



PREPARATION, CHARACTERIZATION AND SUITABILITY ANALYSIS OF ARECA HUSK ACTIVATED CARBON FOR REMOVAL OF LEAD FROM AQUEOUS SOLUTION

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ABSTRACT

Heavy metal contamination is being reported in alarmingly high amounts in recent past. This study was carried out to analyze the utilization of Areca Husk carbon as an adsorbent for the removal of Lead from aqueous solution and to explore its possibility as a low cost adsorbent. Batch adsorption experiments were conducted to study the effect of adsorbent dosage, contact time, initial lead concentration and pH for the removal of lead. The structural and morphological characteristics of the activated carbon were characterized by FTIR and SEM studies respectively. XRD analysis was done to identify crystalline nature of the adsorbent. The results have established that the carbon particles possess a good potential to be used as an adsorbent for the removal of lead from waste water. This Batch experiment was performed in the laboratory scale. The experiment achieved upto 93% removal efficiency.

Keywords—Adsorption, SEM, FTIR, XRD, Areca husk carbon, removal efficiency, optimum conditions

I. INTRODUCTION

Since a long time, major population of India depends on groundwater as a source of drinking water. Attributing to its purity, a major section of rural Indian population relies on groundwater for their agriculture and domestic purposes. A vast majority of groundwater quality problems present today are caused by contamination and by over-exploitation, or by combination of both. Rapid urbanization and industrialization in India has resulted in steep increase of generation of wastes. It is quiet disturbing that heavy metal contamination of groundwater mostly goes unnoticed and remains hidden from the attention of public. Several metals are present in groundwater that play significant roles in the human body provided their concentration remains within the range specified by the WHO (Mridul Buragohain et al. 2009). Metals in water supply may occur as a result of the contamination or may be present naturally. Trace amounts of metals are common in water, and these are normally not harmful to human health. The main threats to human health from heavy metals are associated with exposure to lead, arsenic, mercury and cadmium (Vaishaly A.G et al. 2014).

The lead contamination in ground water and surface water in India has been studied. Ground water contamination with excessive lead content has been reported in Kamrup and Dhemaji districts of Assam (Mridul Buragohain et al. 2009), Uttarakhand (Sandeep Gaur et al. 2011), Chhattisgarh (K.S. Patel et al. 2011) and Bhopal. Alarmingly higher level of lead was present in ground water of Kalamassery and Ambalamugal regions in Cochin, Kerala (CPCB survey, 2007). Presence of excessive amount of lead in drinking water is life threatening. It accumulates in food chain and cause its imbalance and also harmful to flora and fauna even in smallest concentrations. It is a potential threat to the Normal life of people especially when mostly people rely on groundwater as the drinking water source. The aim of this work is to determine the feasibility of locally available and low cost adsorbent material in treating the lead contaminated water. If successful, it can be adopted as a decentralized method of water treatment in households. As the adsorbent is cost effective and easily available, it will be comparatively easy for the people to adopt this technology.



II. METHODOLOGY

A. Preparation of the Areca Husk adsorbent

The areca husk fibres were cut into small pieces, dried in sunlight until the moisture was partially evaporated and was further dried in a hot air oven at 65°C for 24 hours until the residual moisture was completely evaporated. The dried material was mixed with concentrated H₂SO₄ (ratio 1:2 by mass) and kept at room temperature overnight. The activation process of AHC by sulphuric acid treatment leads to the creation of a carbonaceous material surface with the introduction of macro, micro and meso pores. It was then washed with double distilled water for more than 5 times to remove the excess acid if any, and kept in hot air oven at 110°C for 12 hours. It was carefully taken in an iron vessel, covered with a tight fitting lid to avoid contact with atmospheric air, and was placed in a muffle furnace and the temperature was gradually raised up to 550°C and kept for an hour. After cooling, the material was taken out and ground well by using mixer grinder and taken for ash removal (A. Basker et al. 2014). The adsorbent prepared was suspended in 1.5g/30ml and agitated for 2 hours in an ultrasound bath. The suspension was pumping filtered and dried in oven to remove moisture content. The resulting dried activated carbon was stored in an air tight container for further use.

