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Contributed Paper

Batch Removal of Pb (II) from Aqueous Solution using Activated Carbon Prepared from Mangosteen Shell Activated with H₂SO₄

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ABSTRACT

Lead is a toxic pollutant which has serious effects on the environment and human health. In this work, an activated carbon was produced from Mangosteen shell (BTMC) and used in the elimination of Pb(II) from aqueous solution. The adsorbent was characterized by FT-IR, SEM and XRD studies. Batch experiments indicated that the quantitative removal Pb (II) occurs at an optimum experimental condition at pH 5 and carbon dose of 40 mg/100 mL. The maximum removal of Pb (II) was obtained at 27° C. Langmuir isotherm found to be applicable with Q₀ value of 58.48 mg g⁻¹. The results of the adsorption kinetics are described better with the pseudo-second-order model (R² = 1). The practical utility of the produced carbon was tested using lead battery wastewater. The recovery of the Pb (II) from mangosteen shell is found to be 86% using 0.1 M HCl after five cycles. The results were compared with commercial activated carbon and BTMC is observed to be an efficient and economical adsorbent for the removal of Pb (II) ions from aqueous solution.

Keywords: adsorption, mangosteen shell, lead, isotherm, desorption.

1. INTRODUCTION

Industrial and agricultural wastewater introduces heavy metals into water bodies. The presence of these heavy metals is considered to be toxic, persistent, carcinogenic and mutagenic in nature [1]. Lead a toxic inorganic pollutant enters into the water from industries, manufacturing batteries, pigments, electroplating, ammunition, mining, steel, and automobiles. Research on lead has become a dominant topic for environmental and medical scientists since it has no known

biological use and it is toxic to most living things [2]. Lead can cause anemia, brain damage, anorexia, malaise, loss of appetite [3]. To mitigate lead poisoning WHO has restricted its maximum concentration in drinking water to 0.01 mgL⁻¹ [4] and Environmental Regulatory Authority of India has directed to lower the concentration of lead to 1.0 mgL⁻¹ in effluents before disposal [5]. Many conventional methods like chemical filtration, coagulation, flocculation, ion exchange

are used for removal of lead from wastewater but adsorption is considered as a very suitable method because of its simplicity and cost-effectiveness [6]. Agricultural waste like banana peel [7], Lentil husk [8], Pistachio Shell [9], cashew nutshell [10], groundnut shell [11] has been used to remove lead. Pretreatment of plant waste can extract soluble organic compounds and enhance chelating efficiency. Concentrated sulphuric acid is being a powerful dehydrating agent, low-cost chemical with different porous structure is used as an activation agent for the removal of lead from water [12, 13]. Mangosteen (*Garcinia Mangostana* Linn.) belongs to Hypericaceae family. It is considered as Queen of fruits due to its taste and function in disease treatment. The pericarp (outer shell or rind) is hard and for every 10 kg of mangosteen harvested, more than 6 kg of mangosteen peel is generated [14]. Mangosteen shell possessed antioxidant, antitumeral, anti-allergy, anti-inflammation, antibacterial and antiviral activities. Xanthones are claimed to be responsible for these biological activities [15]. The utilization of mangosteen shell as biosorbent is due to the presence of phenolic acid in the pericarp which has the ability to bind heavy metals from the aqueous solution [16]. This work aims to produce sulphuric acid and bicarbonate treated mangosteen shell activated carbon for use in the removal of lead from aqueous solution. Commercial activated carbon procured from the market was used for comparison purpose.

2. MATERIALS AND METHODS

2.1 Preparation of Adsorbent

Mangosteen shell an agricultural waste was obtained from Thenkasi, India. The sample was washed with distilled water to eliminate the soluble organic matter and, dried in the oven at 110°C. The dried shell was powdered, sieved to 20-50 ASTM mesh size. It was then treated with H_2SO_4 (1:1) weight ratio and kept in an air oven at $150 \pm 5^\circ\text{C}$ for 24 h. The resulting carbonized material was washed with distilled water to remove the free acid and soaked in 1% sodium

bicarbonate until the effervescence ceases and further soaked in the same solution for 24 h to remove the residual acid. The activated carbon (BTMC) obtained by this method was washed with distilled water, dried at 105°C and then sieved to 20-50 ASTM mesh size.

2.2 Preparation of Pb (II) Solution

A stock solution (1000 mg L^{-1}) of Pb (II) was prepared by dissolving 1.599 g of $\text{Pb}(\text{NO}_3)_2$ in distilled water. The solution was diluted as required to prepare standard solutions containing 10-25 mg L^{-1} of Pb (II). 100 mL of Pb (II) solution of the desired concentration is adjusted to the desired pH using dilute hydrochloric acid solution or dilute sodium hydroxide solution.

2.3 Batch Experiments

To evaluate the efficiency of produced materials for the removal of Pb(II) in the aqueous solution, experiments were conducted using BTMC and CAC.

