

# Removal of $\text{Cu}^{2+}$ & $\text{Fe}^{3+}$ Ions from Oil Wells by Local Activated Carbon in Batch Adsorption Process

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## Abstract

In this study the efficiency of activated carbon produced from rice husk (RHAC) was studied to remove of  $\text{Cu}^{2+}$  &  $\text{Fe}^{3+}$  ions from waste oil water which comes out from the oil industry in Basrah city, and the efficiency of RHAC has been compared with efficiency of raw rice husk (RH) and commercial activated carbon (CAC). The effects of operating parameters were studied such as (contact time, adsorbent size, adsorbent dose, initial concentration of adsorbate, temperature, pH and rotation speed). The adsorption efficiency is arranged by (RHAC > CAC > RH) which removed 96.588, 92.9, 69.3% of  $\text{Cu}^{2+}$  ions, and 94.54, 92.23, 72.66% of  $\text{Fe}^{3+}$  ions onto RHAC, CAC & RH, respectively. The FTIR results showed that a different in functional groups was scattered in surface of adsorbent material, that causing the different in adsorption efficiencies of heavy metal ions.

**Keywords:** Adsorption, Activated carbon, Rice husk, Commercial activated carbon, Heavy metals, Waste oil water, FTIR

## 1. Introduction

The heavy metals are of special concern because they are non-degradable and therefore persistent. Commonly encountered metals of concern include  $Pb^{2+}$ ,  $Cu^{2+}$ ,  $Zn^{2+}$ ,  $Co^{2+}$ ,  $Ni^{2+}$  etc. These metals are toxic both in their chemically combined forms and in the elemental form (Al-tameemi et al., 2011). Conventional methods of removing toxic heavy metal ions include chemical precipitation, chemical oxidation or reduction, filtration, ion exchange, electrochemical treatment, application of membrane technology and evaporation recovery. However, these processes have considerable disadvantages such as incomplete metal removal, expensive equipment and monitoring system, high reagent or energy requirements, generation of toxic sludge and other waste products that require disposal (Rao & Prabhakar, 2011). Among all the methods adsorption is highly effective, economical, technological, environmental and biological importance. Furthermore the practical applications of adsorption process in industry and environmental protection are important. Adsorption is a method for separation of mixtures that based on the change in concentration of components at the interface (Prabakaran & Arivoli, 2012; Shanthi & Selvarajan, 2012). Though the use of commercial activated carbon is a well-known adsorbent for the removal of heavy metals from water and wastewater, the high cost of activated carbon limits its use as an adsorbent in developing countries. Hence, it is a growing need to derive the activated carbon from cheaper and locally available waste materials. Several workers used different low-cost adsorbents from agriculture (Shanthi & Selvarajan, 2012) such as seed shell of palm tree (Gueu et al., 2006) dates stones (Alhamed & Bamufleh, 2008) apricot stones (Philip & Girgis, 1996) oil palm fibre (Nwabanne & Igbokwe, 2012) pine, oak woods, olive seeds (Skodras, 2007) apricot stones (Philip & Girgis, 1996) waste seeds husks of *Moringaoleifera* (Warhurst, 1996) coconut shell (Satya, 1997) *ArundoDonax* canes (Basso, 2002) pistachio-nut shells (Yang, 2003) and coir pith (Adekola & Adegoke, 2005) sun flower (Surchi, 2011).

In this study, the efficiency of AC produced previously from rice husk (RHAC) to removal of  $Cu^{2+}$  &  $Fe^{3+}$  was investigated, and compared with raw rice husk (RH) and commercial activated carbon (CAC). The effect of operating parameters was studied such as (contact time, initial concentration of adsorbate, adsorbent size, temperature, adsorbent dose, pH and rotation speed).

## 2. Experimental Section

### 2.1 Materials

Copper chloride anhydrous with purity (99%) was supplied from BDH (Chemicals) Company. Iron nitrate (ferric nitrate) with purity (99%) was supplied from MERCK Company. Hydrogen chloride with purity (37 %) was supplied from Scharlab. S. L Company. Sodium Hydroxide with purity (97.5%) was supplied from THOMAS BAKER (Chemicals) Company.

### 2.2 Adsorbent

Activated carbon produced from rice husk by physical method (RHAC) was used as an adsorbent material in this study, the efficiency of RHAC was compared with raw rice husk

(RH) and commercial activated carbon (CAC). The preparation method was described following:

After well washing the RH with distilled water and dried in electrical oven for 24 hr at 120°C, the carbonization step was carried out in electrical furnace for 2hr at 500°C and heating rate of 30 °C/min in absence air using nitrogen at flow rate is 200 L/min. The product from carbonization step was activated by passing carbon dioxide (CO<sub>2</sub>) instead of nitrogen for 2hr at 700°C.

Table 1. Characteristics of the adsorbent

Adsorbent	BET surface area m <sup>2</sup> /g	Bulk density g/ cm <sup>3</sup>	Particle density g/ cm <sup>3</sup>	Porosity	Pore Volume cm <sup>3</sup> /g	pH
CAC	-	0.5103	1.2203	0.5819	1.2367	9.4
RH	0.9672	0.3533	1.1048	0.6802	2.2341	7.1
RHAC	201.1518	0.2553	1.4369	0.8223	3.6876	9.3

### 2.3 Source of Waste Oil Water

After measuring the concentrations of heavy metals ions in waste oil water from different locations (West Qurna, NahranBinUmer, Barjessia) by using atomic absorption were found that different concentrations as shown in Table 2.

Table 2. Heavy metal ions concentration (ppm) in different oil location in basrah city.

Location	<b>Cu<sup>2+</sup></b>	<b>Fe<sup>3+</sup></b>
Nahran Bin Umer	2.3	7.2
West Qurna	0.5	2.4
Barjessia	4	3.6

For simulated waste oil water, solutions were prepared with lower and higher concentrations of these two heavy metal ions in the locations mentioned above.

### 2.4 Preparation of Standard Solutions

The stock solutions of 1000 mg/L (ppm) of Cu<sup>2+</sup> and Fe<sup>3+</sup> were prepared by dissolving 2.1368 g of CuCl<sub>2</sub> and 7.3073 g of Fe(NO<sub>3</sub>)<sub>3</sub> in 1000 ml volumetric flasks and fill up to the mark with distilled water.

