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# Uptake of Pb(II) ion From Aqueous Solution Using Silk Cotton Hull Carbon: An Agricultural Waste Biomass

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**Abstract**: Activated carbon prepared from silk cotton hull (SCH) was used for the adsorptive removal of Pb(II) ion from aqueous solution. The raw material used for the preparation of activated carbon is the waste of agricultural product; the production of this carbon is expected to be economically feasible. Parameters such as agitation time, metal ion concentration, adsorbent dose, *p*H and Particle size were studied. Adsorption equilibrium was reached within 80 min for 10, 20, 30 and 40mg/l of Pb(II) ion with 50mg of carbon per ml of solution. Adsorption parameters were determined using both Langmuir and Freundlich isotherm models. The adsorption efficiency reached 100% for 20, 30 and 40mg/l of Pb(II) ion with 120, 140 and 150mg of carbon. Pb(II) ion removal increased as the *p*H increased from 2 to 5 and remains constant up to *p*H 10. Desorption studies were also carried out with dilute hydrochloric acid to know the mechanism of adsorption. Quantitative desorption of Pb(II) ion from carbon indicates that adsorption of metal ion is by ion-exchange. Efficiency of the adsorption of SCH was also studied with Pb containing industrial wastewater by varying *p*H and carbon concentration.

**Keywords:** Silk cotton waste, Adsorption, *p*H, Desorption, Metal removal

## Introduction

The standards imposed on industries that discharge heavy metals in their waste are continually lightened due to increasing industrial production and updated knowledge regarding the toxicities of heavy metals that enter the human food chain after accumulating in plants and animals[1].

Acute poisoning from contamination of humans and animals occurs occasionally from industrial or agricultural accidents, but many of today's problems are the result of long-term exposure to heavy metals as environmental contaminants. Since medieval times lead has been used in piping, building materials, solders, paint, type metal, ammunition and castings. In more recent times, lead is introduced into natural water from a variety of sources such as storage batteries, lead smelting, tetraethyl-lead manufacturing, and the mining, plating, ammunition, ceramic and glass industries [2, 3].

Lead contamination of drinking water occurs as a result of corrosion and leaching from lead pipes and Pb/Sn soldered joints associated with the copper service lines commonly used in household plumbing[4]. Lead poisoning in humans causes severe damage to the kidney, nervous system, reproductive system, liver and brain, and causes sickness or death[2]. Severe exposure to lead has been associated with sterility, abortion, stillbirth and neonatal deaths[5].

The permissible limit for lead in drinking water[6] is 0.05mgdm$^{-3}$. The permissible limit (mgdm$^{-3}$) for Pb(II) ion in wastewater, given by the Environmental Protection Agency[7] (EPA), is 0.05mgdm$^{-3}$ and that of the Indian Standards Institution[8] (BIS) is 0.1mgdm$^{-3}$

A number of technologies have been developed over the years to remove toxic metal ions from water[9]. Such methods include chemical precipitation, electro deposition, ultra filtration, ion exchange, activated carbon adsorption and biological processes[6]. Adsorption, compared with other methods, appears to be an attractive process in view of its efficiency and the ease with which it can be applied in the treatment of wastewater containing heavy metals. The use of activated carbon for the adsorption of heavy metal was first reported in 1929 by Watonabe and Ogawa [10, 11]. This adsorption by activated carbon appears to be particularly competitive and effective in the removal of heavy metals at trace quantities [12]. Other adsorbents such as flyash[13], red mud[14], blast furnace sludege[15], waste Fe(II)/Cr(III) hydroxide [16], biogas residual slurry [17], olive mill product [18] and bentonite[19] are also used. Heavy metal removal using naturally occurring materials like coal, peat moss, vermiculite [7, 20, 21] and inorganic materials such as natural clay [22] and activated clay [23] are known.

Pb(II) ion removal using activated carbon prepared from silk cotton hull carbon can thus be viewed as an effective alternative to conventional methods in wastewater treatment. In the present study, the efficiency of (SCH) for Pb(II) ion removal was assessed. The effect of contact time, adsorbent dosage, *p*H, adsorbate concentration and competing ions on Pb(II) ion removal and desorption were also studied.

