



BIO-ADSORPTION OF HEAVY METAL ION FROM WATER USING ACTIVATED CARBON

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ABSTRACT

The discharge of effluent polluted with heavy metals have become a growing concern for researchers around the world. This study evaluated the removal efficiency of commercial activated carbon and rice husk activated carbon as adsorbents for the removal of copper ion in water. A nominal size of 1 mm was obtained after sieving the Rice Husk, washed with distilled water, dried in an oven at 80 °C for 12 hours, and pyrolyzed in a furnace at 550 °C for 30 minutes. The chars produced were later air-dried and then activated with lemon juice. The Rice Husk Activated Carbon (RHAC) and Commercial Activated Carbon (CAC) purchased from the market were both subjected to the following analyses: bulk density, X-ray Fluorescence (XRF), Brunauer–Emmett–Teller (BET), Scanning Electron Microscope (SEM) and Energy Dispersive X-ray (EDX) in order to characterise the adsorbents and to understand their suitability for the removal of copper ion in water. One of the properties of an adsorbent is large pores which is exhibited by the activated carbons as revealed by the SEM analysis. Likewise, the XRF and EDX analyses confirmed that the adsorbents had larger proportion of Silica (50.1 – 50.25%), Carbon (60.06 – 84.87 wt. %) and Oxygen (15.13 – 21.60 wt. %) which is a property of a good adsorbent. BET analysis showed that the surface areas of the rice husk activated carbon and the commercial activated carbon were 998.35 and 1208.25 m²/g, respectively. The bulk densities of the rice husk activated carbon and the commercial activated carbon were 0.3325 and 0.2812 g/cm³, respectively. The maximum removal efficiency using RHAC was observed at 60 °C and 120 minutes at 83.96 and 89.21 %, respectively while for CAC the maximum removal



efficiency was observed at 60 °C at 84.61 % and 30 minutes at 83.3 %. Initial concentration of 20 mg/l was observed to have the highest removal efficiency for the two activated carbon specimens. The modelled effect of initial concentration, temperature and contact time on removal efficiency yielded R² values of 1, 0.918; 1,1 and 1,1 respectively for the CAC and RHAC.

KEYWORDS

Activated carbon, heavy metal removal, adsorbent, biological activator.

1. INTRODUCTION

The discharge of industrial effluent has become a growing source of concern for environmentalists all around the world. Pollutants are constantly released into the environment—in the form of water, soil, and air that produces a large amount of residue. There has been research going on for years to add value to wastes that previously had no value. In this view, the desire to make use of a variety of wastes has sparked interest in the development of innovative procedures for the manufacturing of adsorbents. As a result, low-cost residues with high carbon and low inorganic content can be used to make activated carbon, one of the most well-known and widely used adsorbents, ([Azner, 2011](#)).

Activated carbon, activated charcoal or activated coal is a porous carbonaceous substance with a large internal surface area that is non-hazardous. It is capable of adsorbing a wide range of chemicals, including various colours and heavy metals. It is named adsorbent because it has the ability to attract molecules to its interior surface. It is a type of carbon that has been treated to be very porous and therefore has a large surface area that can be used for adsorption and chemical reactions. ([Ahiduzzaman and Sadrul, 2016](#)). The adsorption capacity of activated carbon is one of its most important features. This property is governed by the porous structure and chemistry of activated carbon surface. Both are related to crystal composition.

This can be used as adsorbent because of its high porosity, particularly in the micro and meso ranges ([Herawan, et al, 2013](#); [Opafola, et al 2021](#); [Akomah, et al. 2021](#)).

The utilization of agricultural waste as raw material for high adsorption capacity activated carbons is a hot spot in recent literature. For example, ([Mohammad et al, 2014](#)) prepared activated carbon from rice husk by H_3PO_4 activation using oven drying. ([Efeovbokhan et al, 2019](#)). Prepared activated charcoal and biological activator from psyllium skin and coconut shell.

This study aims to develop activated carbon from rice husk to serve as an adsorbent in water and wastewater treatment; characterizes the adsorbents in terms of textural and chemical properties in order to understand their suitability for the removal of copper ion and its efficiency.

The novelty of this work is the use of lemon juice as the biological activator and the reduction of environmental burden caused by indiscriminate dumping of agricultural waste (rice husk) into the environment.