B. Preparation of Lead Nitrate solutions

Lead(II) Nitrate (Pb(NO₃)₂) supplied by Nice Chemicals was used as an adsorbate and was not purified prior to use. Lead Nitrate has a molecular weight of 331.2 g/mol. Distilled water was employed for preparing all the solutions and reagents. The Lead stock solutions were prepared by dissolving accurately weighed Lead Nitrate in distilled water upto the concentration of 1000 mg/L. The experimental solutions were obtained by diluting the Lead stock solutions in accurate proportions to different initial concentrations. All the experimental solutions were of 250 ml in volume.

C. Adsorption studies (Batch experiments)

Different samples were prepared by varying adsorbent dosage and adsorbate concentration. The mixture was stirred on a rotary shaker at 150 rpm and at room temperature. Then samples were filtered using 0.2 micron syringe filter, and final absorbance was checked. Residual Lead content was measured using an Atomic Absorption Spectroscope (absorption at 283.3 nm). Duplicate trials were performed and the average value was taken as the final reading. The experiment was repeated by varying pH and contact time. The quantity of lead adsorbed was calculated and the efficiencies were found out.

III. RESULTS AND DISCUSSION

A. Characteristics of Areca Husk Carbon

The characterization of the material gives a rough idea about the performance of the adsorbent. WHO lays out a minimum requirement for the adsorbent quality, the same can be verified by analyzing the properties. The Areca Husk Carbon had about 2.31 % of moisture is contained in it. It will not influence the adsorption capacity of the activated carbon. High moisture content will increase the load on activated carbon hence reducing the power of adsorption. The fixed carbon content was found to be 76.2%. A significant ash content of 3.88% was noted and hence the ash removal was necessary before the material is stored for further use.

Table 1: Characteristics of areca husk carbon

Moisture content, %	2.31
Ash content, %	3.88
Volatile matter, %	8.4
Fixed Carbon, %	76.2
pH	6.4
Bulk density, g/ml	0.42

Characteristics of the Areca Husk carbon analyzed are as in Table 1. The impurities in the adsorbent affect the quality of the treated water. The prepared areca husk carbon has been refined to the purest form possible by ash removal and subsequent washing and drying. The addition of acid in the carbon structure makes the material acidic (pH 6.4) and the ion exchange capacity increases.

B. SEM morphology

It has been used to determine the porous space available, surface morphology, particle shape and nature of the surface of the adsorbent. Various magnifications of the SEM images reveal the surface characteristics and the pore structure of the adsorbent. The SEM images are shown in Fig. 1. SEM images revealed that the AHC has a rough and uneven surface with more pores, which resembles a cave like structure. Various shapes of the pores provide a wide range of possibilities to the discrete particle to get accommodated. The selectivity and efficiency of adsorption depend upon the shape, nature and distribution of the pores in the adsorbent surface. The SEM images of AHC adsorbed with Lead ions are in Fig 1 (right). It is evident from the images that the surface of the adsorbent is saturated with the lead ions adsorbed in it. The surface modification of the adsorbent was identified to be a monolayer formation.