## Experimental

### *Adsorbent*

The silk cotton hull was cut into small pieces, dried in sunlight, then 60˚C for 24 hours in hot air oven. The dried material is subjected for acid treatment (ratio 1:1) and kept at room temperature overnight. Then it was washed with doubled distilled water to remove the excess acid and kept in hot air oven at 110˚C for 12 hours. Then it was taken in an iron vessel in muffle furnace and the temperature was gradually raised to 550˚C for an hour, ground well by using ball mill and then sieved into particle size of 250,150 and 100 BSS mesh numbers. The characterization of SCH was carried out and the results were tabulated in Table 1.

**Table 1**. Characteristics of Activated Carbon

| S.No | Parameters | Obtained Result |
|---|---|---|
| 1. | *p*H solution | 6.7 |
| 2. | Moisture content (%) | 2.4 |
| 3. | Ash content (%) | 1.856 |
| 4. | Decolorizing power (mg/g) | 22.5 |
| 5. | Ion-exchange capacity (milliequi/g) | 0.0415 |
| 6. | Surface area ($m^2/g$) | 158-228 |
| 7. | Apparent Density (g/l) | 0.42 |
| 8. | Particle size (µm) | 125 |
| 9. | Volatile matter (%) | 12.0 |
| 10. | Fixed carbon (%) | 86.0 |
| 11. | Calcium (%) | 16.0 |
| 12. | Sodium (mg/g) | 7.0 |
| 13. | Potassium (mg/g) | 13.0 |
| 14. | Water soluble matter (%) | 2.0 |
| 15 | HCl soluble matter (0.25N) (%) | 7.0 |
| 16. | Phenol number, mg | 11.2 |

The adsorbent SCH sieved particle size of 250BSS mesh number was magnified by Scanning Electron Microscope (SEM) studies by using JOEL JSM 8404 Scanning microscope as shown in the Figure 1. The X-ray Diffraction studies of SCH were carried out using Rotoflux X-ray Diffractometer 20KW/20A, Model 10.61 with a Microprocessor recorder. The XRD pattern of the SCH is shown in the Figure 2. The morphological and XRD studies clearly revealed that the adsorbent is amorphous and highly porous in nature. From the SEM analysis it was found that there were holes and cave type openings on the surface of the adsorbent, which would have more surface area available for adsorption.