2. MATERIALS AND METHODS

2.1. Materials

The carbonization, adsorption and the modelling processes is presented in the graphic abstract presented in Fig. 1. Rice husk was collected from a milling industry in Lafenwa, Abeokuta, Ogun State and lemon fruit purchased from Kuto Market, Abeokuta, Ogun State. Commercially produced activated carbon was purchased from Ibadan, Oyo State, Nigeria. Copper (II) Chloride, was obtained from Abeokuta, Ogun State.

2.2. Carbonization process

Muffle furnace, Rice Husk (RH), distilled water, 1 mm Sieve and Oven were used in the experimental set up. The dehydration of the rice husk was achieved in six days through sun-drying as specified by (Abdulrazak et al, 2017). Residues and impurities such as ash and dust were removed from the Rice Husk using distilled water and later oven-dried at 80 °C for 12 hours as specified by (Lam and Zakaria, 2008). The RH was then pyrolyzed in a muffle furnace at a temperature of 550 °C for 30 minutes as specified by (Adekunle et al, 2020). The char produced was then air dried. Two hundred and fifty (250) ml of the lemon juice was added and mixed with 500 g of the rice husk charcoal sample. Drying of the mixture at 110 °C for 24 hours took place after agitation and filtration process and transferred to a muffle furnace for heat treatment at 400 °C for one hour. The samples were then cooled and washed several times until the pH is neutral. The final sample was dried again to a constant weight at 110 °C in an oven (Ambali et al, 2015). The simulated wastewater was prepared by adding 2.118 g of Copper (II) Chloride with molecular weight to 1 litre of distilled water to get a stock solution of 1000 mg/l. 100 mg of the RHAC is added to 5 different conical flasks containing 100 ml of varied concentrations of Copper (II) ion solutions.

The copper ion was removed by using activated carbon as adsorbent using batch adsorption technique. (Abdulrazak et al, 2017). The conical flask was stirred at five agitations per minute. The conical flasks were agitated for 30, 60, 90, 120, 150 minutes, respectively. The content in each flask were filtered using filter paper and prepared for analysis. The filtrates were then analysed using atomic adsorption spectrometer. The removal efficiency is then calculated using:

$$\text{Removal efficiency (\%)} = \left(1 - \frac{C_e}{C_i}\right) \times 100\% \quad (1)$$

Where C_e is and C_i is The procedure is then repeated for commercially produced activated carbon.

2.2.1. Effect of contact time

100 mg of activated charcoal prepared from rice husks was placed in 5 different Erlenmeyer flasks containing 100 mL of copper (II) ion solution according to (Abdulrazak et al, 2017). The Erlenmeyer flask was stirred with 5 movements per minute. The Erlenmeyer flask was shaken for 30, 60, 90, 120 and 150 minutes, respectively. The contents of each flask were filtered using filter paper and prepared for analysis. The filtrate was then analyzed using an atomic adsorption spectrometer. The removal efficiency is then calculated using

$$\text{Removal efficiency (\%)} = \left(1 - \frac{C_e}{C_i}\right) \times 100\% \quad (1)$$

The procedure is then repeated for commercially produced activated carbon.

2.2.2. Effect of temperature

100 mg of the charcoal produced from rice husk is added to 5 different Erlenmeyer flasks containing 100 ml of copper (II) ion solution as specified by (Abdulrazak et al, 2017). The flasks together with its content were placed in a water bath of 20 °C, 40 °C, 60 °C, 80 °C, 100 °C respectively for 20 minutes. The solutions were then filtered and the filtrates were analysed using atomic adsorption spectrometer. The removal efficiency was then calculated using Equation 1 The procedure is then repeated for commercially produced activated carbon.