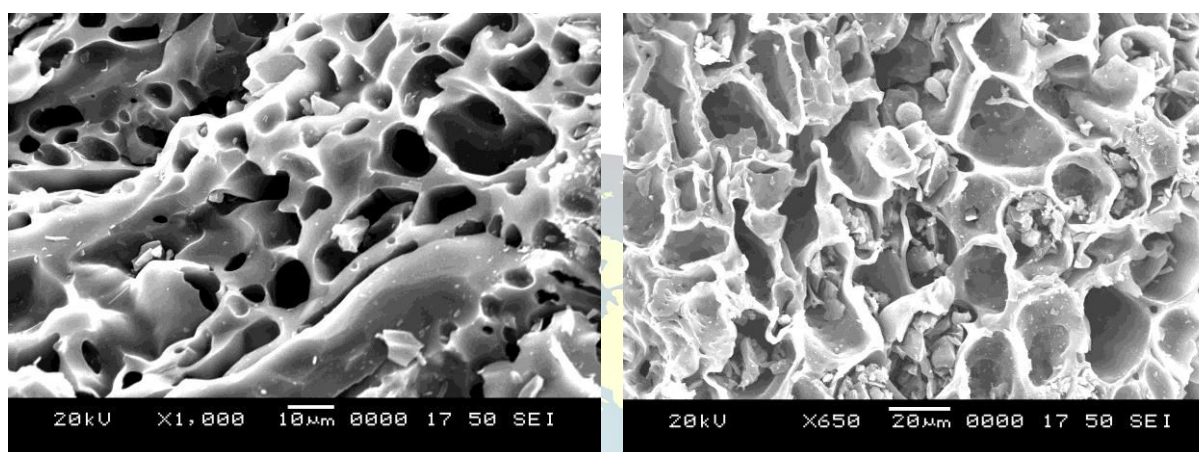


Figure 1: SEM image of the adsorbent before and after adsorption (left to right)

C. Determination of functional group

The FTIR spectrum of AHC before adsorption was detected in the range of 400 to 4000 cm^{-1} (Adina Elena Segneanu et al. 2012) was presented in Fig 2. The band observed at 3427.32 cm^{-1} was due to the presence of (O-H) and also due to the presence of amines (N-H). The absorption band at 3094.12 cm^{-1} and 2879.22 cm^{-1} is due to the stretching vibrations of (C-H) bonds in aromatic, alkane and aldehyde groups where carbon is bonded with hydrogen bonds. Adsorption bands at 2879.22 and 2389.12 cm^{-1} corresponds to the presence of carboxylic acid functional groups. The band at 1574.47 cm^{-1} shows the aromatic (C=C) and Nitroso (N=O) functional groups. The band at 1567.34 cm^{-1} may be attributed to the aromatic (C=C) stretching vibration. At 1278.94 cm^{-1} , the band is highly intense (C-O) and is related to the (N-O) stretching vibration of the aromatic amine oxide groups. The dip shown at 1173.4 cm^{-1} corresponds to the Alcohol (C-O), Ether (C-O), Acids (C-O) and Amines (C-N). The band corresponding to 849.43 cm^{-1} and 817.42 cm^{-1} in the fingerprint area indicates alkane functional group (C-C). All other peaks below this value correspond to amines and esters. It is evident more functional groups are involved in the removal of lead from aqueous solution.

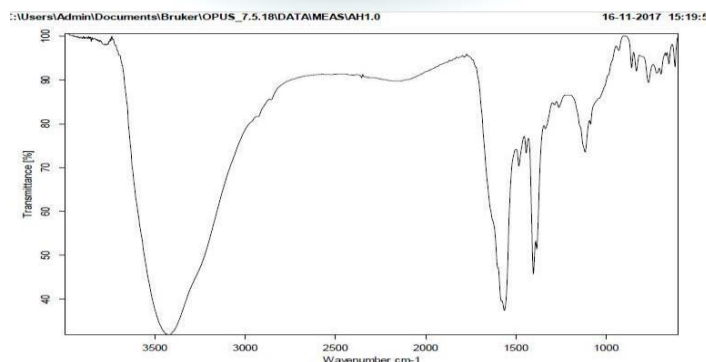


Figure 2: FTIR spectra of fresh AHC

D. X-ray Diffraction Studies

Crystalline or amorphous nature of the material is determined through X-ray diffraction. In X-ray diffraction, crystalline materials exhibit well defined peaks while non-crystalline or amorphous materials show shallow or smooth peaks (K. Ramesh et al. 2014). The XRD pattern of unloaded AHC is amorphous in nature as the XRD output does not give any sharp peak as shown in the Fig. 3.

