



PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM ACACIA ARABICA AND ITS POTENTIALITY IN THE REMOVAL OF LEAD BY ADSORPTION

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ABSTRACT

The most commonly used adsorbent is activated carbon and it generally has high metal adsorption capacity. Although activated carbon is effective in the removal of metal ions from wastewater, it is expensive and requires chelating agents to enhance its performance, thus increasing treatment cost. For the past two decades, researchers have focused on using low-cost, efficient sorbents for heavy metal adsorption. The aim of this study is to investigate the surface characterization, chemical composition and adsorption properties of KOH activated *Acacia Arabica* seed (AASC) carbon in the removal of heavy metal ion removal. Activated carbon was prepared from *Acacia Arabica* seed (AASC) by potassium hydroxide activation and characterized for pH, bulk density, moisture content, ash content, volatile matter, iodine number, and oxygen functional groups. Its potentiality tested to remove lead (II) from aqueous solutions in order to consider its application to the purification of metal finishing wastewater. The equilibrium adsorption data was applied to the Langmuir, Freundlich isotherm models. Adsorption experiments were performed in batch systems, using Pb^{2+} initial concentration, pH of solution, and contact time as variables. The results showed that the experimental data were fitted well by the Langmuir model. Kinetic results revealed that the adsorption process obeyed a pseudo-second order model and intra-particle diffusion was the rate controlling step. Agricultural waste material being highly efficient, low cost and renewable source of biomass can be exploited for heavy metal remediation. The high adsorption capacity of AASC makes it a suitable low-cost material for the removal of Pb (II) from aqueous solutions.

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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]. A number of well-documented methods are known and reported in the literatures for potential applications in removal, extraction, separation, and pre-concentration of lead from various water matrices as well as other environmental samples. These include precipitation, ion exchange, coagulation, floatation, reverse osmosis, membrane filtration, and solvent extraction [5]. Among these methods, adsorption is highly effective and economical one [6]. The use of various adsorbents for the removal of heavy metal ions from aqueous solution is of great interest due to environmental concerns. Activated carbons are widely used for the removal of heavy



metal ions from aqueous solution [7]. The production of activated carbon from agricultural byproducts has both economic and environmental effects, as it converts unwanted, low-value agricultural waste to useful high-value adsorbent [8]. 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 [9]. 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 [10], *Euphorbia rigida* [11], hazelnut husks [12], wheat bran [13], apple waste [14], coconut shell [15], and *Terminalia arjuna* nuts [16] have received increasing attention for the removal and recovery of pollutants in aqueous media. Atomic absorption spectrometry (AAS) with its relative low cost good analytical performance is the main instrument in the laboratories for the determination of heavy metals.

The production of activated carbon from agricultural byproducts has potential economic and environmental impacts. Firstly, it converts unwanted low-value agricultural waste to high-value adsorbents. Secondly, activated carbons are increasingly used in water to remove organic chemicals and metals of environment and economic concern. Finally, it will reduce the importation of activated carbon wherefore increasing our economic base as a country [17]. Thus the purpose of this study is to prepare and characterize the carbons obtained from plantain fruit stem, activated with orthophosphoric acid zinc chloride and the untreated carbon. Within the current paper, we describe our efforts to remove lead (II) ions from aqueous solution by using the activated carbon produced from *Acacia Arabica* seed carbon (AASC) with KOH chemical activation. AASC, an agricultural byproduct, was used for the preparation of the activated carbon. The adsorption of lead (II) ions onto the activated carbon was investigated with variations in the parameters of pH, contact time, lead (II) ions concentration and Dose. The adsorption isotherm and kinetic models for lead (II) adsorption onto the activated carbon was also studied.

Materials and Methods

Adsorbent Preparation

Seeds (Dry fruits) of *Acacia Arabica* were obtained from local agricultural fields (Nellore) and were washed well with tap water to remove dust and other impurities and completely dried in natural manner. Dried in sunlight about one week and cut into small pieces, carbonized in muffle furnace (in absence of air) about 7 hours at 500°C. After carbonization, carbon was poured in double distilled water, filtered and washed in several times with cold double distilled water and dried in an air oven at 110°C for overnight. Then, the carbon was sieved into desired particle sizes. The carbonized material (AASC) in powder form (about 50 g) was taken in a one liter volumetric flask and mixed with approximately 700-800 ml of 1% KOH solution and boiled for 2 to 3 hours on flame for chemical activation. After KOH treatment they were filtered and washed several times with hot water to remove un-reacted base from carbon surface, until the effluent pH was retained i.e. 7.00.