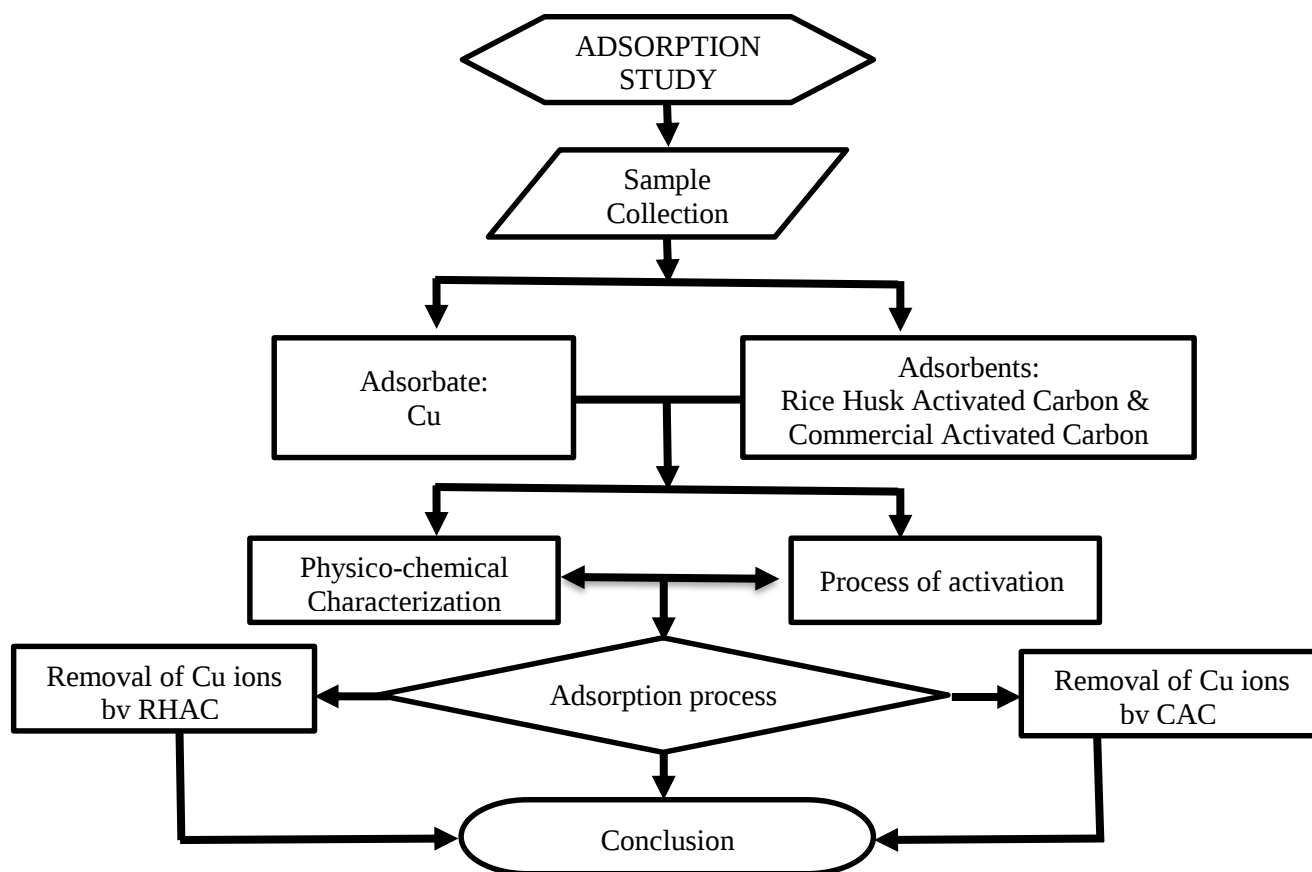


Fig. 1. Graphic abstract for the experimental set up.

3. RESULTS AND DISCUSSION

3.1. Physiochemical properties

Table 1 shows the physiochemical properties of the CAC and RHAC. It shows that the commercially obtained activated carbon has a higher surface area to the low cost activated carbon and the rice husk activated carbon is denser than the commercially obtained activated carbon.

Table 1. Physiochemical properties of the adsorbents.

Parameters	Activated Carbon	Rice Husk Activated carbon
Bulk Density (g/cm ³)	0.2812	0.3325
Surface Area (m ² /g)	1208.25	998.35

3.1.1. XRF analysis

The results obtained from XRF analysis of the adsorbent materials are shown in Fig. 2 which revealed that the two activated carbon contains Silica (SiO₂ – 50.1 to 50.25 Wt. %), Alumina (Al₂O₃ – 31.23 to 32.9 Wt %), Ferric Oxide (Fe₂O₃ – 0.77 to 4.05 Wt.%), Calcium Oxide (CaO – 2.38 to 5.43 Wt.%), and other oxides. The activated carbons do not contain ash which is an impurity as it lowers the activated carbon's adsorptive capability, (Ohanaka et al, 2021). The two activated carbons contain trace elements like Barium and Cerium. Rubidium, Chromium, Copper, Nickel and Lead are of higher concentration in the commercially gotten activated carbon and contain less than 1 ppm in the rice husk activated carbon. Zirconium is present in the commercially gotten activated carbon at a concentration of 70 ppm and absent in the rice husk activated carbon.