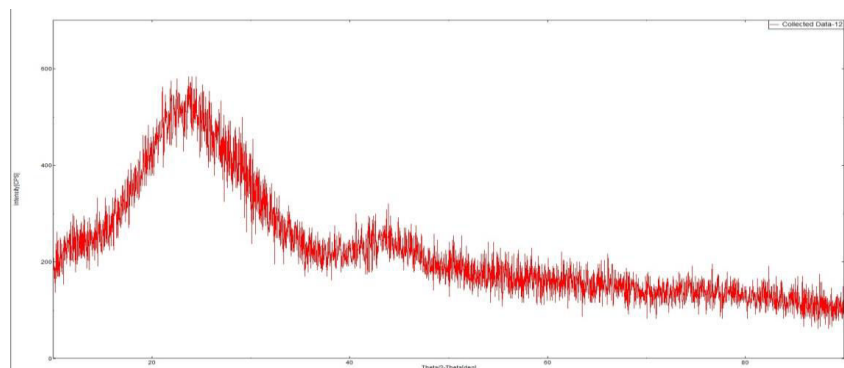


Figure 3: XRD pattern of fresh AHC

E. Effect of amount of adsorbate to adsorbent ratio

Initial concentration of lead was varied from 1mg/L to 5mg/L fixing the adsorbent dosage at 500 mg. pH was kept slightly acidic and contact time of 90 minutes was given. Figure 4 shows the trend of removal efficiency of lead with amount of adsorbate to adsorbent ratio (mg/gm). The initial rise in the graph could be due to the reason that at higher concentration there is higher driving force for mass transfer. With increase in metal ion concentration the active sites of adsorbent are surrounded by higher number of metal ion molecules which leads to more efficient adsorption within a period of time. As the concentration of metals increases, the adsorption by the biochar also increases till a point, indicates that the material used has a good adsorption capacity for this metal. The experiment was repeated with different values of adsorbent mass and best fit with the trend line was found to be that of adsorbate mass of 500 mg.

The equation of the trend line explains the effect of any values of amount of adsorbate to adsorbent ratio (mg/gm) on removal efficiency of lead. The trendline indicates a slightly decreasing trend towards the concentration near 5mg/L giving the optimum at 4 mg/L (ratio= 2). This may be because of the start of the desorption process. The R^2 value of 0.8773 indicates a good fit of trendline to the experimental data.

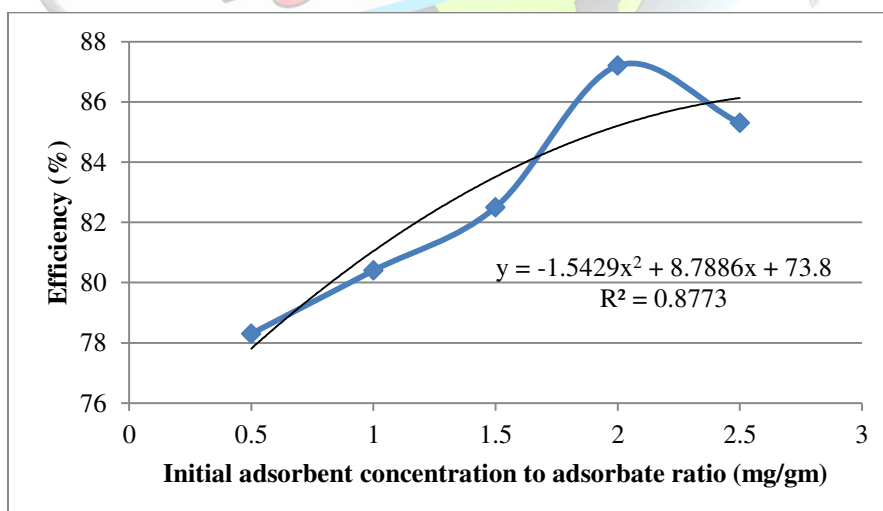


Figure 4: Effect of amount of adsorbate to adsorbent ratio on removal of lead using AHC