The diluted concentrations were prepared from stock solutions for carrying out experiments.

Following the equation below:

$$N_1 * V_1 = N_2 * V_2 \quad (1)$$

And then a certain volume (10 ml) of oil has been added to all above solution with efficient agitation for simulated waste oil water (1% oil in waste water), because the highest value of oil contains in waste oil water of the locations mentioned above doesn't exceed 1 %.

### *2.5 The Calibration Curve Method of Analysis.*

In 25 ml volumetric flasks were prepared a series of standard solutions containing 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65 and 70 ppm of heavy metals ions. After determining the absorbance by AAS, making a plot of absorbance vs. concentration using EXCEL software. The results and the equations which were described calibration curve are shown in Figure 1.

### *2.6 Analyze a Sample of Heavy Metal Ions by Using Atomic Absorption Spectrophotometer (AAS)*

The concentration of metal ions was measured by using atomic absorption spectrophotometer (BUCK Scientific, Model 210 VGP). In atomic absorption spectroscopy, metal atoms were vaporized into a flame, and the metal vapor absorbed radiation from the specific hollow cathode lamp in proportion to the number of atoms present. Beer's Law was followed in the part-per-million range (remember that ppm means mg of metal/liter of solution).

### *2.7 Fourier Transform Infrared Spectroscopy (FTIR)*

The surface functional groups of the prepared activated carbon were detected by Fourier Transform Infrared spectroscope in State Company For Petrochemical Industries Basrah using Shimadzu FTIR 8400S, A spectrum was recorded in the mid-IR range from 4000 to 400  $cm^{-1}$  with a resolution of 1  $cm^{-1}$ .

### *2.8 Batch Adsorption Processes*

Adsorption experiments were carried out in double neck(125 ml) flask using 100 mL metal bearing solution with a certain dosage of the AC and installed in the water bath (MammertGmbh Type WMB 22). The pH values were controlling by adding 0.1N NaOH or 0.1N HCl, and stirred for different rotation speed using Variable-Speed Benchtop, model 5850, Eberbach. Before the metal solution analyzed using AAS was filtered through Whatman4 filter papers.

The efficiency of each bed was measured in the case of batch adsorption with different parameters such as the effect of temperature (8-55°C ), Contact time (2-30 minutes), pH (3-11), adsorbent Dose (1-5 g), adsorbent Diameter (0.075-1.14 mm), Rotation speed (0-1500 rpm) and initial Concentration of heavy metal ions (10-60 ppm), and comparison between the effect of operating conditions on adsorption of heavy metals ion by CAC, RHAC and RH.

## **3. Results and Discussion**

### *3.1 Efficiency of Adsorption*

The Removal Percentage (R.P.%) which described the efficiency of adsorbent to adsorbed a heavy metals ions is calculated by following equation (Kumar & Gayathri R, 2009):

$$R.P.\% = \frac{C_i - C_f}{C_i} * 100 \quad (2)$$

Where:  $C_i$  and  $C_f$  are the initial and final concentration in (ppm), respectively.