To determine the dependence of the removal efficiency on pH and temperature, the initial pH values of Pb(II) solutions and adsorption temperature were varied from 1 to 10 and 27°C to 47°C, respectively. The effect of the adsorbents dosage on Pb(II) removal efficiency was investigated by applying various amounts of adsorbents (40-320 mg). Adsorption isotherm experiments were performed into a different initial concentration of Pb (II) solutions were shaken in a mechanical shaker to reach equilibrium.

The carbon was separated by filtration and the filtrate was analyzed by a spectrophotometric procedure using PAR [4-(2-pyridylazo) resorcinol reagent for Pb (II) content at 520 nm [17].

3. RESULTS AND DISCUSSION

3.1 Characterization of the Adsorbent

3.1.1 Examination of carbon characteristics

The examination of carbon characteristics reveals that the surface area of BTMC is $209 \text{ m}^2 \text{ g}^{-1}$ and CAC is $150 \text{ m}^2 \text{ g}^{-1}$ and porosity is 50.11% and

36.41% for BTMC and CAC respectively. The decolorizing power of BTMC indicates that the carbon has good adsorption capacity.

3.1.2 FT- IR studies

Fourier Transform Infrared Spectrometer was used to identify the functional groups present in the mangosteen shell and prepared activated carbon. Figure 1(a,b) demonstrates the FTIR spectrum of BTMC before and after adsorption of Pb(II) ion. The spectra show the presence of polyfunctional groups. The strong absorption peak at 3425 cm^{-1} ,

is due to the -OH stretching vibration due to inter and intramolecular hydrogen bonding of alcohols, phenols, and carboxylic acids. The peaks at 2981 cm^{-1} , 2372 cm^{-1} and 1222 cm^{-1} are due to the C-H stretching, $\text{C}\equiv\text{C}$ stretching and -CO stretching vibration of ether. The presence of a sulphonic acid group is confirmed by the peak at 1427 cm^{-1} . It is apparent that some of the peaks shift or become weak showing the incorporation of heavy metal ion Pb (II) within the adsorbent through the interaction of the active functional group after adsorption.

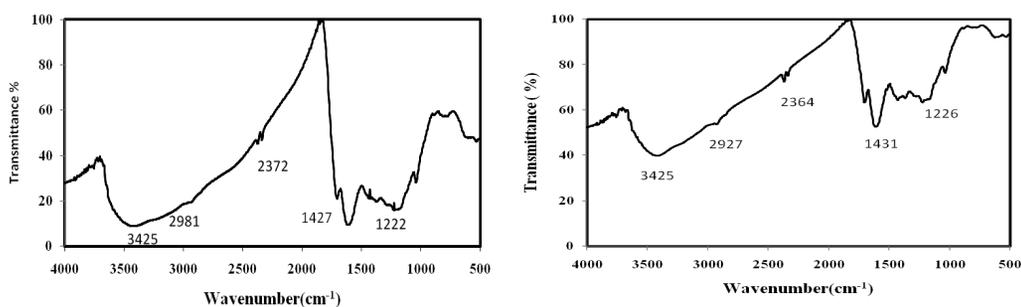


Figure 1. FTIR spectra of BTMC before adsorption of Pb(II) (a), FTIR spectra of BTMC after adsorption of Pb(II) (b).

3.1.3 SEM analysis

Adsorption capacity depends on the chemical structure of the adsorbent. The pore size, shape and volume play important role in the adsorption process. The SEM observation of BTMC shows the presence of isolated pores of varying dimension before and after adsorption of Pb (II) (Figure 2a,b). The cave-like opening before adsorption in BTMC is clearly seen and filled with Pb (II) ion after adsorption due to the chemical treatment with H_2SO_4 . concluded that the BTMC and CAC adsorbent has an adequate morphology for Pb (II) ion adsorption.

3.1.4 X- ray diffraction studies

The X- ray Diffraction (XRD) pattern of BTMC (Figure 3a, b) shows amorphous nature due

to the presence of cellulosic material as explained by Reddy et al [18]. X-ray diffractogram of Pb (II) adsorbed BTMC shows peak at 9.42° , 27.82° which shift to 8.8° , 28.8° in peaks which may be due to the binding of lead on BTMC.

3.2 Effect of Contact Time

The effect of contact time on removal of 10 mg L^{-1} of Pb (II) ion with carbon dosage of $100\text{ mg L}^{-1}/100\text{ mL}$. is shown in Figure 4. The removal of the lead ion increases with time and attains equilibrium in 30 min for BTMC and 210 min for CAC. The rate of removal is higher in the beginning due to a large number of available adsorption sites. The maximum percentage removal of Pb (II) in case of BTMC is 100% and 93% in the case of CAC.