F. Effect of pH

Figure 5 shows the effect of pH on lead adsorption by the areca husk carbon. The pH of the solution was varied from 2 to 14 with an initial adsorbate concentration of 4mg/L, adsorbent dosage of 500mg and a contact time of 90 minutes. The rotation speed was maintained as 150 rpm for all runs. Graph shows that at lower pH value, the removal of lead was very low. It increased with increase in pH. The lesser value of pH in the acidic range may be because of the H^+ ion concentration with the metal ions for the binding sites. The optimum value was observed at a pH of 5, after which the efficiency decreased. This may be due to the formation of soluble hydroxyl complexes. The equation of the trendline explains the effect of any values of pH on removal efficiency of lead. The R^2 value of 0.8993 indicates a good fit of trendline to the experimental data obtained.

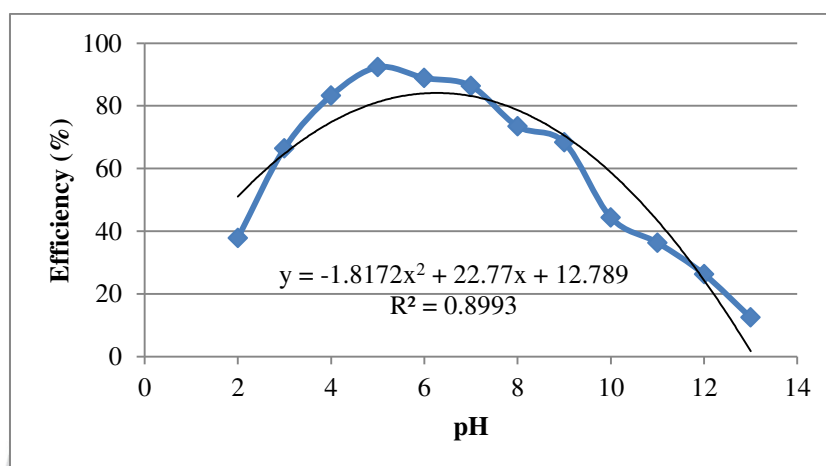


Figure 5: Effect of pH on removal of lead using AHC

G. Effect of Contact time

Batch study was conducted with four different contact time varying from 30 minutes to 150 minutes. The change in adsorption efficiency with contact time is shown in the Figure 6. The results indicate that the optimum removal efficiency is achieved at a contact time of 90 minutes. This may be due to the saturation of active sites on the adsorbent surface and the system reached equilibrium. The equation of the trend line explains the effect of any values of contact time on removal efficiency of lead. The R^2 value of 0.9086 Indicates a good fit of trend line to the experimental data obtained.

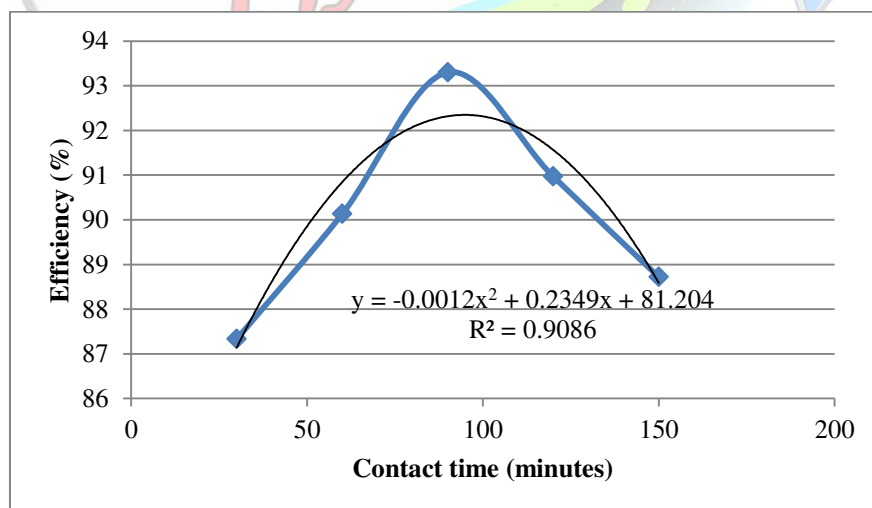


Figure 6: Effect of contact time on removal of lead using AHC



IV. CONCLUSION

The efficiency of adsorption of Lead is nearly 93%. The results of the present investigation reveals that the activated carbon prepared from the Areca Husk Fibre can be used as a promising adsorbent for the removal of lead ions from an aqueous solution.