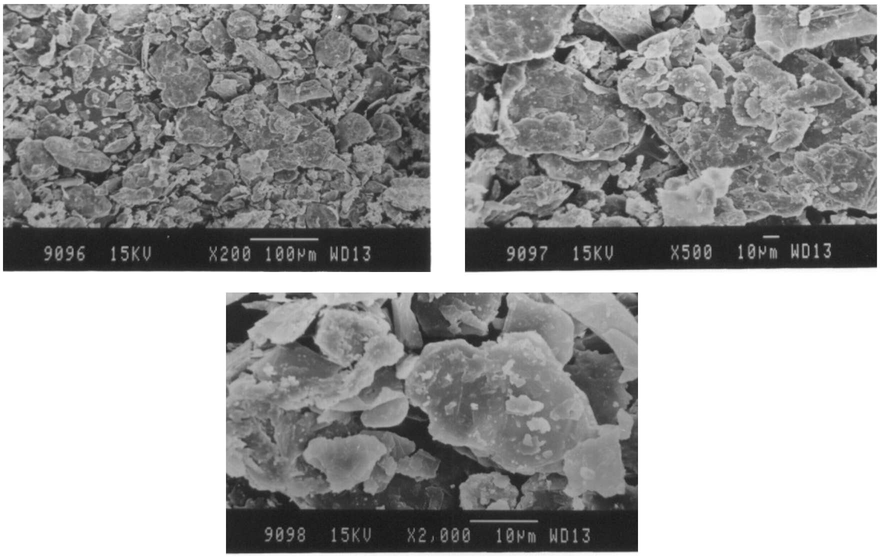


**Figure 1.** SEM photograph of Silk cotton hull activated carbon (SCH) at various magnifications (x200, x500 and x2000)

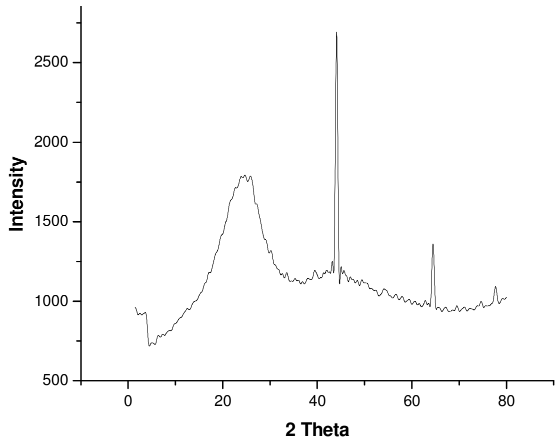


**Figure 2.** X-ray Diffraction pattern for SCH

## Batch Adsorption Experiments

### *Adsorbate*

1000mg/l of Pb(II) ion was prepared by dissolving 1.599g of $PbNO_3$ in acidified double distilled water to prevent hydrolysis formation and made up to 1000ml. The stock solution was diluted with distilled water to obtain required standard solution. The effect of agitation time was studied by shaking solution at 170rpm, using 50ml of 10, 20, 30 and 40 mg/l solution of Pb(II) ion containing 50mg of the adsorbent and adjusted to an initial *p*H of 5.0. After agitation, the adsorbate and adsorbent were separated by centrifugation and supernatant was used to estimate Pb(II) ion by spectrophotometer using PAR reagent (4,2-pyridylazo resorcinol) [26].

The effect of adsorbent dose on Pb(II) ion removal was studied by agitating 50ml of 20, 30, 40mg/l of Pb(II) ion, adjusted to an initial *p*H of 5.0ml and containing different doses of adsorbent (10-150mg/50ml) for a time period greater then their respective equilibrium times. The effects of *p*H on Pb(II) ion removal was studied by using 50ml of 20, 30, 40mg/l solution of Pb(II) ion, adjusted to an initial *p*H of 2, 3, 5, 4, 6, 8, 9 and 10 mixed with 50mg of carbon and agitated for 90min. The effect of adsorbate concentration on Pb(II) ion was studied by agitating 50ml of different concentrations (5-65mg/l) of Pb(II) ion solution with 50mg of adsorbent for a time period greater than the equilibrium time at 170rpm at *p*H of 5.0.

### *Desorption Studies*

After adsorption experiments with a 20mg/l solution of Pb(II) ion and 50mg of carbon, the Pb(II) ion laden carbon was separated out by filtration and the filtrate was discarded. The carbon was given a gentle wash with double-distilled water to remove any unadsorbed Pb(II) ion molecules entrapped between carbon particles and Pb(II) ion on the carbon surface. Desorption studies were carried out using those carbon samples agitated with 50ml of HCI of various strengths (0.25 to 2M). After desorption experiments samples were estimated for Pb(II) ion by spectrophotometer as before.

## Removal of Pb(II) ion from radiator-manufacturing industry wastewater

The Pb(II) ion-containing wastewater was collected from the radiator-manufacturing industry and characterized. This was used to study the effect of *p*H and adsorbent concentration. To show the effect of carbon concentration, the initial *p*H of Pb(II) ion wastewater was adjusted to 3.0, different concentrations of carbon added, and the mixture agitated for 90min. The samples were then centrifuged and was analysed as before.

# Results and Discussion

### *Effect of agitation time and initial Pb(II) ion concentration on adsorption*

Figure 3 shows the effect of agitation time on Pb(II) ion adsorption by SCHC. The figure shows that increase in agitation time increased the uptake of lead ions and attained equilibrium in 10, 20, 30 and 40 mg/l Pb(II) ion, respectively. The contact time required for the metal ion is very short. This result is interesting because equilibrium time is one of the important considerations for economical wastewater treatment applications. According to the results, the contact time was fixed at 90 min the rest of the batch experiments to make sure the equilibrium is reached.