Chemicals and equipment

The chemicals lead (II) nitrate [$Pb(NO_3)_2$] of AR grade were received from Sd Fine Chemicals Limited, India. The pH of the wastewater was adjusted by using 0.1 N HCL and 0.1 N NaOH and the pH was measured using a pH meter (Elico Limited, India). The concentration of Pb(II) ions were observed using an atomic adsorption spectrophotometer (AAS) (SL 176 Model, Elico Limited, Chennai, India).

Frontier Transform Infrared Spectroscopy (FTIR)

Surface chemistry of the synthesized AASC in this study was investigated using FTIR spectroscopy. FTIR spectra for prepared sample were recorded using Perkin Elmer Spectrophotometer. All measurements



were undertaken in the range from 400 to 4000 cm⁻¹ with a resolution of 1 cm⁻¹ for each scan. To prepare samples for the run, all samples of the AC were grounded with KBr salt at a ratio roughly 1/50 of all samples. After that, the sample was made as pellet. The results of FTIR for AASC are shown in Figure 1.

Adsorption experiments

Batch Mode Adsorption experiments were carried out to investigate the adsorption efficiency at varied initial concentration (10-50 mg/L), pH (2.0-8.0), adsorbent dosage(1.5-12 g/L) and contact time(10-50 min.). All the experiments were performed at room temperature of 30±1°C [11]. Batch mode experiments were carried out in 100 ml conical flask with 50 ml of the test solution and shaken in Benchtop incubator cum orbital shaker(Model: Remi Shaker). At the end of required time interval, the samples were removed from the shaker and filtered using Whatman No. 42 filter paper The unadsorbed metal ion was recorded using Elico (India) made Atomic adsorption spectrophotometer (AAS). The amount of Lead taken up and the percentage of removal of the Pb(II) by the adsorbent were calculated by applying Eqn. 1 and 2, respectively:

$$q = \frac{(C_o - C_f)}{m} \cdot V \quad \dots\dots\dots(\text{Eqn 1})$$

$$\% \text{Removal} = 100 \cdot \frac{(C_o - C_f)}{C_o} \quad \dots\dots\dots(\text{Eqn 2})$$

where *q* is the amount of lead taken up by the adsorbent (mg g⁻¹), *C_o* is the initial lead concentration put in contact with the adsorbent (mg L⁻¹), *C_f* is the lead concentration (mg L⁻¹) after the batch adsorption procedure, *m* is adsorbent mass (g) and *V* is the volume of the lead solution (L).

The Point Zero Charge/ pH zero point charge (pH_{ZPC}) of the Activated Carbon Sample: The isoelectric point of these materials was investigated as point zero charges. The PZC value for the AC sample was calculated according to the potentiometric titration [18]

Surface Characterization

Surface functionality of carbon sorbents is characterized by its responsibility for all activity and reactivity as well as capability for all adsorption properties and processes. In this paper, IR was used to obtain information about the chemical structure and functional groups of the AASC. The FTIR spectrum represented in Figure 1 is showing few characteristic peaks that are mainly related to the organic nature of AC [20]. A peak centered at 1110–1120 cm⁻¹ is mainly due to ν (C–O) of –carbonyl functional group. The strong bands appearing at 1520 and 1670 cm⁻¹ are ascribed to the formation of oxygen functional groups based on highly conjugated C=O stretching in carboxylic or carboxylate group as well as carbonyl group. A band was observed at 2860 cm⁻¹ and ascribed to the presence of aliphatic compounds. A broad band in the region 3300–3600 cm⁻¹ is typically attributed to ν O–H stretching or adsorbed water molecule [21]. After adsorption of heavy metal these peaks are remain same but intensity of peaks slightly changes. The spectral analysis of AASC before and after adsorption of Pb ions showed that the peaks either decreases in intensity or disappear might involve in metals adsorption[22]. These results strongly suggested that the adsorption process is purely physisorption process