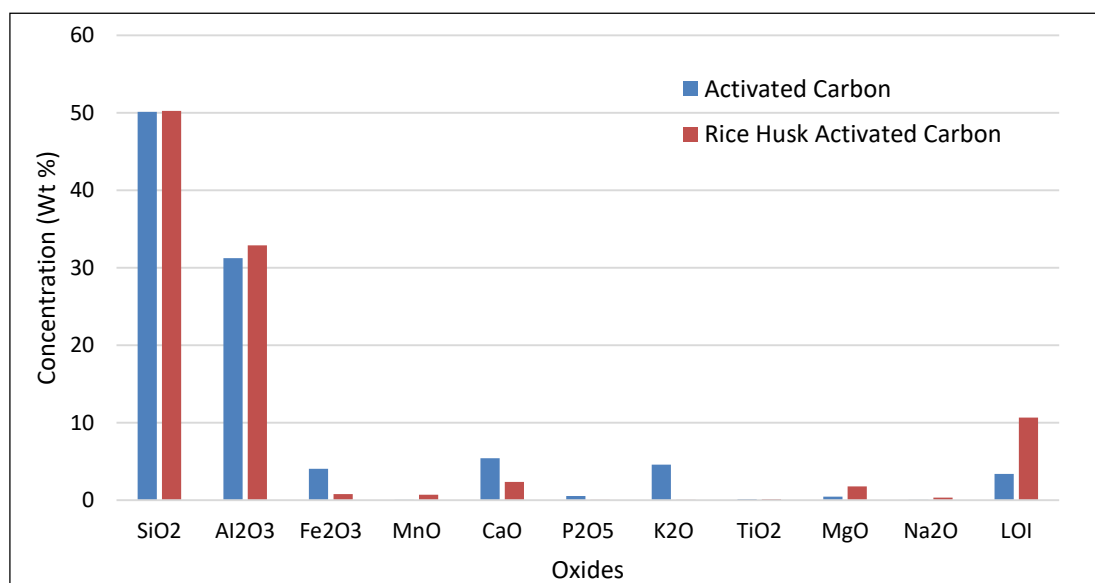


Fig. 2. Bar chart showing the percentage concentration of major oxides in CAC AND RHAC.

Table 2 shows the ANOVA result for the XRF analysis for the commercial activated carbon (CAC) and the rice husk activated carbon (RHAC). The P value as shown in the table is greater than the alpha value of 0.05 which means that there is no significant difference between the mean of CAC and RHAC. This means that RHAC will be effective for the purpose of heavy metal removal.

Table 2. ANOVA result for the XRF analysis.

SUMMARY						
Groups	Count	Sum	Average	Variance		
Activated Carbon	11	100	9.090909	265.5233		
Rice Husk Activated Carbon	11	100	9.090909	282.234		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	1.82E-12	1	1.82E-12	6.64E-15	1	4.351244
Within Groups	5477.574	20	273.8787			
Total	5477.574	21				

3.1.2. SEM Analysis

The surface morphologies of the adsorbents determined using SEM are as shown in Fig. 3 and 4. It can be observed from Fig. 3 that the structure of the low-cost activated carbon is a honeycomb-like structure at the magnification of 9,000 times. The two adsorbents possess small pores and approximately the same size (Opafola, et al 2021).

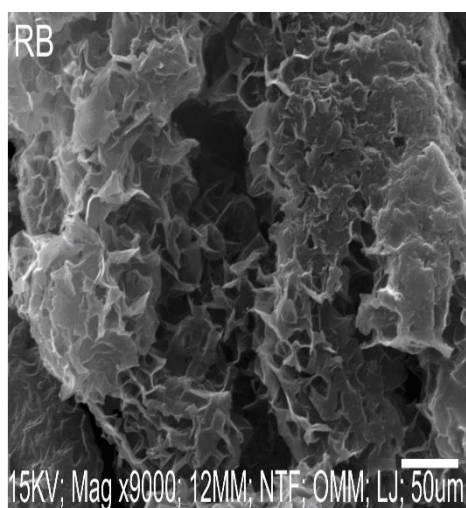


Fig. 3. SEM Analysis Showing Surface Morphology of the Rice Husk activated carbon.