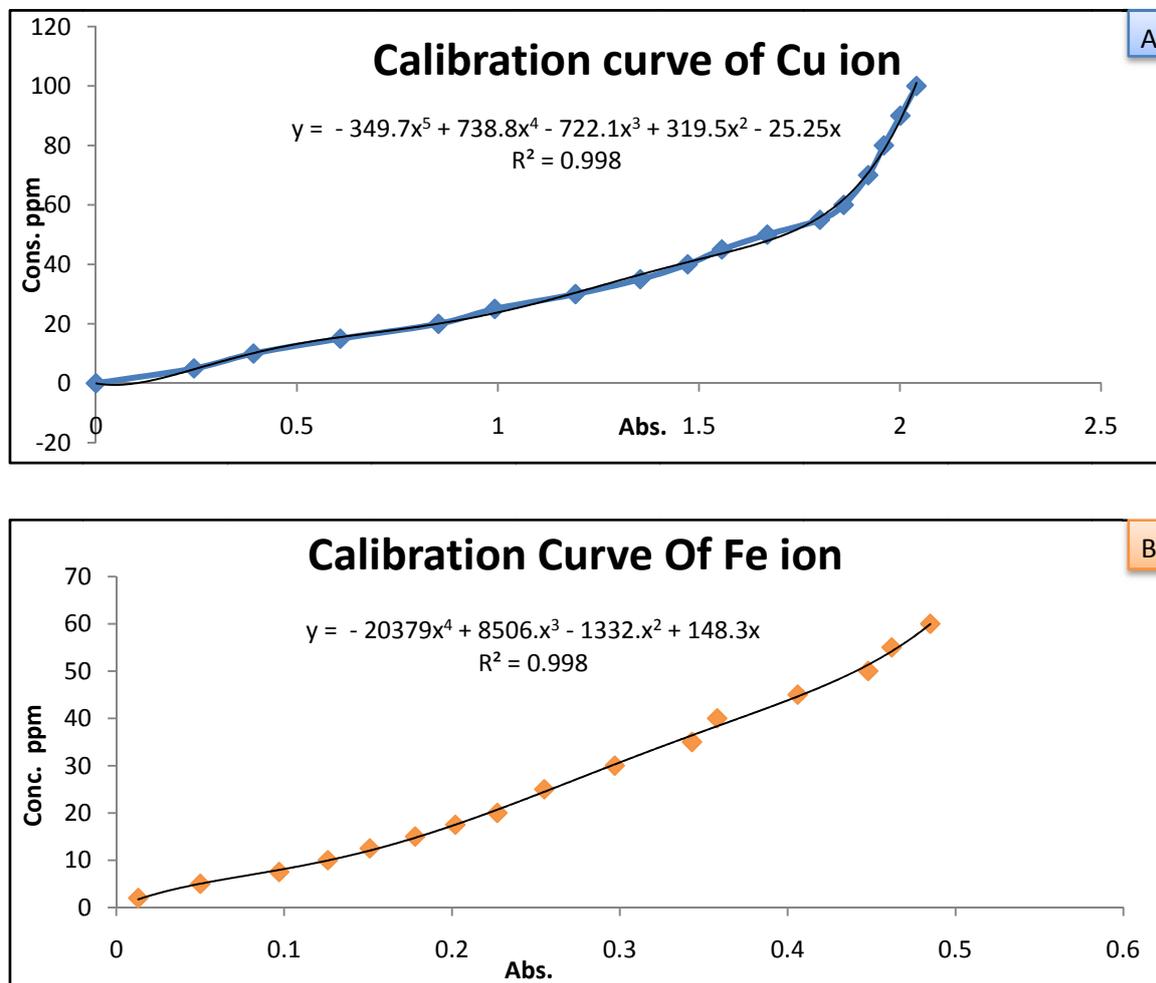


Figure 1. Calibration curves of heavy metal ions

### 3.2 Effect of Contact Time

The effect of contact time on the adsorption of heavy metal ions (H.M.I.) was investigated. The experimental work was done under the conditions of constant temperature (25°C), adsorbent dose (1g), particle size (0.3), rotation speed (600 rpm), PH (2.9), initial concentrations (50ppm) and contact time(0-30 min), the experimental data are presented in Figure 2.

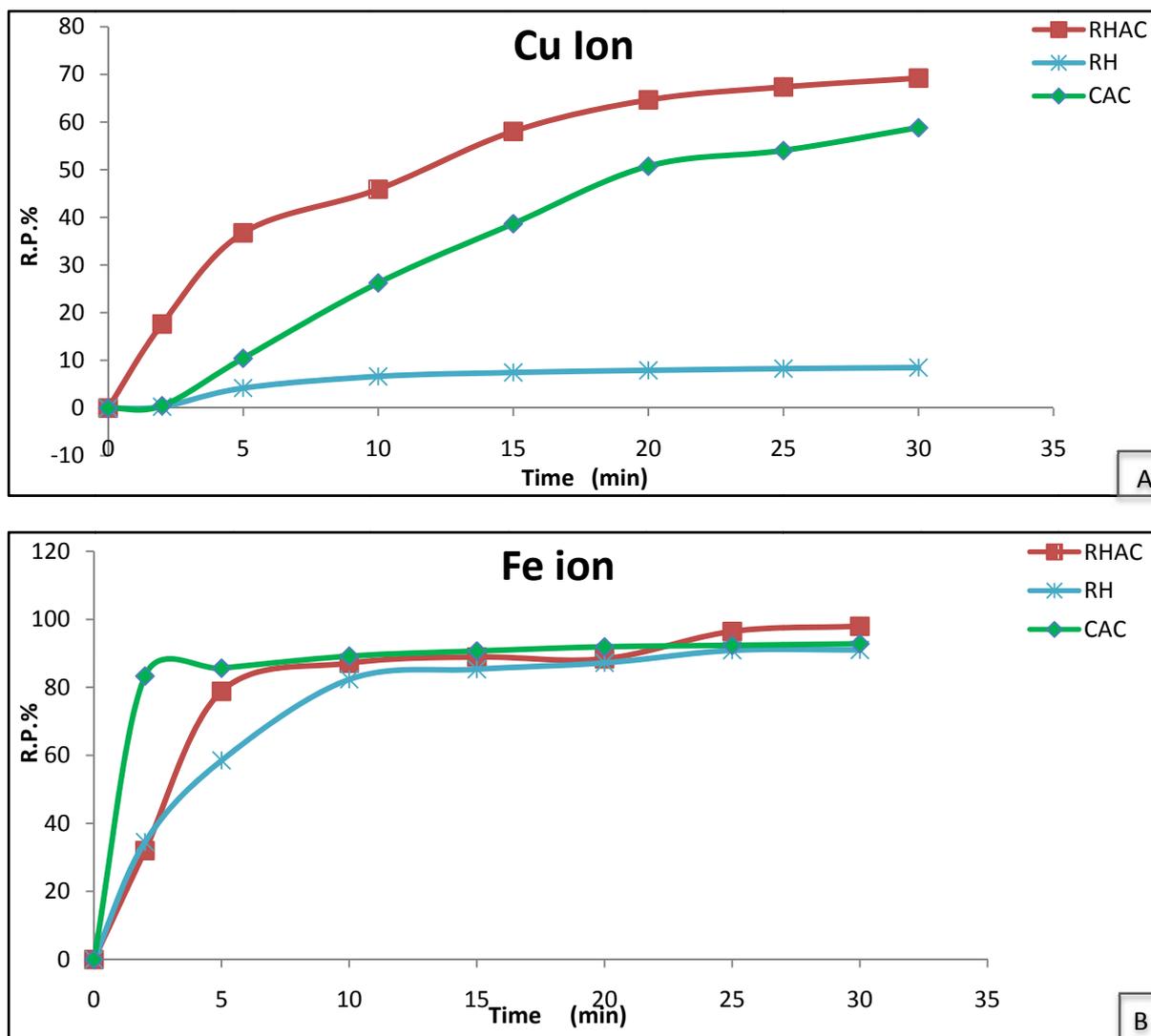


Figure 2. Effect of time contact on the adsorption of heavy metal ions

The results shown that the R.P. of adsorption first increases with increasing in contact time, then remains almost constant. This may be due to the availability of the uncovered surface area of the adsorbents, since the adsorption kinetics depend on the surface area of the adsorbents. The adsorption of heavy metal ions takes place at the more reactive sites. As these sites are progressively filled the more difficult the sorption becomes (BADMUS et al., 2007).

### 3.3 Effect of Temperature

The efficiency of adsorption of heavy metal ions was studied with different temperature range (8-55°C), while the other operating parameters were kept constant at initial concentration (50 ppm), rotation speed (600 rpm), pH (3), adsorbent dose (1g), contact time (15min) and adsorbent size (0.3 mm). The experimental data are shown in Figure 3.