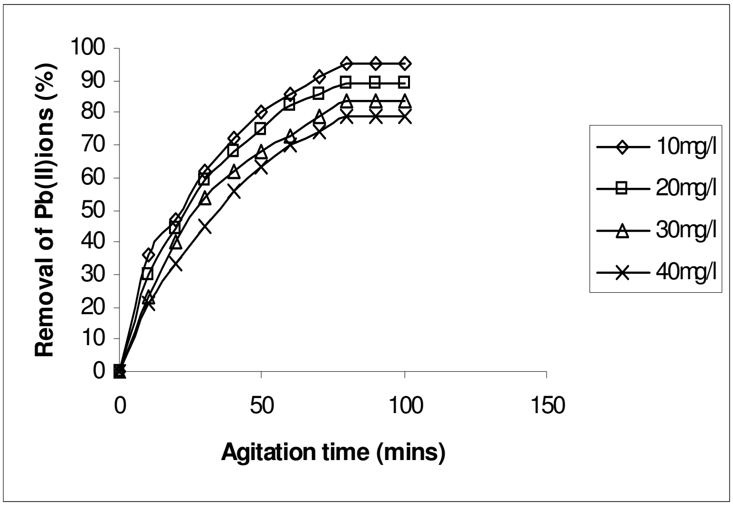


**Figure 3.** Effect of agitation time and initial Pb(II) ion concentration on adsorption of Pb(II) ions on to SCHC

### *Adsorption Kinetics*

The rate constant of Pb(II) ion adsorption on carbon followed the first order rate expression given by Lagergren[24]

$$\text{Log}\,(q_e - q) = \log_{10} q_e \;\; (K_{ad} t / 2.303) \qquad (1)$$

Where q and $q_e$ are amount of metal ion adsorbed per mg of carbon at time t (min) and at equilibrium time, respectively, and $K_{ad}$ is the rate constant of adsorption (1/min). The linear plots of log($q_e$– q) *vs* t for the metal ion studied at different concentration indicate the applicability of Eqn.1. The values of $K_{ad}$, calculated from the slope of linear plots are 3.47, 3.64, 3.36, $3.37 \times 10^{-2}$ for 10, 20, 30, 40mg/l of Pb(II) ion concentration, Kadirvelu and Namasivayam have reported.

*Effect of carbon concentration on metal ion adsorption*

Figure 4 shows the effects of carbon concentration on Pb(II) ion adsorption. The figure shows that the removal of metal ions increased with an increase in carbon concentration and attained complete removal after a particular carbon concentration for the metal ion studied. This was due to the availability of more surface area with higher functional groups at higher carbon concentration [28, 29]. The figure shows that for the removal of Pb(II) ion concentration of 20, 30 and 40mg/l, a maximum carbon dosage of 120, 140 and 150mg/50ml required.

## Adsorption Isotherms

*Langmuir isotherms*

The Langmuir isotherm was applied for adsorption equilibrium represented by the following equation:

$$(C_e / q_e) = (1/Q_ob) + (C_e / Q_o) \qquad (2)$$

Where $C_e$ is the equilibrium concentration (mg/l) and $q_e$ is the amount adsorbed (mg/g) at equilibrium and $Q_o$ and b are Langmuir constants related to adsorption capacity and energy of adsorption, respectively. The Langmuir isotherm model is based upon then values present in the Table 3. From which the plots of $C_e/q_e$ Vs $C_e$ are linear for carbon which show that the adsorption follows the Langmuir isotherm model for Pb(II) ion adsorption. The values of $Q_o$ and b were found to be 43.14 mg/g and 0.01647mg/l, respectively, which were calculated from the slope and intercept of the Langmuir plot.