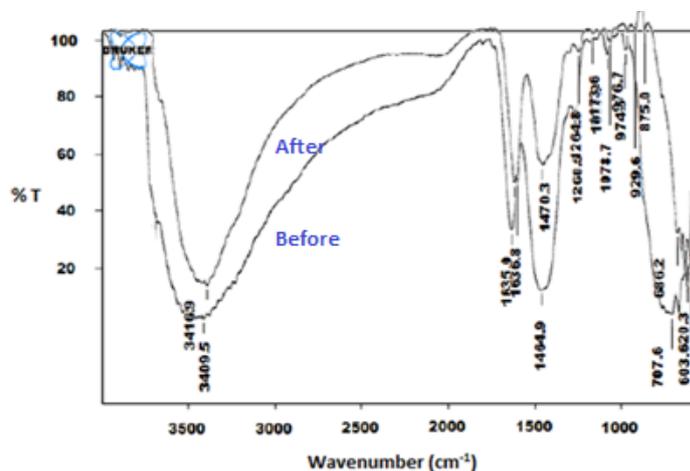


Figure 1: FT-IR spectrum of AC sorbent before and after adsorption of Pb²⁺ on to AASC

SEM-EDX analysis

The morphology of the Ag-NPs was analyzed using an SEM. The powdered AASC sample uniformly spread and sputter coated with platinum in an ion coater for 120 seconds, then observed by SEM JEOL-JSM 6360 MODEL, JAPAN). Elemental analysis of the powdered AASC was conducted using an EDX detector (EDS, EDAX Inc., Mahwah, NJ, USA) attached to the SEM machine.

Results and Discussion

Physicochemical Characteristics of Activated Carbon

The physicochemical characteristics like pH, moisture content, bulk density, surface area, and loss of mass on ignition studied as per the standard procedure [19] 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 1.5 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 (0.25%) 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 352 m²/g and is higher than a low cost agro-based adsorbent such as palm pith carbon (188 m²/g) [23] but lower than peanut hull carbon (354 m²/g) [24]. The value of pH_{ZPC} of the AASC is clear that all the samples showed weak alkaline pH value i.e., 8.02.

Table 1: Characteristics of the AASC sample

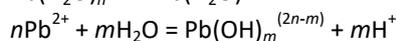
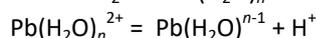
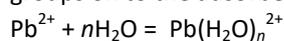
Parameter	Value
pH	7.02
Moisture (%)	0.25
Bulk density (gmL ⁻¹)	0.61
Surface area (m ² /g)	352
Particle size range (µm)	75–120
% Loss of mass on ignition	0.65
pH _{ZPC}	8.02



Lead Sorption in Various Controlling Experimental Factors

Effect of pH

The pH of the solution is an important controlling parameter for the adsorption of heavy metals on the sorbents and influences the metal speciation and surface metal binding sites. The effect of pH on the adsorption of Pb (II) at different initial Pb (II) concentrations (10, 25, and 50 mg/L) at 25°C is shown in Figure 2. The results show that adsorption of Pb (II) onto AASC is affected by pH. This is partly due to the fact that hydrogen ion (H⁺) themselves is strong competing ions and partly that the solution pH influences the chemical groups on to the adsorbent surfaces [25]. The process involved for Pb (II) adsorption are the following [25]:



The maximum percent removal of Pb (II) was observed at pH 4 and significantly decreased at higher pH values. Above pH 4, the sorption yield decreases with increasing pH because hydrolysis and precipitation begin to play an important role in the sorption of Pb (II) [26].