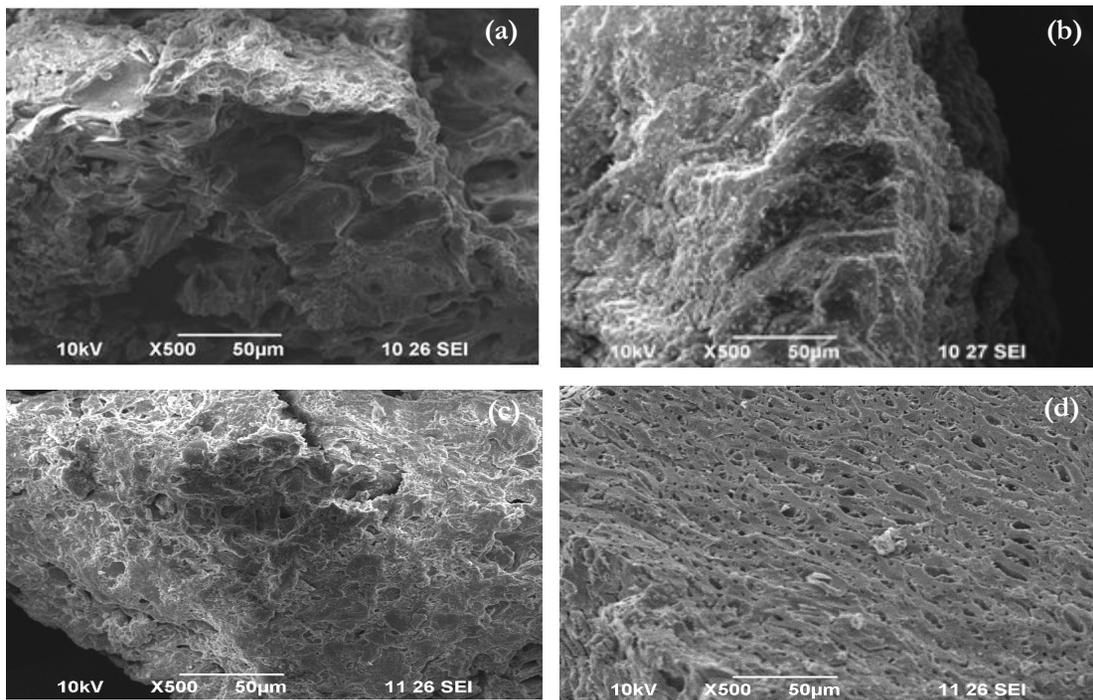


Figure 2. SEM image of BTMC before adsorption of Pb(II) (a), SEM image of BTMC after adsorption of Pb(II) (b), SEM image of CAC before adsorption (c), SEM image of CAC after adsorption (d).

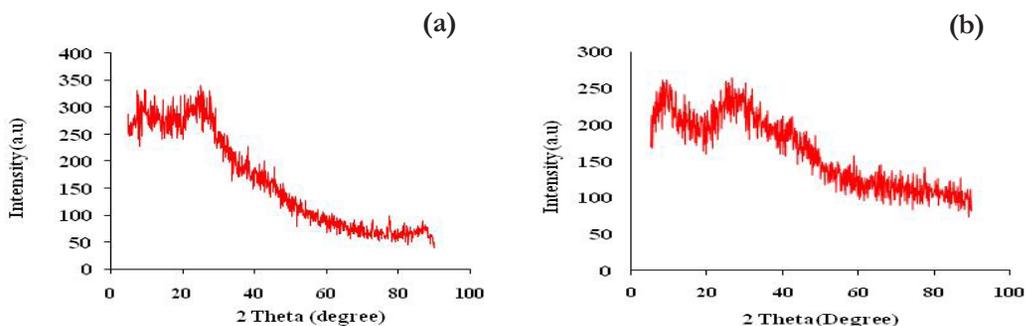


Figure 3. X-ray diffraction pattern of BTMC before adsorption of Pb(II) (a), X-ray diffraction pattern of BTMC after adsorption of Pb(II) (b).

3.3 Effect of Carbon Dosage

Figure 5 illustrates lead uptake by both BTMC and CAC at different carbon dosage. It exhibits that the 100% removal of Pb (II) occurs at a carbon dosage of 40 mg/100 mL in case of BTMC and

85% removal of Pb(II) occurs at a carbon dosage of 120 mg/100 mL for CAC. The data justifies that BTMC is 3 times more efficient than CAC. This may be due to the ion exchange and porous nature of BTMC when compared to CAC.

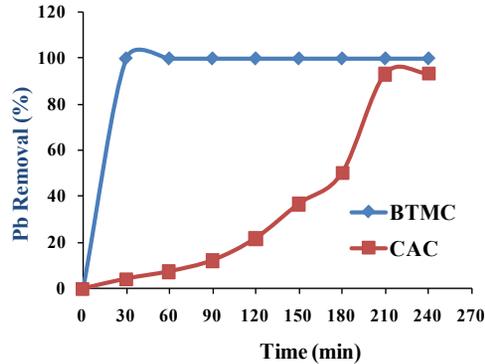


Figure 4. Effect of contact time on the removal of Pb(II) by BTMC and CAC.