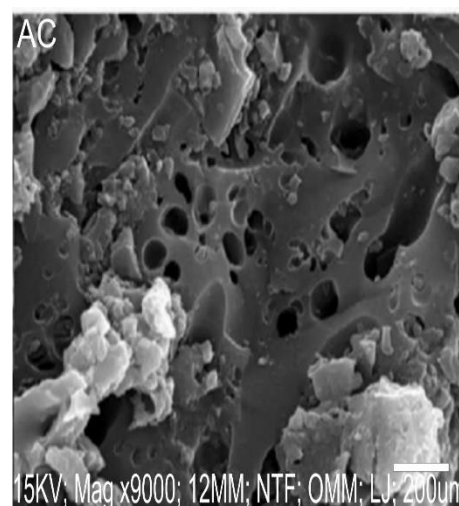


Fig. 4. SEM Analysis Showing Surface Morphology of the Activated Charcoal.

3.1.3. Energy Dispersive X-ray Results

Figs. 5 and 6 show the EDX results. Further chemical characterization (that is, EDX analysis) of the CAC shown in Fig. 5 revealed that the activated carbon contains Iron, Oxygen and Carbon in ascending order while the EDX analysis of the RHAC as shown in Fig. 6 revealed that the low cost activated carbon contains Oxygen and Carbon in ascending order

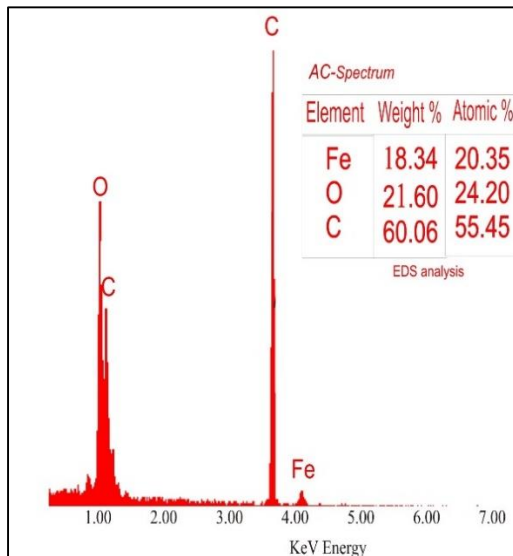


Fig.5. EDX Analysis Showing Chemical Characterisation of the commercially purchased

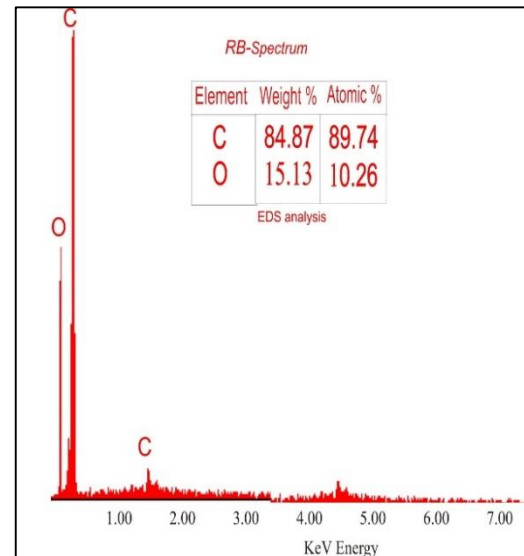


Fig. 6. EDX Analysis Showing Chemical Characterisation of Rice Husk activated carbon

4. REMOVAL OF HEAVY METALS

4.1. Effect of initial concentration on removal efficiency

From Fig. 7, 20 mg/l has the highest efficiency for the two activated carbons.