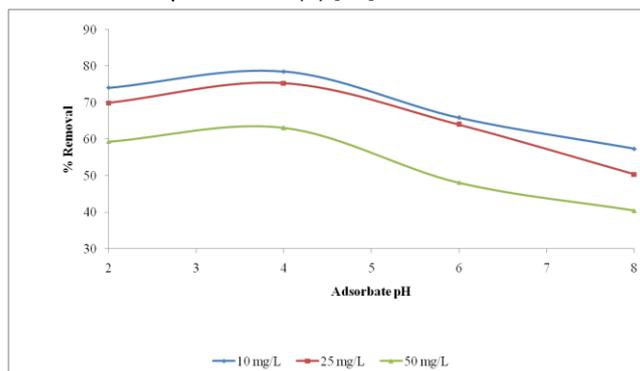


Figure 2. Effect of pH for the removal of Pb (II) onto AASC

Effect of adsorbent Dose

The adsorbent dosage was varied from 2 to 10 g L⁻¹. The effect of the amount of AASC on Pb²⁺ removal is presented in Figure 3 and the removal of Pb²⁺ increases with increasing AASC dosage. The Pb²⁺ removal was not changed significantly for 5 g/L adsorbent dosage and higher. For this reason, the adsorbent dosage of AASC for Pb²⁺ removal was determined to be 5 g/L for further adsorption experiments. Similar observations also found in Pb (II) removal using mineral adsorbents by other researchers also.

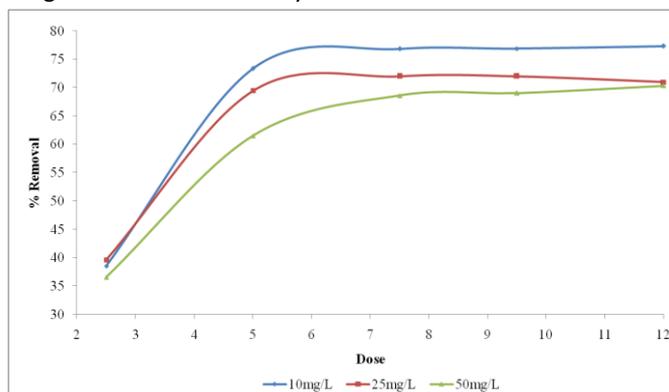


Figure 3: Effect of adsorbent dosage for the removal of Pb (II) onto AASC

Effect of Initial Lead Concentration

The effect of initial concentration on the percentage removal is also shown in Figure 4. 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. The SEM image and X-ray spectrum of AASC after adsorption can be seen in Figure 5. The existence of a peak on the spectrum belonging to lead clearly proves that the accumulation of lead (II) ions onto AASC occurred.