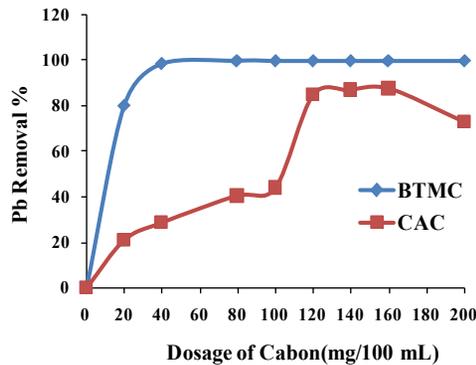


Figure 5. Effect of carbon dosage on the removal of Pb (II) by BTMC and CAC at (pH =5).

3.4 Effect of pH

The pH of the solution has a significant impact on the uptake of heavy metals since it determines the surface charge of the adsorbent, the degree of ionization and the speciation of the adsorbent.

Figure 6 shows that the removal of Pb(II) increases with increase in pH and attains a maximum removal of 100 % and 75% at pH 5 for BTMC and CAC. The adsorption of Pb(II) is low at acidic pH .It can be explained by the fact that at low pH values, electrostatic repulsive forces acts between H_3O^+ and Pb^{2+} ion. As pH increases, adsorption of Pb (II) ions takes place on the surface of the adsorbent replacing H_3O^+ . Above optimum pH, Pb (II) starts precipitating

as $Pb(OH)_2$ and hence causing a decrease in the adsorption yield. Similar results were obtained by other studies on Pb (II) adsorption by Banana Pseudo stem [19].

The mechanism of Pb (II) removal may be explained using ion - exchange model. The pure carbon surface is generally considered to be a non-polar material. According to Frumkin [20], groups such as C_xO or CO_x or C_xO_2 formed on the surface of carbons during activation process make the carbon slightly polar. It has been suggested that surface oxide groups participate in the adsorption of strongly ionized acids and bases as well as in the hydrolytic adsorption of inorganic salts.

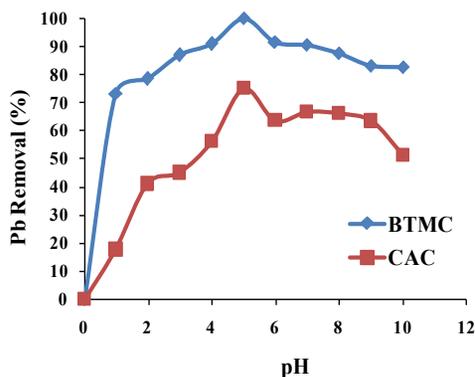
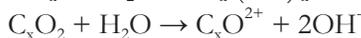
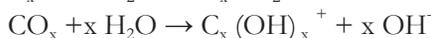
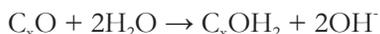
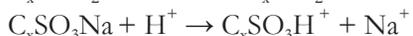
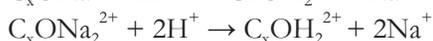
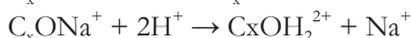
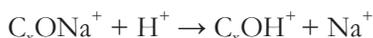


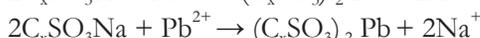
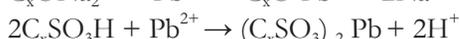
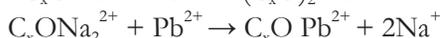
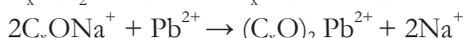
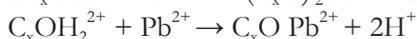
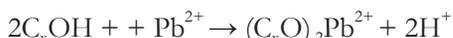
Figure 6. Effect of pH on the removal of Pb(II) by BTMC and CAC at (Carbon dose = 100 mg/100 mL).



Since BTMC was prepared upon treatment with concentrated sulphuric acid followed by soaking in sodium bicarbonate solution. Groups such as C_xONa^+ , $C_xONa_2^{2+}$, C_xSO_3H and C_xSO_3Na may be present. The Na^+ ions in the above groups were exchanged with H^+ ions in the medium as follows



Excess Na^+ ions might have been introduced into BTMC by soaking in sodium bicarbonate solution. When the lead ion is present in solution, its adsorption will free some H^+ ions. At the same time Na^+ ions will be released as follows



3.5 Effect of Temperature

The adsorption of Pb(II) was tested at three different temperatures 27°C, 37°C, 47°C for the initial Pb (II) ion concentration of 10 mg L⁻¹ at a constant adsorbent dosage of 100 mg L⁻¹/100 mL at optimum pH of 5. The percentage removal is found to be 94%, 82%, 79% for BTMC and 56%, 50%, 44% for CAC. The maximum removal of Pb (II) was obtained at 27°C. The percentage removal of Pb (II) ion decreases with increase in temperature. This may be due to the decrease in the surface activity of Pb (II). Similar observation was noted by Gupta et al [21].

3.6 Adsorption Isotherm

The adsorption isotherms studies were conducted with Pb (II) solution of concentration 10-50 mg L⁻¹ maintained at pH 5 at 27°C, a stirring speed of 220 rpm and equilibrated for 24 h.