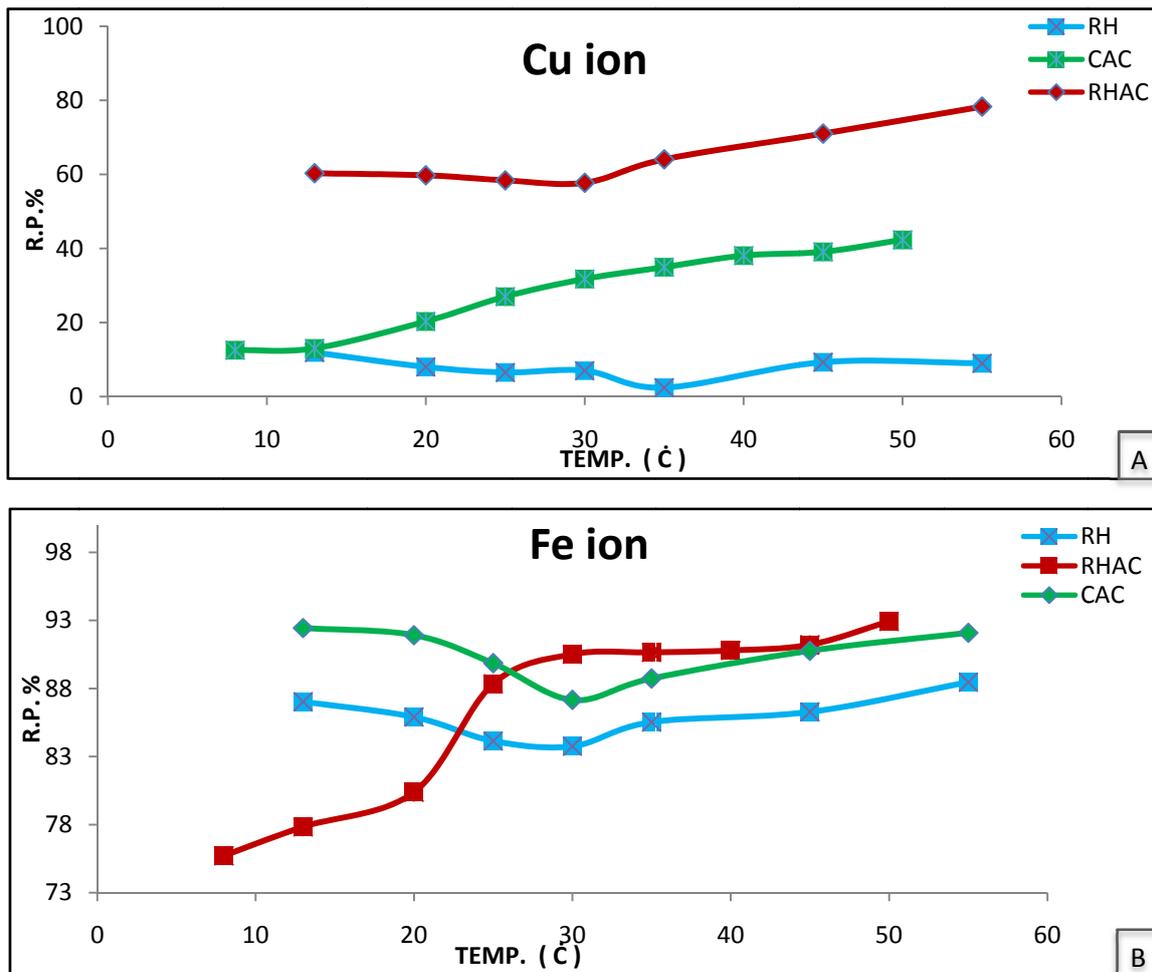


Figure 3. Effect of temperature on the adsorption of heavy metals ions

The plots of R.P. as a function of temperature shows an increasing amount of adsorbed H.M.I with increasing temperature. But the adsorption capacity does not largely depend on temperature. The enhancement in adsorption with rise in temperature may be attributed to increase in the number of active surface sites available for adsorption, increase in the porosity and in the pore volume of the adsorbent (Chowdhury et al., 2010). An increase of temperature increases the rate of diffusion of the adsorbate molecules across the external boundary layer and within the internal pores of the adsorbent particle, due to decrease in the viscosity of the solution (Chowdhury & Saha, 2010; Hameed et al., 2008). This phenomenon is characteristic of a chemical reaction or bonding being involved in the adsorption process (Guo et al., 2002).

### 3.4 Effect of pH

The pH of the aqueous solution was an important variable. Adsorption experiments were carried out at pH (3-10). The acidic or alkaline of the solutions was maintained by adding the required amounts of dilute HCL or NaOH solutions. The experimental work was done under the conditions of constant temperature (28<sup>0</sup>C), adsorbent dose(1g), particle size (0.3), rotation speed (600 rpm), initial concentrations (50ppm) and contact time(15 min), the experimental

data are presented in Figure 4.