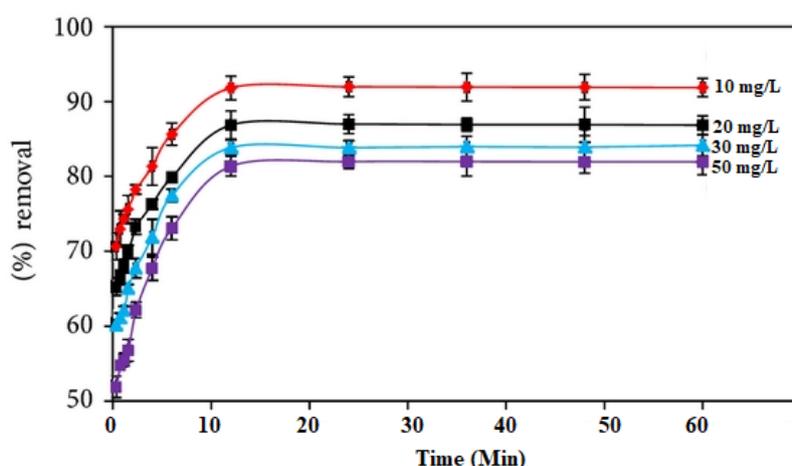


Figure 4: Effects of agitation time on lead ions removal

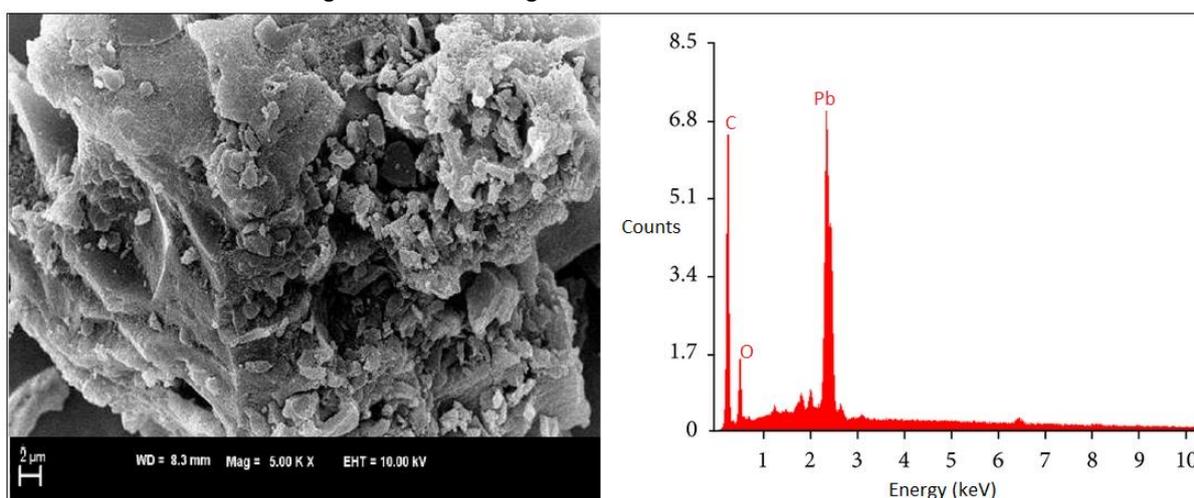


Figure 5: SEM image and EDX spectrum of AASC after lead (II) adsorption.

Adsorption Isotherm study

A number of different equations can be used to predict theoretical adsorption capabilities for different adsorbents. For this study, Langmuir and Freundlich isotherm equations have been used to predict adsorption capabilities of metals on AASC.



The Langmuir isotherm assumes that adsorption takes place at specific homogeneous sites within the adsorbent and has found successful applications in many adsorption processes of monolayer adsorption. The linearized Langmuir isotherm is written as follows [22-26]:

$$C_e / q_e = 1 / K_L q_m + C_e / q_m$$

where q_m is the maximum monolayer adsorption capacity of the adsorbent (mg/g), and K_L is the Langmuir adsorption constant (L/mg), which is related to the free energy of adsorption.

The plot of C_e/q_e versus C_e gives a straight line with slope $1/q_m$, and intercept $1/K_L q_m$. Fig. 6a, presents the plot of C_e/q_e vs C_e for the Pb (II) removal by adsorption onto AASC.

In addition, a dimensionless constant called separation factor, R_L , can be used to express an essential feature of Langmuir isotherm [26].

$$R_L = \frac{1}{1 + aC_m}$$

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 $R_L < 1$ or $R_L = 0$. The calculated was 0.46 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 2.

The Freundlich isotherm [22-26] has been derived by assuming an exponentially decaying sorption site energy distribution. This experimental model can be applied to non-ideal sorption on heterogenous surfaces as well as multilayer sorption. The empirically derived linearized Freundlich isotherm is defined as follows:

$$\log q_e = \log K_f + 1/n \log C_e$$

where K_f (L/g) and n (dimensionless) are the constants that can be related to the adsorption capacity and the adsorption nonlinearity intensity, respectively.

The values of K_f and $1/n$ may be calculated by plotting $\log q_e$ against $\log C_e$. The slope is equal to $1/n$ and the intercept is equal to $\log K_f$. Fig. 6b, presents the plot of $\log q_e$ as a function of $\log C_e$ for the Pb (II) removal.

The Freundlich constants n and K_f Langmuir constants b and q_m and the correlation coefficient R^2 are given in Table 2. The calculated value of Freundlich constant is within the range ($0.1 < n < 1$), reported in the literature [27] 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.