Adsorption isotherm was studied by Freundlich, Langmuir and Temkin isotherm models.

The Freundlich model is based on the distribution of solute between the solid at equilibrium. The linear form of Freundlich equation is represented as

$$\log \frac{x}{m} = \log K + \frac{1}{n} (\log C_e) \quad (1)$$

where C_e is the equilibrium concentration (mg L⁻¹) and x/m is the amount adsorbed per unit weight

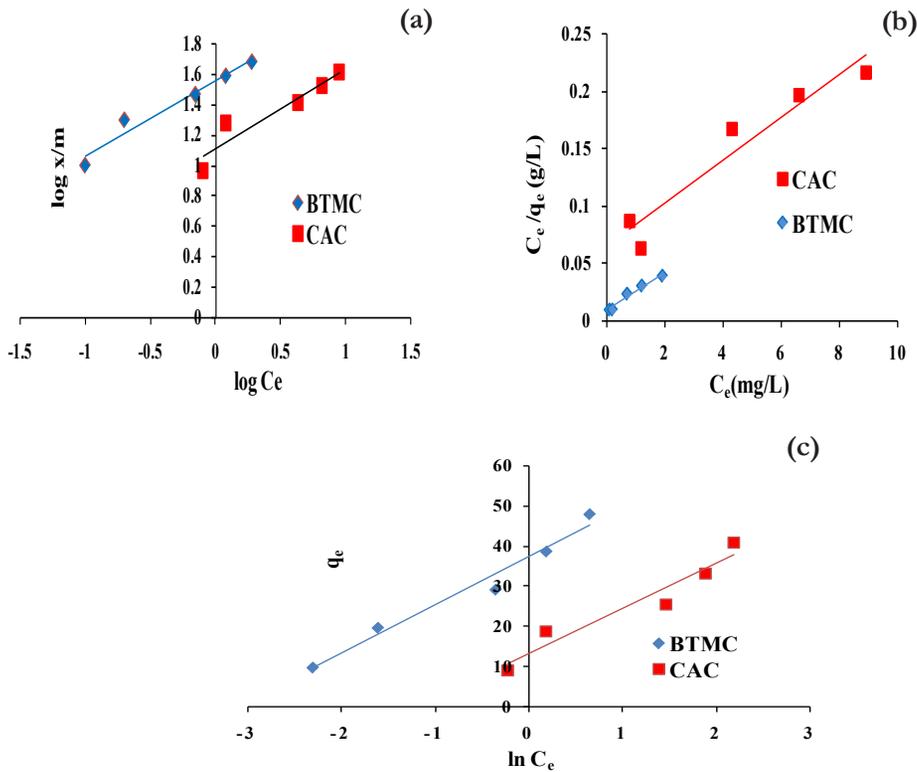


Figure 7. Freundlich adsorption for Pb(II) with BTMC and CAC (a), Langmuir adsorption isotherm for Pb(II) with BTMC and CAC (b), Temkin adsorption isotherm for Pb(II) with BTMC and CAC (c).

of adsorbent (mg g^{-1}). A plot of $\log(x/m)$ versus $\log C_e$ is linear and Freundlich constants can be obtained from the Figure 7a.

The Langmuir equation is used to explain single layer and uniform adsorption.

$$\frac{C_e}{q_e} = \frac{1}{Q_0} b + \frac{C_e}{Q_0} \quad (2)$$

where C_e is the equilibrium concentration (mg g^{-1}), q_e is the amount adsorbed at equilibrium (mg g^{-1}) and Q_0 and b is Langmuir constants related to adsorption capacity and energy of adsorption respectively.

The essential characteristics of Langmuir isotherm can be expressed in terms of a dimensionless constant separating factor or equilibrium parameter R_L which is given as $R_L = 1/(1+b C_0)$ where b is Langmuir constant and C_0 is the initial concentration

of metal ion [22].

The Temkin isotherm is an empirical relationship which was chosen to evaluate the adsorption potential of the adsorbents. Temkin isotherm is represented by

$$q_e = A + B \ln C_e \quad (3)$$

where q_e is the amount of solute adsorbed at an equilibrium concentration of a metal ion in mg g^{-1} . C_e is the equilibrium of the solute in mg g^{-1} . A is the Temkin constant related to adsorption capacity in mg g^{-1} and B is the Temkin constant related to the intensity of adsorption in L mg^{-1} [23].

It is evident from the Table 1 that n value lies between 1 to 10 in BTMC and CAC. This show the favorable adsorption of Pb(II). Q_0 value was found to be 58.48 mg g^{-1} for BTMC and 53.48 mg

Table 1. Isotherm parameters for the removal of Pb(II) from aqueous solution.