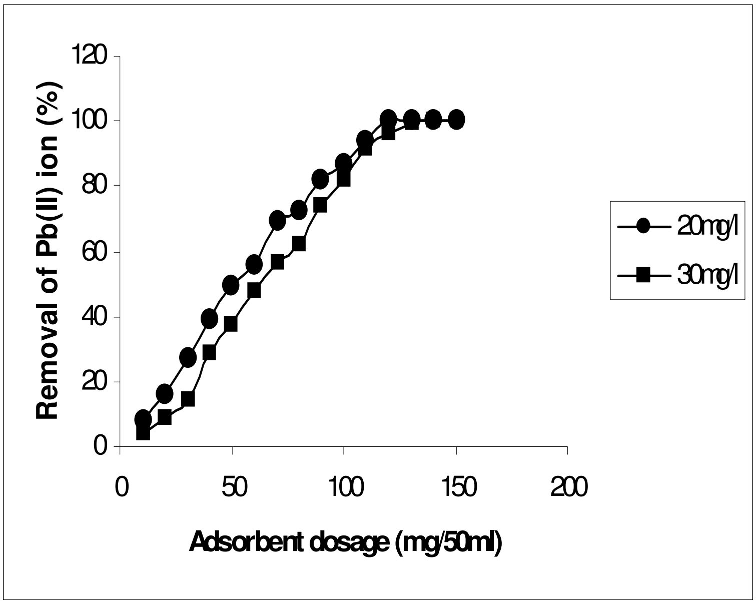


**Figure 4**. Effect of SCHC dosage on the adsorption of Pb(II) ions

**Table 2** Characteristics of Radiator-Manufacturing Industry

| Parameter | | Value |
|---|---|---|
| *p*H | | 3.1 |
| Conductivity | ($mscm^{-1}$) | 15.31 |
| Total dissolved solids | (mg/l) | 6011.00 |
| Total suspended solids | (mg/l) | 84.60 |
| Turbidity | (NTU) | 65.00 |
| COD | (mg/l) | 350.00 |
| Total hardness as $CaCO_3$ | (mg/l) | 7341.00 |
| Sodium | (mg/l) | 285.00 |
| Potassium | (mg/l) | 11.00 |
| Calcium | (mg/l) | 250.00 |
| Lead | (mg/l) | 50.00 |
| Sulfate | (mg/l) | 38.00 |

**Table 3** Data for Langmuir plot for adsorption of Pb(II) ions
Carbon dosage - 50mg/50ml; *p*H- 5.0 $\pm$ 0.2 , Agitation time - 90min

| Conc. of Pb(II) ions (mg/l) | $C_e$ (mg/l) | $q_e$ (mg/l) | $C_e/q_e$ (mg/l) |
|---|---|---|---|
| 10 | 0.41 | 9.5 | 0.0441 |
| 15 | 1.17 | 13.8 | 0.848 |
| 20 | 2.08 | 17.8 | 0.1169 |
| 25 | 3.58 | 21.2 | 0.1684 |
| 30 | 4.92 | 24.9 | 0.1975 |
| 35 | 6.65 | 28.0 | 0.2375 |
| 40 | 8.57 | 31.2 | 0.2746 |
| 45 | 10.56 | 32.4 | 0.3259 |
| 50 | 16.75 | 33.5 | 0.4458 |
| 55 | 16.92 | 37.9 | 0.5000 |
| 60 | 21.07 | 38.4 | 0.5486 |
| 65 | 23.62 | 40.9 | 0.5768 |

Langmuir constants: $Q_0$ (mg/g) - 43.10; b (l/mg) - 0.316

*Freundlich isotherm*

The Freundlich adsorption isotherm is plotted from the values present in the Table 4, it is a linear, from which the adsorption of the metal ion Pb(II) ion by carbon.

$$q_e = K_f C_e^{1/n} \quad (3)$$

Rearranging Eqn.3 gives:

$$\mathrm{Log}_{10} X/m = \log K_f + 1/n \log_{10} C_e \quad (4)$$

Where $C_e$ is the equilibrium concentration (mg/l) and X/m is the amount adsorbed (mg/g) at equilibrium time and $K_f$ and n are Freundlich constants, n giving an indication of the favorability and $K_f$, the capacity of the adsorbent [31]. A linear plot of metal ions onto carbon follows the Freundlich isotherms model, indicating that the average energy of adsorption decrease with increasing adsorption density. The values of n and $K_f$ were found to be 13.44 and 2.78.