Table 2: Optimum values of each parameter

Parameter	Adsorbate to adsorbent ratio (mg/gm)	Adsorbate concentration (mg/ L)	Adsorbent dosage (mg)	pH	Contact time (minutes)
Optimum Value	2	4	500	5	90

REFERENCES

- [1] A.Basker, P.S.Syed Shabudeen, S.Daniel and P.Vignesh Kumar, "Adsorptive Removal of Malachite Green from Aqueous Solution using Areca Husk Carbon," *RASAYAN J.Chem*, 2014, 1-15,
- [2] A. Basker and P.S. Syed Shabudeen, "Preparation and Characterization of Activated Carbon from Areca Husk Fibre for the Removal of Dyes from Textile Industrial Effluent," *International Journal of Research in Sciences*, 2014, 38-46
- [3] K.S. Patel, K. Shrivastava, P. Hoffmann & N. Jakubowski, "A survey of lead pollution in Chhattisgarh State, central India," *Environmental Geochemistry and Health* (2006) 28:11-17
- [4] Mridul Buragohain, Bhabajit Bhuyan and Hari Prasad Sarma, "Seasonal variations of lead, arsenic, cadmium and aluminium contamination of groundwater in Dhemaji district, Assam, India," *Environ Monit Assess*, 2010, 170:345-351
- [5] Sandeep Gaur, Nutan Singh, Sandeep Saxena, "Status of lead present in ground drinking water samples of Uttarakhand (Garhwal Region) in India," *Asian Journal of Biomedical and Pharmaceutical Sciences 1 (1) 2011*, 32-38
- [6] Adina Elena Segneanu, Ioan Gozescu, Anamaria Dabici, Paula Sfirloaga and Zoltan Szabadai(2012), "Organic Compounds FT-IR Spectroscopy, Macro to Nano Spectroscopy," Dr. Jamal Uddin (Ed.), ISBN: 978-953-51-0664-7.
- [7] K. Ramesh, and V. Nandhakumar, "IR, XRD and SEM Studies on the Adsorption of Methylene Blue Dye onto Microwave Assisted $ZnCl_2$ Activated Carbon Prepared from Delonix Regia Pods," *International Journal of Current Research in Chemistry and Pharmaceutical Sciences*, 1(4), 2014, 15-19.
- [8] Dixit S and Tiwari S, "Impact Assessment of Heavy Metal Pollution of ShahpuraLake, Bhopal, India," *Int. J. Environ. Res.*, 2008, 2(1): 37-42
- [9] R. Srikanth, A. Madhumohan Rao, CH. Shravan Kumar and Anees Khanum, "Lead, Cadmium, Nickel and Zinc Contamination of Ground Water around Hussain Sagar Lake, Hyderabad, India," *Bull Environ. Contamn. Toxicol*, 1993, 50, 138-143
- [10] Jimoh AA, Adebayo GB, Otun KO, Ajiboye AT, Bale AT, Jamiu W and Alao FO, "Sorption Study of Cd(II) from Aqueous Solution Using Activated Carbon Prepared from Vitellaria paradoxa Shell," *Jimoh et al., J Bioremed Biodeg* 2015, 6:3
- [11] C.K. Singh a, J.N. Sahu a, K.K. Mahalik a, C.R. Mohanty b, "Studies on the removal of Pb(II) from wastewater by activated carbon developed from *Tamarind wood* activated with sulphuric acids," *Journal of Hazardous Materials* 153, 2008, 221-228
- [12] Vaishaly A.G, Blessy B. Mathew and N.B. Krishnamurthy, "Health Effects Caused by Metal Contained Ground Water," *International Journal of Advances in Scientific Research*, 2015; 1(02): 60-64.