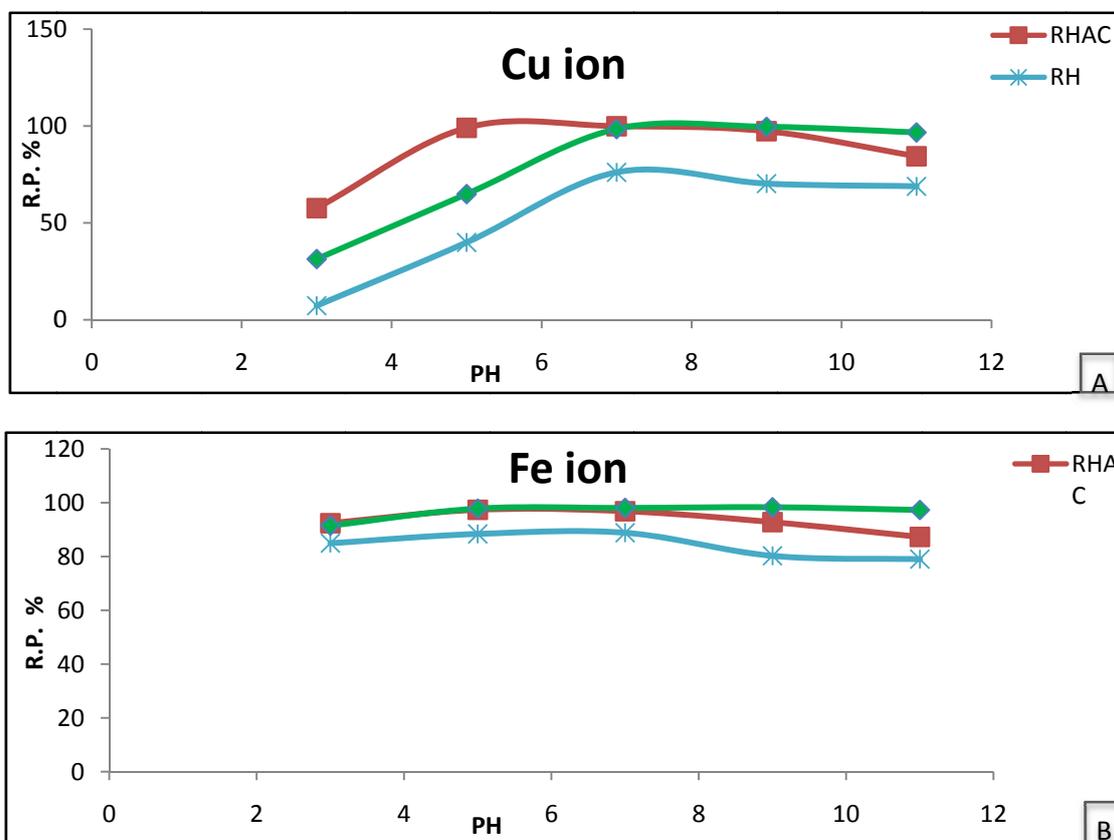


Figure 4. Effect of pH on the adsorption of heavy metals ions

It has been found that the adsorption increases with increase in pH until the optimum pH is reached, the optimum pH of maximum adsorption for H.M.I. on different adsorbent was determined from Figure 4 and the results were listed in Table 3. Increases in R.P. with increased pH can be explained on the basis of decrease in competition between proton and metal cations for same functional groups and by decrease in positive surface charge, which results in a lower electrostatic repulsion between surface and metal ions. Decrease in adsorption at higher pH is due to formation of soluble hydroxyl complexes (Meena et al., 2010).

Table 3. The optimum value of pH of H.M.I. adsorption

H.M.I.	CAC	RHAC	RH
Cu <sup>2+</sup>	9	7	7
Fe <sup>3+</sup>	7-9	7	7

### 3.5 Effect of Initial Concentration

Adsorption experiments were carried out at different initial H.M.I. concentrations ranging from 10 to 60 ppm to study the effect of initial concentration, other conditions of experimental work were kept constant at rotation speed (600 rpm), pH (3.5), adsorbent dose (1g), contact time (15min), temperature (25<sup>0</sup>C), and adsorbent size (0.3 mm). All the adsorption efficiencies obtained are shown in Figure 5.

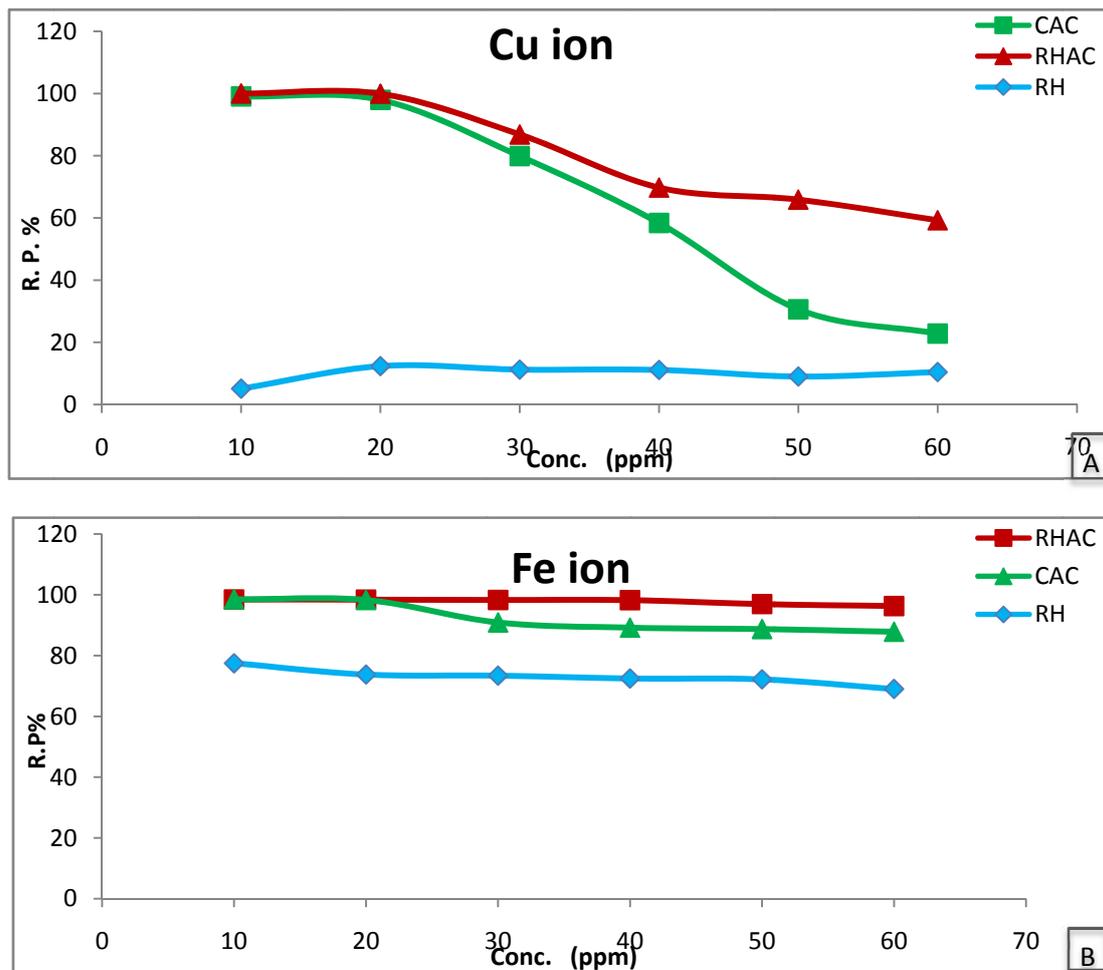


Figure 5. Effect of initial concentration on the adsorption of heavy metals ions

The results are shown that the removal percentage of H.M.I. (efficiency) decreases, with the increase in H.M.I. concentration while the amount of H.M.I. removed will be increased. These data suggests that the removal of metal ions is highly dependent on the concentration. At lower concentrations of metal ions, the number of H.M.I. which is available in the solution is less as compared to the available sites on the adsorbent. However, at higher concentrations the available sites for adsorption become fewer (Mohammadi et al., 2010). In other words at low concentration, most of H.M.I. in the solution might contact with active sites of adsorbent

and when the concentration is increased all H.M.I. will not be available to contact with the active surface due to active sites are already filled up (Rahman et al., 2012). And this was due to the fact that a lower concentration gradient caused a slower transport due to a decrease in the diffusion (Tan et al., 2008).