30mg/l of Pb(II) ion solution, which were calculated from the slope and intercept of the plots respectively. The values of n and $K_y$ were found to be 7.0 and 38.41 for 30mg/l of Pb(II) ion solution by carbon prepared from Eichhornia. 80.2 and 74.8 $K_f$ and 2.37 and 3.01 for 50 and 100 mg/l Pb(II) ion of coir pith carbon.

**Table 4**. Data for Freundlich plot for adsorption of Pb(II) ions
Carbon dosage - 50mg/50ml; *p*H- 5.0 ± 0.2; Agitation time - 90min

| Conc. of Pb(II) ions (mg/l) | % Removal | Log $c_e$ | Log x/m |
|---|---|---|---|
| 10 | 95 | 0.0685 | 0.9777 |
| 15 | 92 | 0.3182 | 1.139 |
| 20 | 89 | 0.5538 | 1.250 |
| 25 | 85 | 0.6919 | 1.327 |
| 30 | 83 | 0.8228 | 1.396 |
| 35 | 80 | 0.9324 | 1.447 |
| 40 | 78 | 1.0236 | 1.494 |
| 45 | 72 | 1.2240 | 1.510 |
| 50 | 67 | 1.2284 | 1.525 |
| 55 | 69 | 1.3236 | 1.584 |
| 60 | 64 | 1.3732 | 1.612 |

Freundlich constants: $K_f$ - 2.783; n – 13.44

### *Effect of pH on Pb(II) ion removal*

Figure 5 shows that increasing the *p*H increases the removal of Pb(II) ion both in the presence and absence of carbon. One of the conventional methods of removing heavy metals from aqueous solution is the precipitation of metal hydroxides using an alkali [32]. This method has however some demerits in that the complete removal of metals is not possible due to the solubility product of metal hydroxide. Hence, comparison is made between carbon adsorption and precipitation as metal hydroxide. Maximum adsorption occurs below the *p*H of metal hydroxide precipitation. The removal of Pb(II) ion by SCHC was observed over a *p*H range of 2–10. The rate of adsorption increased from 2–5 and remains static upto 6-10. The ion exchange mechanism between $H^+$ ions at the SCHC surface and metal ion may follow these two reactions.

$$2S\text{-}COH + M^+ \leftrightarrow S\text{-}(CO)_2 M^+ + 2H^+ \quad (5)$$

$$S\text{-}COH_2^{2+} + M^+ \leftrightarrow S\text{-}COM^{2+} + H^+ \quad (6)$$

Where, S is the SCHC surface.

In the absence of carbon, precipitation of metal hydroxide begins at *p*H 5.5. In the presence of carbon, maximum adsorption was observed in the *p*H range of 2.0-5.0. Above *p*H 5.0, Pb(II) ion starts precipitating as $Pb(OH)_2$, and this complicates the removal of Pb(II) ion by adsorption. At higher *p*H (>6.2), precipitation of Pb(II) ion takes place. Hence it was decided to maintain the *p*H at 5.0 for all other studies.