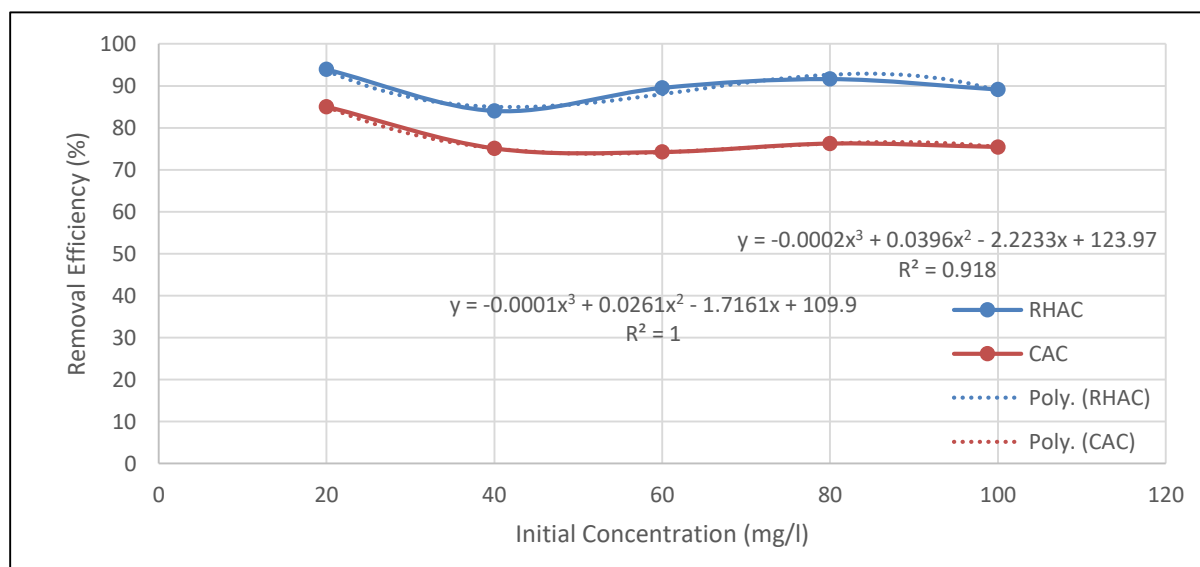


Fig. 7. Effect of initial concentration on copper ion removal efficiency for CAC and RHAC.

4.1.1. Effect of temperature on removal efficiency

Fig. 8 shows that the optimum removal efficiency for both RHAC and CAC occurred at 60 °C. The generated models indicate a good performance with coefficient of determination value at one.

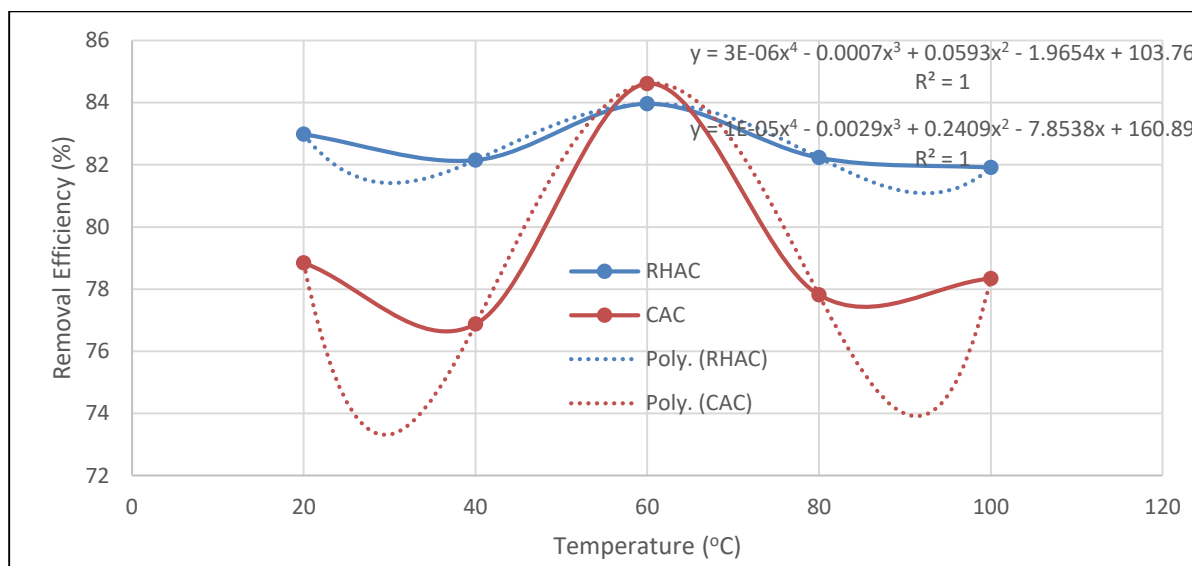


Fig. 8. Effect of temperature on copper ion removal efficiency for CAC and RHAC.

4.2. Effect of contact time on removal efficiency

Fig. 9 shows that the optimum removal efficiency for RHAC occurred at about 120 minutes while that of CAC occurred at 30 minutes. The generated models indicate a good performance with coefficient of determination value at one.