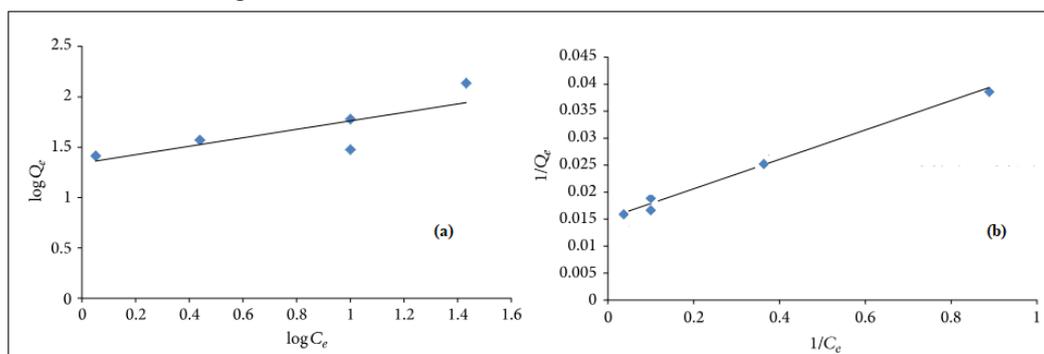


Figure 6 (a) The linear Langmuir adsorption isotherm (b) The linear Freundlich adsorption isotherm for Pb(II) adsorption by the AASC



Table 2: Langmuir and Freundlich constants for Pb²⁺ adsorption using AASC

Adsorption isotherm	Parameter	Value
Langmuir	q_m (mg/g)	29.64
	b (L/mg)	0.062
	R_L	0.46
	R^2	0.9984
Freundlich	K_f	0.077
	n	0.482
	R^2	0.9515

Effect of Contact Time

In the adsorption system contact time plays a vital role, irrespective of the other experimental parameters that affects the adsorption kinetics. The effect of contact time on the percent removal of Pb (II) was investigated at the optimum initial concentration of Pb(II). It was found that the removal of metal ions increases with increase in contact time to some extent. The relative increase in the extent of removal of metal ion (Q_e) after 30 minutes of contact time is negligible and hence it is the optimum contact time. Preliminary investigations on the uptake of metal ions on the adsorbent materials at their optimum conditions indicated that the process is quite rapid.

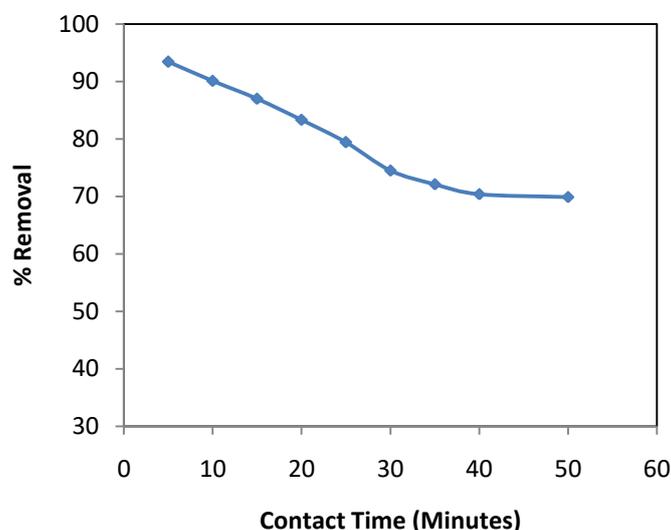


Figure 7: Effect of Agitation time on the % removal of Pb (II) by adsorption onto AASC

Adsorption Kinetic models

The adsorption kinetics shows the evolution of the adsorption capacity through time and it is necessary to identify the types of adsorption mechanism in a given system. The following models are used to describe the adsorption kinetics behavior

Lagergren’s Pseudo-First Order Model

The adsorption kinetics can be described by a pseudo-first order equation as suggested by Lagergren and modified after definite integration by application of the conditions $t = 0$ to $t = t$ and $q = 0$ to $q = q_e$, becomes



$$\ln(q_e - q_t) = \ln q_e - \ln k_1 t$$

Where: $k_1(\text{min}^{-1})$ is the rate constant of the pseudo-first order model, q_t (mg /g) denotes the amount of adsorption at time t(min), and q_e (mg /g) is the amount of adsorption at equilibrium. The adsorption rate constant, k_1 , can be experimentally determined by the slope of linear plots ($\ln(q_e - q_t)$ vs. t and tabulated in Table 3 and in Figure 8.

Pseudo-Second Kinetic Order Model

The linear form of pseudo-second order equation developed by can be written as

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$

Where: $k_2(\text{g/mg.min})$ is the rate constant of the pseudo second order. Where k_2 and q_e can be obtained from the intercept and slope of plotting t/q_t v.s. t and tabulated in Table 3.