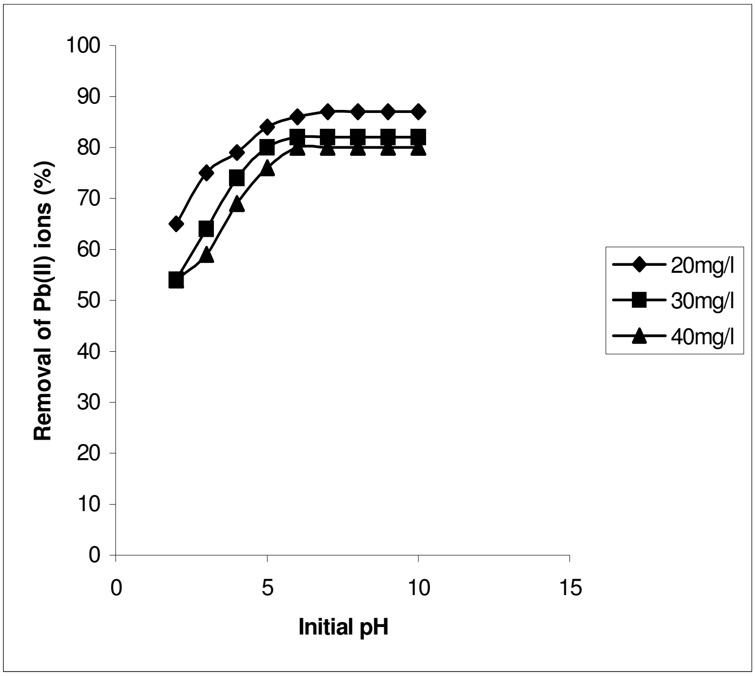


**Figure 5**. Effect of initial *p*H on the removal of Pb(II) ions from aqueous solution by SCH carbon

*Desorption studies*

A suitable method of desorption is needed for the recovery of precious metals, and regeneration of carbon and elucidate the adsorption mechanism. The effect of HCl concentration on the desorption of Pb(II) ion was presented in Figure 6. The quantitative desorption of Pb(II) ion was obtained 0.2M HCl. The relatively in expensive HCl eluted almost all the bound Pb(II) ion from the carbon. In contrast $NaNO_3$, NaCl, $Na_2SO_4$, $HNO_3$, and $H_2SO_4$ were not efficient in the desorption of Pb(II) ions. Similar results were obtained in the case of desorption of Pb(II) ion from activated carbon prepared from coir pith Eichrronia and activated carbon cloths [33]. The hydrogen ions from HCl easily displace Pb(II) ion ions adsorbed to carbon during the desorption process. Quantitative desorption of Pb(II) ion clearly shows that adsorption of metal ion is by ion-exchange process.

*The removal of Pb(II) ion from industrial wastewater using SCHC*

The physico-chemical characteristics of radiator-manufacturing industry wastewater are shown in Table 2. The removal of Pb(II) ion from industrial wastewater depends upon its composition, nature and *p*H. The increase in carbon concentration increases the adsorption of Pb(II) ion from industrial wastewater (Figure 7). The amount of carbon required was found to be higher because of the presence of other elements in the wastewater. The amount of carbon required was found to be 150mg/50ml of wastewater containing 10mg of Pb(II) ion. The experimental result of the removal of Pb(II) ion from industrial wastewater confirms the validity of the result obtained in the batch mode studies, *i.e.* carbon can be used effectively for the removal of Pb(II) ion from industrial wastewater.

