Pb(II) on commercial and prepared activated carbon.

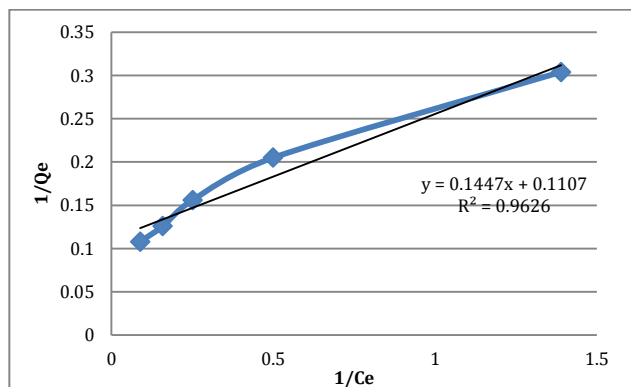


Figure 8: Langmuir adsorption isotherm model of Pb(II) ions adsorbed onto PAC at pH = 6 and constant temperature

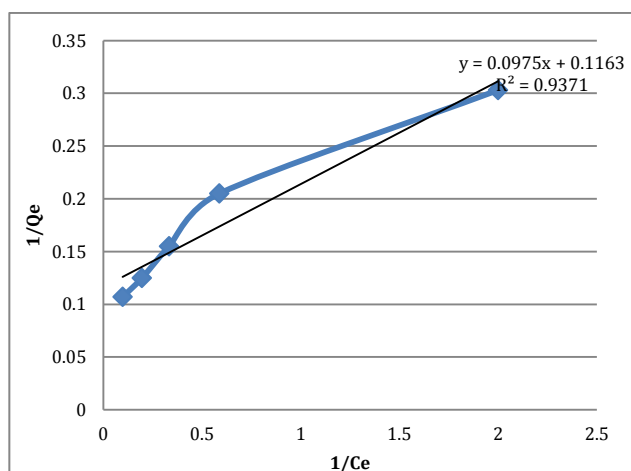


Figure 9: Langmuir adsorption isotherm model of Pb(II) ions adsorbed onto CAC at pH = 6 and constant temperature

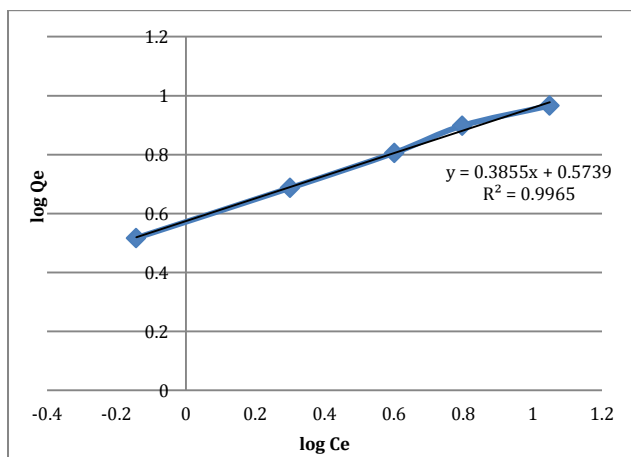


Figure 10: Freundlich adsorption isotherm model of Pb(II) ions adsorbed onto PAC at pH = 6 and constant temperature

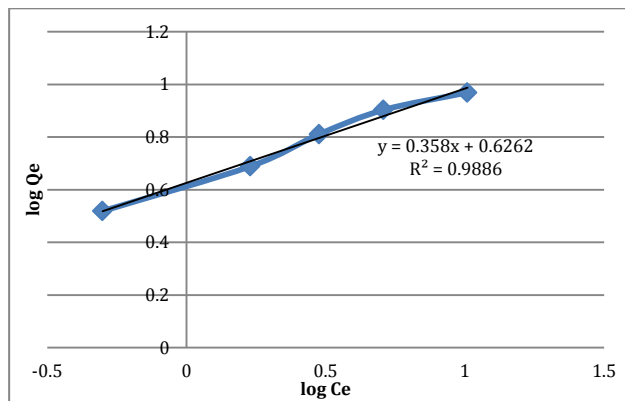


Figure 11: Freundlich adsorption isotherm model of Pb(II) ions adsorbed onto CAC at pH = 6 and constant temperature

Table 1: Numerical amounts of Langmuir isotherms coefficients for different systems

System	1/a	a	1/ab	b	R ²
Commercial activated carbon CAC	0.1163	8.598	0.0975	1.193	0.9371
Prepared activated carbon PAC	0.1107	9.0334	0.1447	0.7651	0.9626

Table 2: Numerical amounts of Ferundlich isotherms coefficients for different systems

System	1/n	n	Log K _f	K _f	R ²
Commercial activated carbon CAC	0.358	2.7933	0.6262	4.2286	0.9886
Prepared activated carbon PAC	0.3855	2.5940	0.5739	3.7489	0.9965

Conclusions

The main conclusions which are achieved from this research are:

- The commercial and the developed activated carbon used in this study are found to have a high capability for removing the Pb(II) ions from aqueous solution.
- The adsorption method is successfully applied to remove Pb(II) ions from aqueous solution. The removal efficiency of lead(II) ions was found to be dependent on various parameters such as pH of the solution, initial lead concentration, mass of adsorbent.
- The results obtained showed that the removal efficiency of the Pb(II) ions by the commercial and prepared activated carbon increased with time, and this increment divided to three significant stages. The first stage is an instantaneous removal of Pb (II) ions which occurs within time less than 10 minutes, after that (second stage), the curves are gradually increased with time from 10 to 200

minutes; till become as plateau after 200 minutes, which recognized as the third or final stage.

- The experimental results showed that the removal efficiency of the Pb(II) ions by the commercial and prepared activated carbon decreases with increase in initial Pb(II) ions concentration, and increases with increase in mass of adsorbent.
- The pH of the solution played an important role in influencing the ability of adsorbents towards the metal ion. The increase in the pH of solution from 2 to 6 led to increase in the removal efficiency; while, the removal efficiency decreased for pH values above 6.
- The optimum conditions which have been obtained in adsorption process for treating the aqueous phase were 50ppm for initial lead concentration, 6 pH for solution acidity, and 15 g/L for mass of adsorbent.
- The equilibrium data obtained for the commercial and prepared activated carbon are fitted to the Freundlich model more than the Langmuir model.

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