Table1: Equilibrium Kinetic constants for Pb^{2+} adsorption on AASC

Equation	q_e (mg/g)	k_1 (min^{-1})	k_2 ($\text{g mg}^{-1} \text{min}^{-1}$)	R^2
Pseudo-First Order	0.1737	0.0643		0.956
Pseudo-Second Order	32.1		0.0857	0.999

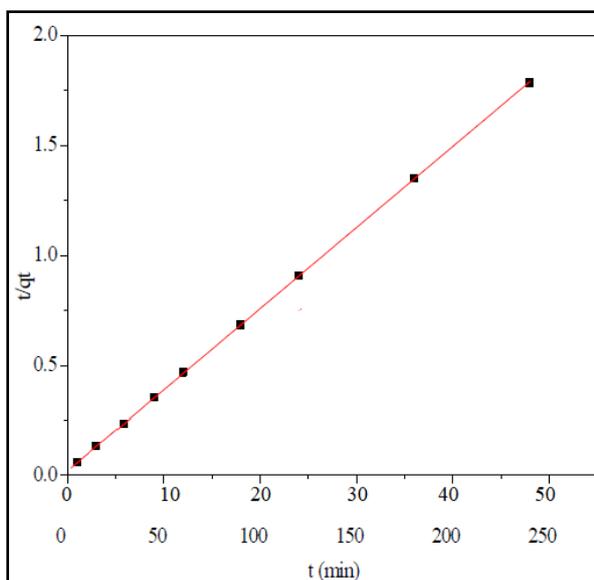


Figure 8: Pseudo-second-order kinetic Model for the adsorption of Pb (II) by AASC

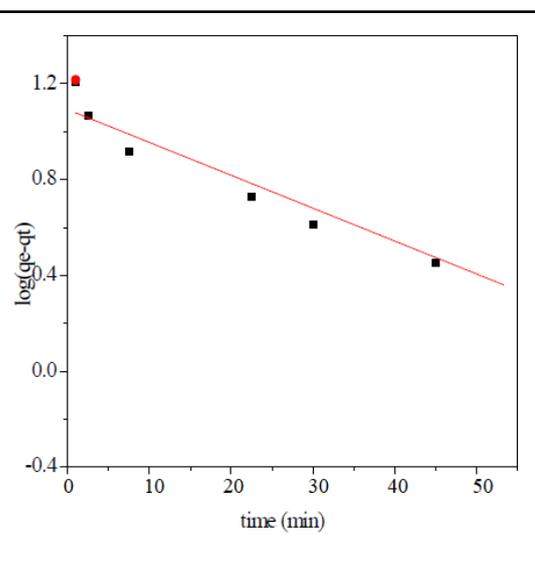


Figure 9: Pseudo-first-order kinetic Model for the adsorption of Pb (II) by AASC

It can be observed from Table 3 that R^2 values for the pseudo-second order kinetic model is all over 0.999. This value suggested that the adsorption processes AASC for Pb^{2+} can be well described by the pseudo-second order kinetic model. The pseudo-second-order adsorption mechanism was predominant referring that adsorption process is controlled by chemisorption which involves valence forces through sharing or exchange of electron between the solvent and the adsorbate Since neither the pseudo-first-order nor the second-order model can identify the diffusion mechanism, the kinetic results were further analyzed by the intra-particle diffusion mode to elucidate the diffusion mechanism.



Conclusion

Based on the results, it is clear that the Pb (II) removal values achieved with KOH modified Acacia Arabia seed activated carbon was higher adsorption capacity. The main advantages of this removal procedure include (i) simplicity, (ii) cost effectiveness and (iii) rapidity. Based upon the experimental results of this study, the following conclusions can be drawn: It was found that the sorption process is pH-dependent and the maximum adsorption capacity of Pb(II) is at pH 5. It was found that the extent of Pb²⁺ adsorption increases upon increasing initial concentration and contact time till reached to equilibrium. Pb²⁺ uptake almost remained constant after 30 min. The kinetic indicated that the adsorption kinetics of lead on AASC adsorbent followed the pseudo-second order model. The equilibrium data were analyzed using the Langmuir and Freundlich isotherms and the characteristic parameters for each isotherm were determined. The results showed that the experimental data were correlated reasonably well by the Langmuir isotherm model.

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