### 3.6 Effect of Adsorbent Dose

The effect of adsorbent dose on H.M.I. adsorption was investigated with different adsorbent dosage ranging (1-5 g), the other conditions of experimental work were kept constant at contact time (15min), rotation speed (600 rpm), pH (3), temperature (25<sup>0</sup>C), initial concentration (50ppm) and adsorbent size (0.3 mm). All the adsorption efficiencies obtained are shown in Figure 6.

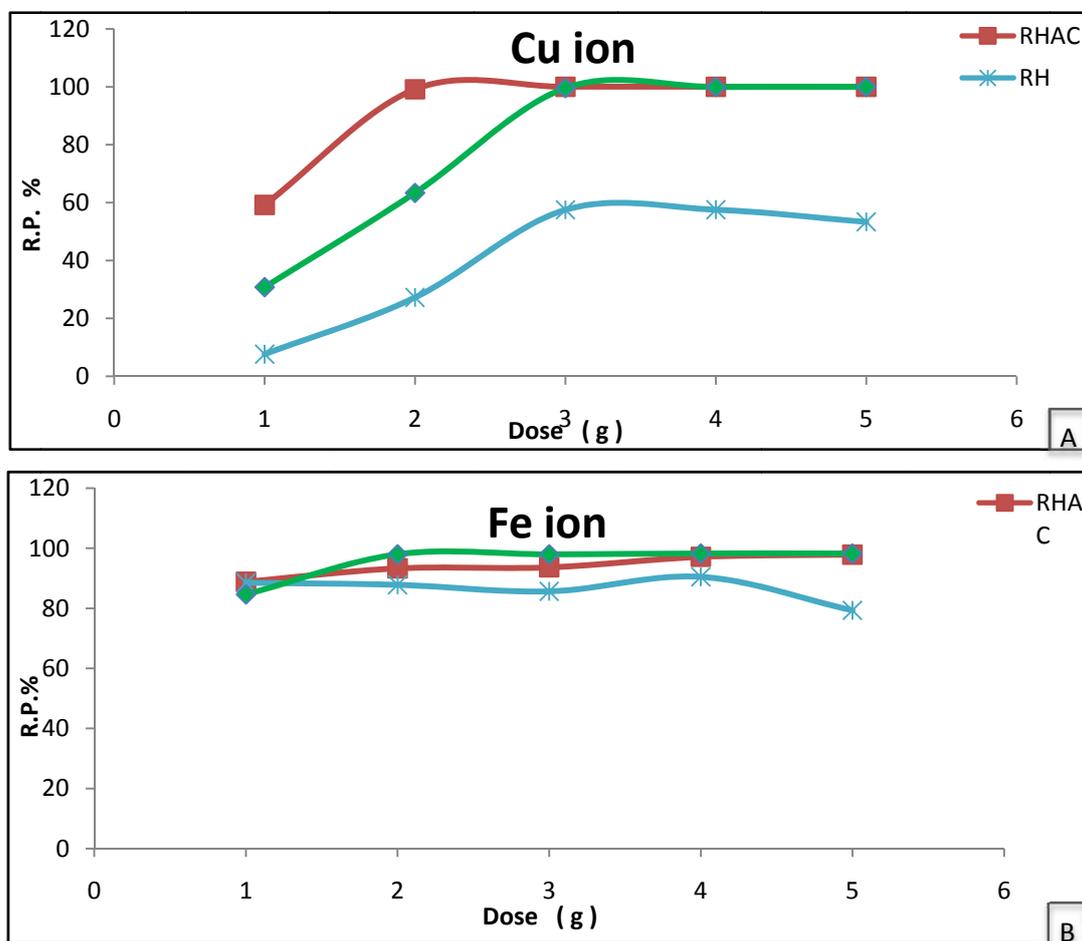


Figure 6. Effect of adsorbent dose on the adsorption of heavy metals ions

The adsorption capacity increased with increasing adsorbent dosage until a certain value, any further addition of the adsorbent beyond this value did not cause any significant change in the adsorption.

The increase in the adsorption percentage with rise in adsorbent dosage is due to increase in active sites on the adsorbent and thus making easier penetration of the metal ions to the sorption sites (Subbaiah et al., 2011), then there was no further increase in adsorption for either metal ions. This may be due to overlapping of adsorption sites as a result of overcrowding of adsorbent particles (Kumar & Gayathri R, 2009).

### 3.7 Effect of Adsorbent Size

Effect of adsorbent size on adsorption of H.M.I. was investigated by conducting different experiments at different adsorbent diameter (0.075-1.2mm), while the other conditions of experimental work were kept constant at pH (2.97), rotation speed (600 rpm), adsorbent dose (1g), contact time (15min), temperature (25<sup>0</sup>C), and initial concentration (50 ppm). The experimental data are presented in Figure (7).