Isotherm model	Parameter	BTMC	CAC
Freundlich	K(mg g ⁻¹)	200.49	82.24
	n (L ⁻¹ mg)	1.68	1.39
	R ²	0.919	0.963
	Q ₀ (mg g ⁻¹)	58.48	53.48
Langmuir	B(L ⁻¹ mg)	1.92	0.289
	R ²	0.969	0.924
	R _L	0.215 -0.838	0.064 -0.257
Temkin	A	12.04	11.27
	B	37.06	13.26

g⁻¹ for CAC. The ratio of Q₀ value of BTMC and CAC is found to be 1.09. This shows that BTMC is superior to CAC in the adsorption process. R_L of both BTMC and CAC lies between 0 to 1 indicating favorable conditions for adsorption of Pb (II) ion.

3.7 Adsorption Kinetics

To understand the adsorption mechanism and the rate- controlling steps the kinetic data were tested for Pb (II) on the BTMC and CAC. Pseudo-first-order and pseudo-second-order models were tested with the Pb(II) of concentration 3,5,7,10 ppm.

Mechanism of BTMC and CAC were studied using pseudo- first -order equation of Lagergren [24]. The linear form is generally expressed as

$$\log(q_e - q_t) = \log q_e - K_1 \frac{t}{2.303} \quad (4)$$

where q_e (mg g⁻¹) and q_t (mg g⁻¹) are the amount of adsorption at equilibrium and at time t (min) respectively. k₁ (min⁻¹) is the rate constant of the pseudo-first order reaction. The values were obtained by plotting log (q_e - q_t) versus time t (min). (Figure 8 a, b)

Pseudo-second-order kinetics was obtained from the linear form

$$\frac{t}{q_t} = \frac{1}{K_2} q_e^2 + \frac{t}{q_e} \quad (5)$$

where q_e is the amount of metal ion adsorbed in equilibrium in mg g⁻¹, q_t is the amounts of metal ion adsorbed at time(t), k₂ (g mg⁻¹min⁻¹) is the rate constant for the adsorption process. Using the graph plotted between t/q_t against time t, q_e and k₂ can be calculated. Figure 9(a,b)

The values of the kinetic parameter of both kinetic models are included in Table 2.

To evaluate the applicability of kinetic data relative deviation (P %) was calculated using the equation:

$$P\% = \frac{100}{N} X \left[\frac{q_e^{(exp)} - q_e^{(cal)}}{q_e^{(exp)}} \right] \quad (6)$$

where q_{e(exp)} and q_{e(cal)} are the experimental and calculated value of Pb(II) adsorbed on the adsorbents, N is the number of measurements made per cycle. The lower value of percentage deviation (P %), better is the fit. When P (%) value is less than 5, the fit is considered to be excellent [25]. All kinetic parameters, correlation coefficients and P (%) are listed in Table 2. The correlation coefficient (R²) for the pseudo-second-order model is equal to unity for BTMC and close to unity for CAC. The calculated q_e value was much closer to the experimental q_e value for both carbons. Also,

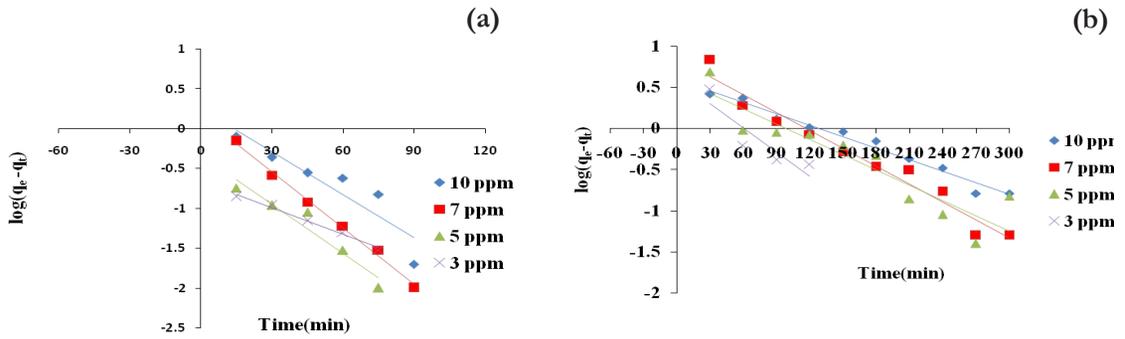


Figure 8. Pseudo -first -order kinetic fit of Pb(II) on BTMC (a), Pseudo –first- order kinetic fit of Pb(II) on CAC (b).