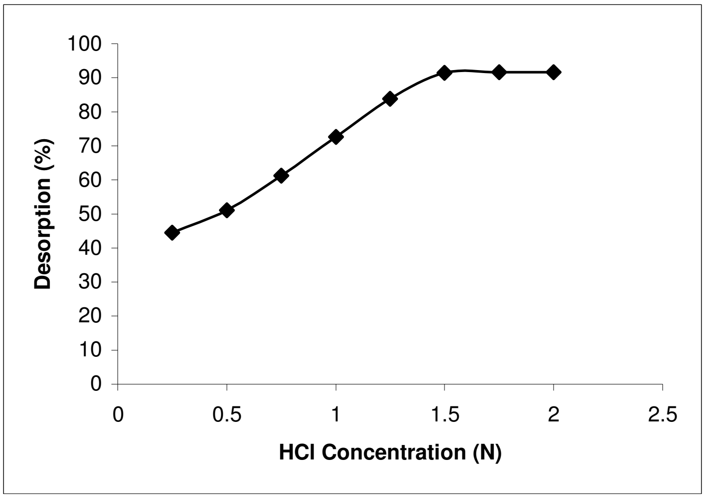


**Figure 6**. Desorption of Pb(II) ions from SCH carbon

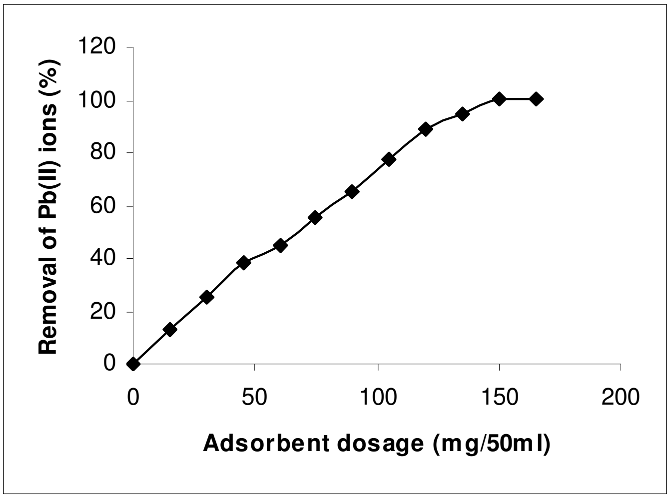


**Figure 7**. Effect of SCHC dosage on the adsorption of Pb(II) ions from radiator manufacturing industry waste water

## Conclusion

The objective of this work was to assess the adsorption of Pb(II) ion on SCHC. It is concluded that the activated carbon prepared from silk cotton hull is efficient in the adsorption of Pb(II) ion from aqueous solution. In batch mode studies, the adsorption was depend on solution *p*H, initial Pb(II) ion concentration and carbon dosage. Adsorption of Pb(II) ion onto carbon followed both Langmuir and Freundlich isotherms models. The maximum adsorption capacity ($Q_0$) was calculated from Langmuir isotherm it was 43.2mg/g of carbon at initial *p*H of 5.0 and 30°C for the particle size of 250BSS mesh number. Kinetic data would be useful for the fabrication and designing of wastewater treatment plants. Removal of lead from radiator-manufacturing industry wastewater confirms the validity of results obtained in batch mode studies. As the silk cotton hull in a waste material, it is an inexpensive source and therefore may have the advantage of economic viability.

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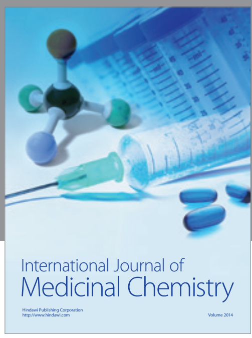

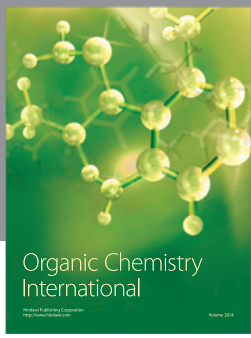

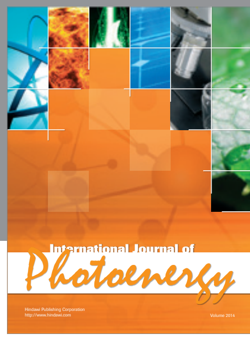

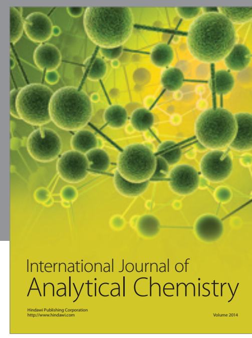

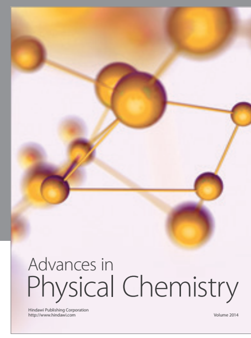

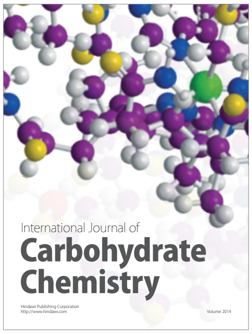


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