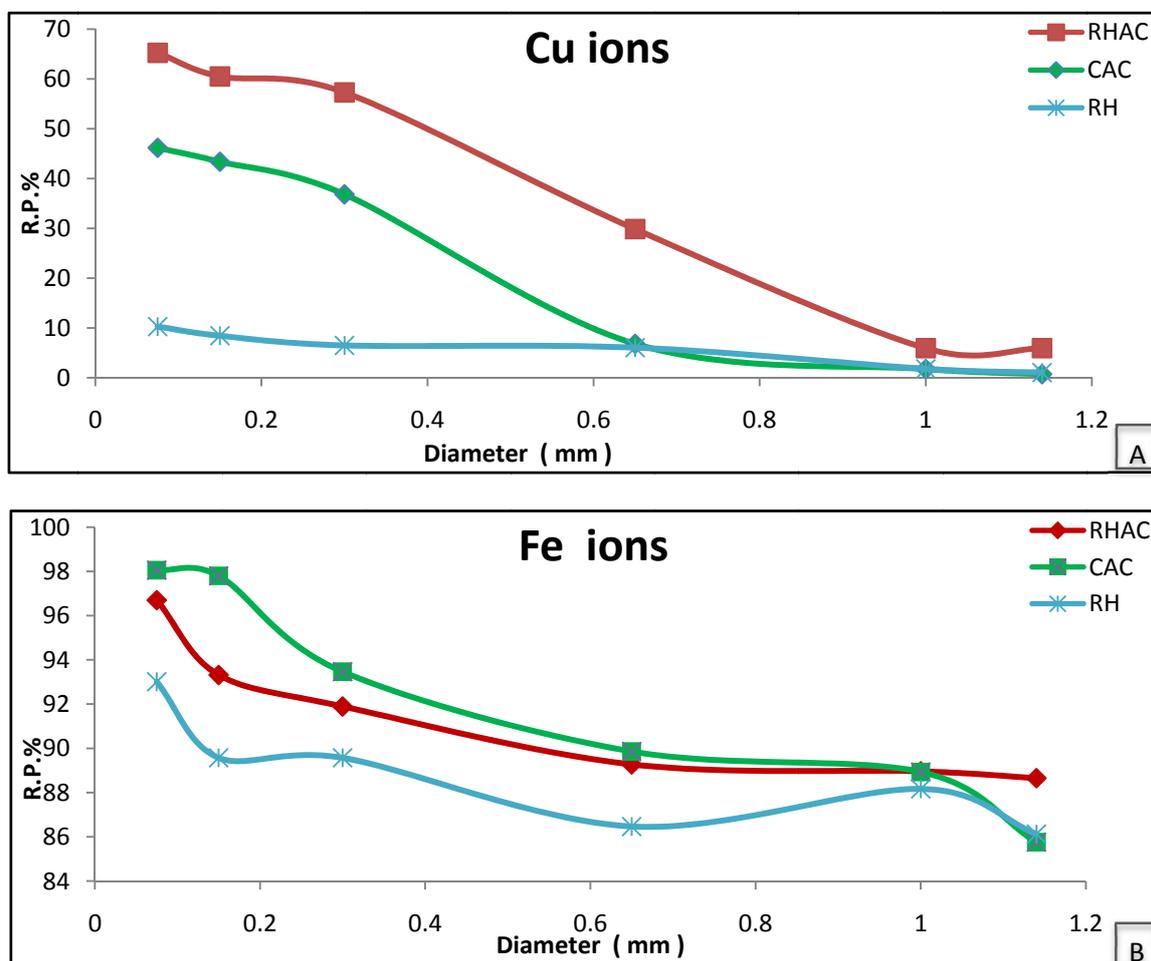


Figure 7. Effect of adsorbate diameter on the adsorption of heavy metals ions

The results shown that the R.P. of H.M.I. increased with decreasing particle size. The particle size of the adsorbent is very important factor. The increase in adsorption capacity may be

attributed to large surface area of the smallest particle size of adsorbent and large number of active sites (Safa & Bhatti, 2011).

### 3.8 Effect of Rotation Speed

The effect of rotation speed on H.M.I. adsorption was investigated. The experimental work was done under the conditions of constant temperature (25°C), adsorbent dose (1g), particle size (0.3), PH(2.95), initial concentrations (50 ppm), contact time(15min) and rotation speed (0-1500 rpm), the experimental data are presented in Figure 8.