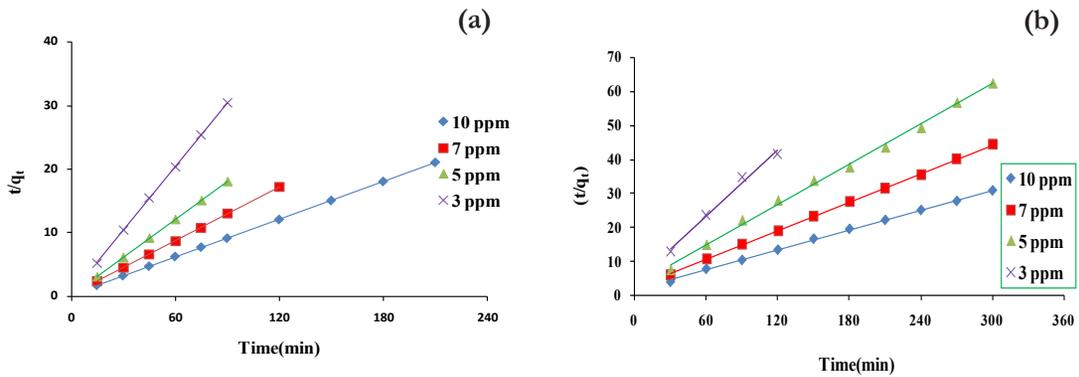


Figure 9. Pseudo- second -order kinetic fit of Pb(II) on BTMC (a), Pseudo- second- order kinetic fit of Pb(II) on CAC (b).

Table 2. Pseudo First-Order and Pseudo-Second-Order constants for Pb (II) adsorption on BTMC and CAC at different initial concentration.

Carbon	CoCon	$q_{e(exp)}$ (mgg^{-1})	$q_{e(cal)}$ ($mg^{-1}g$)	K_1 (min^{-1})	P%	R^2	$q_{e(cal)}$ (mgg^{-1})	K_2 ($gmg^{-1}min^{-1}$)	P%	R^2
Pseudo -first order						Pseudo- second order				
BTMC	10	9.96	1.78	0.018	82.13	0.842	10.10	0.617	1.42	1
	7	6.98	1.46	0.023	79.08	0.994	7.11	0.140	1.82	1
	5	4.98	2.14	0.025	57.03	0.928	5.03	0.215	0.96	1
	3	2.99	1.29	0.112	56.86	0.988	2.99	0.459	0.07	1
CAC	10	9.91	3.98	0.005	59.85	0.977	10.31	0.005	4.01	0.999
	7	6.8	7.05	0.007	11.23	0.969	7.13	0.008	4.82	0.999
	5	4.9	4.11	0.006	16.12	0.840	5.08	0.197	3.75	0.997
	3	2.95	3.9	0.010	32.20	0.810	3.10	0.026	4.95	0.990

the percent relative deviation (P%) is also found to be less than 5% in the case of pseudo-second-order. These values predict that the adsorption kinetics of Pb(II) ions onto the BTMC and CAC is mainly based on a pseudo-second-order equation. A similar observation was noted in cashew nut shell [26].

3.8 Adsorption Mechanism

Weber and Morris introduced a graphical method to prove the occurrence of intra particle diffusion and to determine if it was the rate-determining step in adsorption process [27]. Intraparticle diffusion was characterized using the relationship between specific sorption (q_t) and the square root of time ($t^{1/2}$) as shown below:

$$q_t = k_{id} t^{1/2} \quad (7)$$

where q_t is the amount adsorbed per unit mass of adsorbent (mg g^{-1}) at time $t^{1/2}$ (min) and K_{id} the intra- particle diffusion rate constant ($\text{mg g}^{-1} \text{min}^{-1/2}$).

If the intra-particle diffusion is involved in the adsorption process, then a plot of the amount of Pb(II) adsorbed per unit mass of adsorbent (q_t) against square root of time ($t^{1/2}$) will give a

straight line and the particle diffusion would be the controlling step if this line passes through the origin. The deviation of straight lines from the origin in both BTMC and CAC indicated that intra- particle diffusion was not the only rate- controlling step in the removal of Pb (II) Figure 10.a, b. Some other mechanism like ion-exchange can also control the rate of adsorption.

3.9 Desorption and Regeneration Studies

In order to find out the regeneration lead solution of concentration 100 mgL^{-1} was used HCl solution of concentration varying from 0.1 N to 1 N HCl is used. 0.1 N HCl was used for regenerating BTMC and 0.5 N HCl for CAC for five cycles of operation. Figure 11a,b shows the adsorption-desorption percentage of BTMC and CAC. The results show a slight decrease in the sorption of Pb (II) ions from 94 % to 86 % when treated with BTMC giving better recovery of Pb (II) ions.

The decrease in the sorption may be due to the opening of more surface- active sites present on the sorbent surface on repeated regeneration cycles. However, the recovery of Pb (II) ions decreased rapidly from 72 % to 54 % in the case of CAC in fifth cycle. It was observed that BTMC