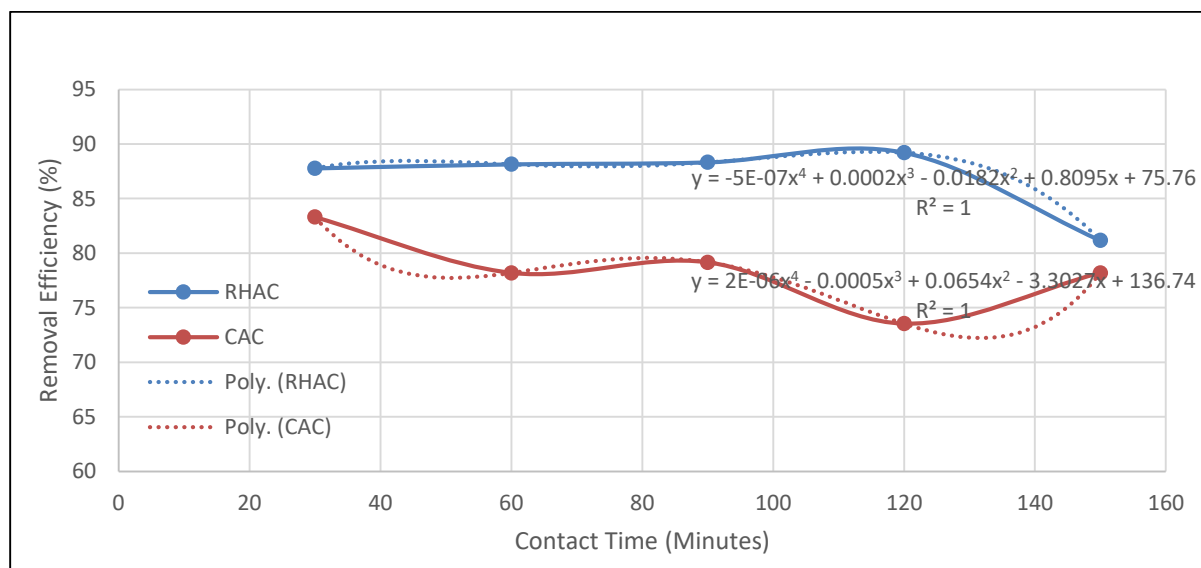


Fig. 9. Effect of contact time on copper ion removal efficiency for CAC and RHAC.

The activated carbon was tested using batch adsorption technique to adsorb the copper ions at varying contact time, temperature and initial concentration. SEM analysis showed that activated carbon developed more pores. This is a characteristic of the adsorbent. XRF and EDX analysis also confirmed a high proportion of silica, carbon and oxygen adsorbents. This is a characteristic of excellent adsorbents. The adsorbents have no ash content which is an impurity in activated carbon. BET analysis showed that the surface areas of the rice husk activated carbon and the commercial activated carbon were 998.35 and 1208.25 m²/g, respectively. The bulk density of the rice husk activated carbon and the commercial activated carbon are 0.3325 and 0.2812 g/cm³, respectively. The maximum removal efficiency of the rice husk activated carbon was observed at 60 °C and 120.

minutes at 83.96 and 89.21 %, respectively while for commercially produced activated carbon, the maximum removal efficiency was observed at 60 °C at 84.61 % and 30 minutes at 83.3 %. Initial concentration of 20 mg/l was observed to have the highest removal efficiency for the two activated carbon specimens. The modelled effect of initial concentration, temperature and contact time on removal efficiency yielded R² values of 1, 0.918; 1,1 and 1,1 respectively for the CAC and RHAC.

5. CONCLUSION

Activated carbon made from rice husks, which is an inexpensive agricultural waste, has shown excellent ability to remove copper ions in water.

The rice husk activated carbon have a lower surface area compared to the commercial activated carbon. The following conclusions can be drawn from the analysis of this study. The rice husk activated carbon was more efficient compared to the commercial activated carbon. That the importation of commercial activated carbon (CAC) can be stopped while utilizing rice husk that would have constituted an unwanted municipal solid waste.

Ethical Approval: Not applicable

Consent to Participate: Not applicable

Consent to Publish: Not applicable

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Availability of data and materials: The data that support the findings of this study are available from the corresponding author, upon reasonable request.

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