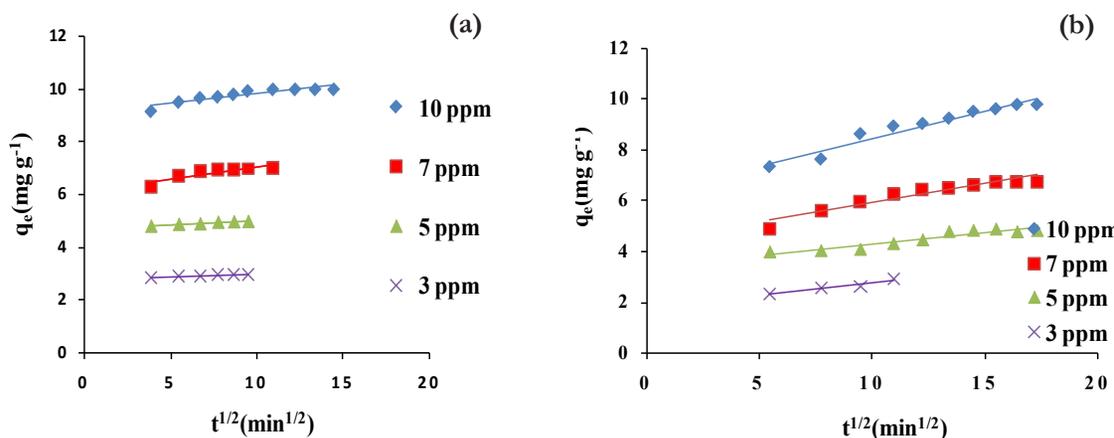


Figure 10. Intraparticle diffusion curve of BTMC (a), Intraparticle diffusion curve of CAC (b).

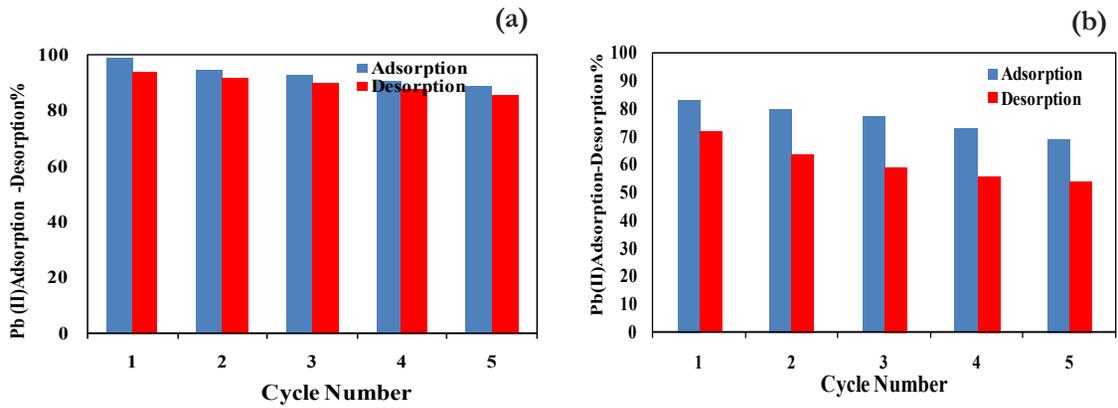


Figure 11. Desorption and regeneration study of BTMC (a), Desorption and regeneration study of CAC (b).

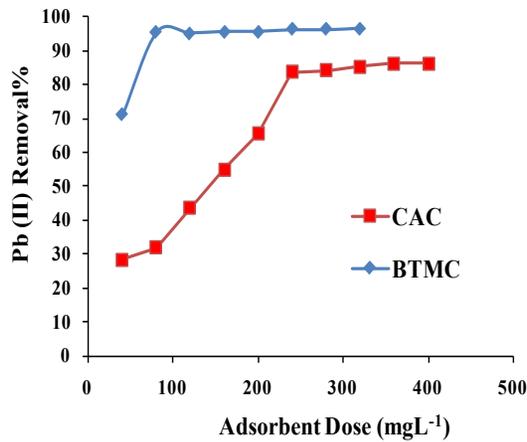


Figure 12. Effect of the adsorbent dose on the removal Pb (II) ions from lead battery wastewater (Con: 10 mg L⁻¹, pH: 5).

has the potential for repeated use and recovery of Pb (II) ions. A similar observation was noted on grapefruit peel [28]

3.10 Removal of Pb (II) from Lead Battery Wastewater

Batch experimental studies with lead battery wastewater is done to know the applicability of adsorbent. The composition of battery wastewater is Lead=10,270 mgL⁻¹, Cadmium=2180 mgL⁻¹, Chloride = 2 009 mgL⁻¹,

pH =5.5. The wastewater was diluted and initial concentration is maintained at 10 mgL⁻¹.

Figure 12 shows the effect of adsorbent dosage on the removal of Pb (II) from wastewater. A minimum dosage of 80 mg BTMC and 240 mg of CAC is essential for the maximum removal of 95 % and 84 % of Pb (II) ions from lead battery wastewater. Hence BTMC can be noted as an efficient adsorbent to remove lead from wastewater due to its moderate ion- exchange property.

4. CONCLUSIONS

Activated carbon was produced from Mangosteen shell and characterized and adsorption capacity of the carbon was analyzed by carrying out adsorption study. Freundlich, Langmuir and Tempkin model are confirmed. The process followed second-order rate. Excellent regeneration capacity was noted for BTMC. The practical utility of carbon was tested using lead battery wastewater. Experimental results also showed that the prepared activated carbon can be used as an effective adsorbent which contributes satisfactory elimination of Pb(II) from aqueous solution. Efficiency and economic availability are the two major advantages of using BTMC as an adsorbent.

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