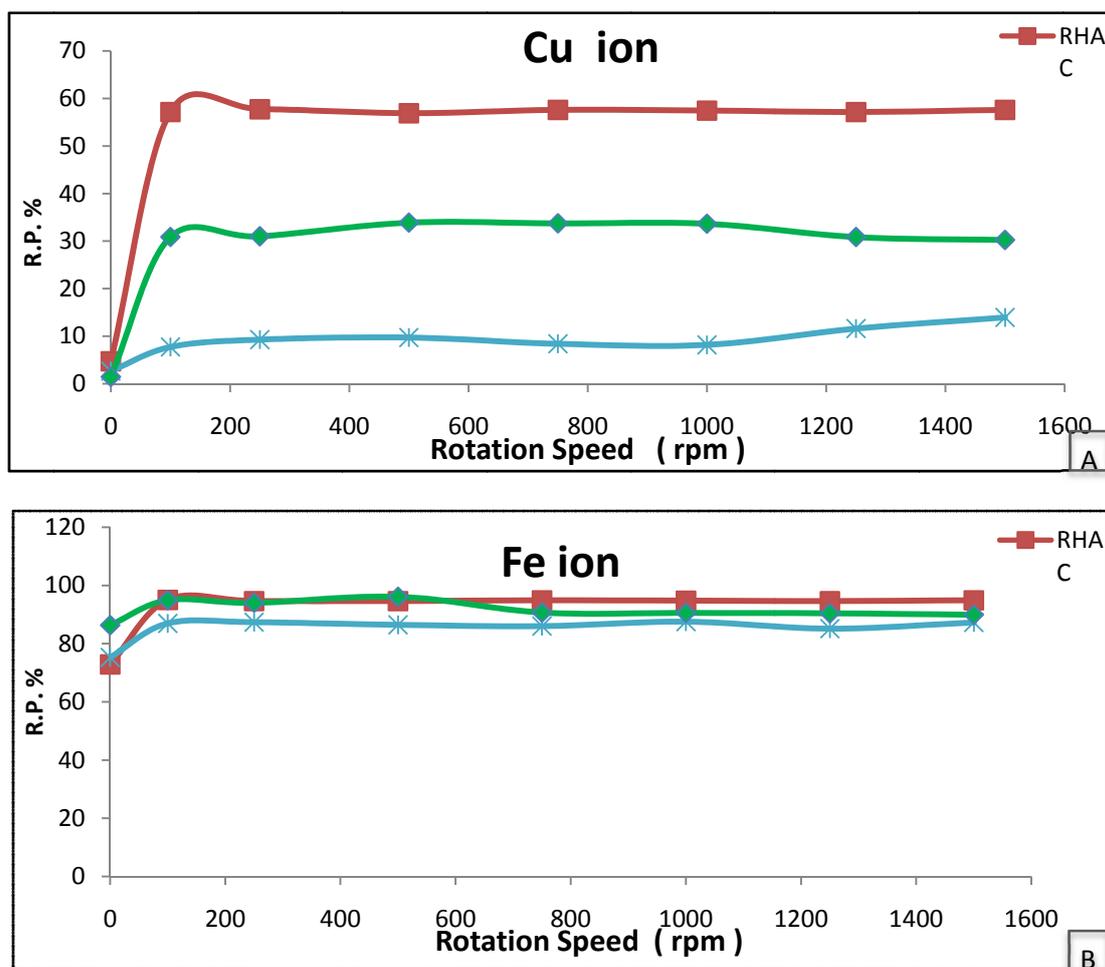


Figure 8. Effect of rotation speed on the adsorption of heavy metals ions

The adsorption capacity increased with increasing rotation speed. Due to the fact that rotation facilitates proper contact between the metal ions in solution and the adsorbent binding sites and consequently promoting effective transfer of adsorbate ions to the sorbent sites (BADMUS et al., 2007). Without rotation speeds the resistance to mass transport is in the bulk solution, and a thin liquid film surrounding the adsorbent particles offered resistance to mass transport by diffusion. As the rotation speeds increased, there would be decrease in the

thickness of the boundary film thereby decreasing the effect of film diffusion (Yacob, 2011).

### 3.9 Fourier Transform Infrared Spectroscopy (FTIR)

From the figures 9 to 11 and results shown below which describe the FTIR analysis, there are a different in functional groups was scattered in surface of adsorbent material, which shows the different in adsorption efficiencies of heavy metal ions. The FTIR spectra obtained were in agreement with the results reported in the study carried out on activated carbons by other researchers (Alhamedand Bamufleh, 2008; Sharma et al., 2011; Markovska & Lyubchev, 2007).

The different offunctional groups on the surface of RACH, RH and CAC were explained following:

The spectra displayed the following bands on the CAC surface:

2350-2400  $\text{cm}^{-1}$ :  $\text{C}\equiv\text{C}$  stretching vibrations, 1600-1800  $\text{cm}^{-1}$ :  $\text{C}=\text{O}$  stretching vibrations and 1400-1600  $\text{cm}^{-1}$ :  $\text{C}=\text{N}$ ,  $\text{NO}_2$  stretching vibrations.

The spectrum of RH shows functional groups from O-H ( $3404\text{cm}^{-1}$ ), C-H ( $2920\text{cm}^{-1}$ ), C=N ( $1677\text{cm}^{-1}$ ), C-O ( $1000-1060\text{cm}^{-1}$ ), C=C ( $1649-1512\text{cm}^{-1}$ ) and Si-O ( $1081\text{cm}^{-1}$ ). While the spectrum of RHAC shows functional groups from O-H ( $3409\text{cm}^{-1}$ ), C=C ( $1660\text{cm}^{-1}$ ), Si-O, C-O ( $1100\text{cm}^{-1}$ ) and Si-H ( $800\text{cm}^{-1}$ ).



Figure 9. FTIR spectra of CAC before (1) and after (2) adsorption processes

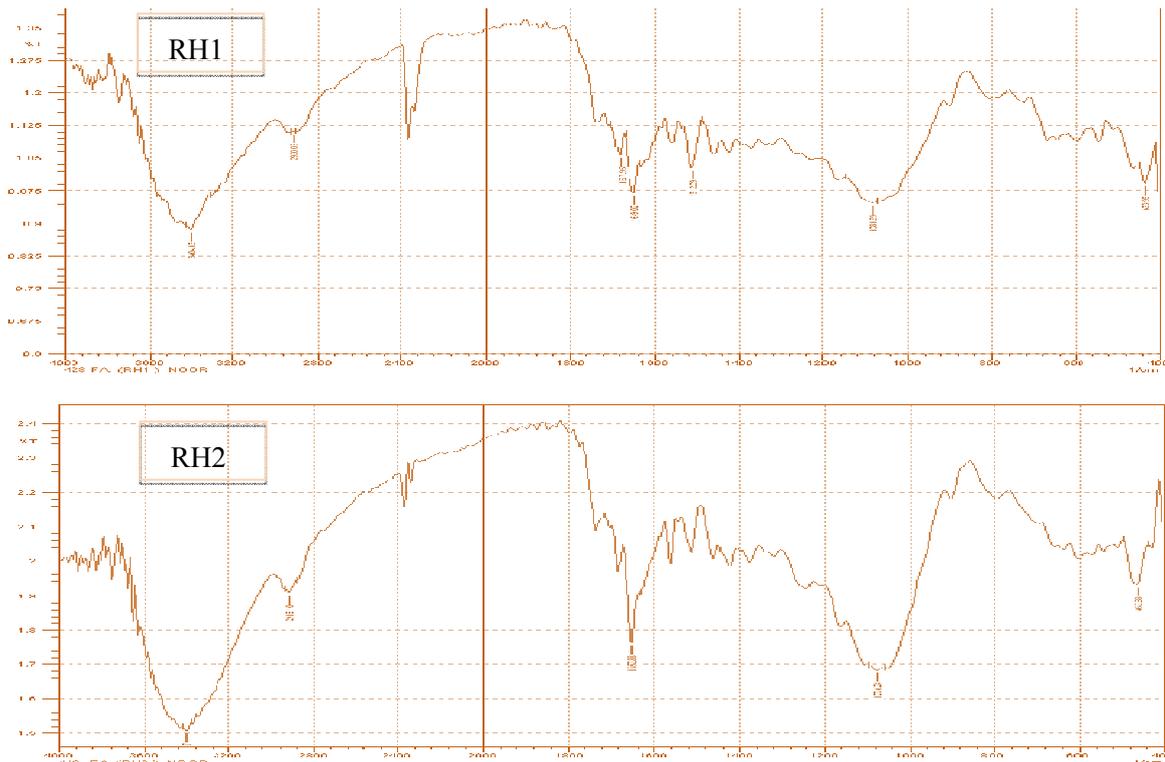


Figure 10. FTIR spectra of RH before (1) and after (2) adsorption processes

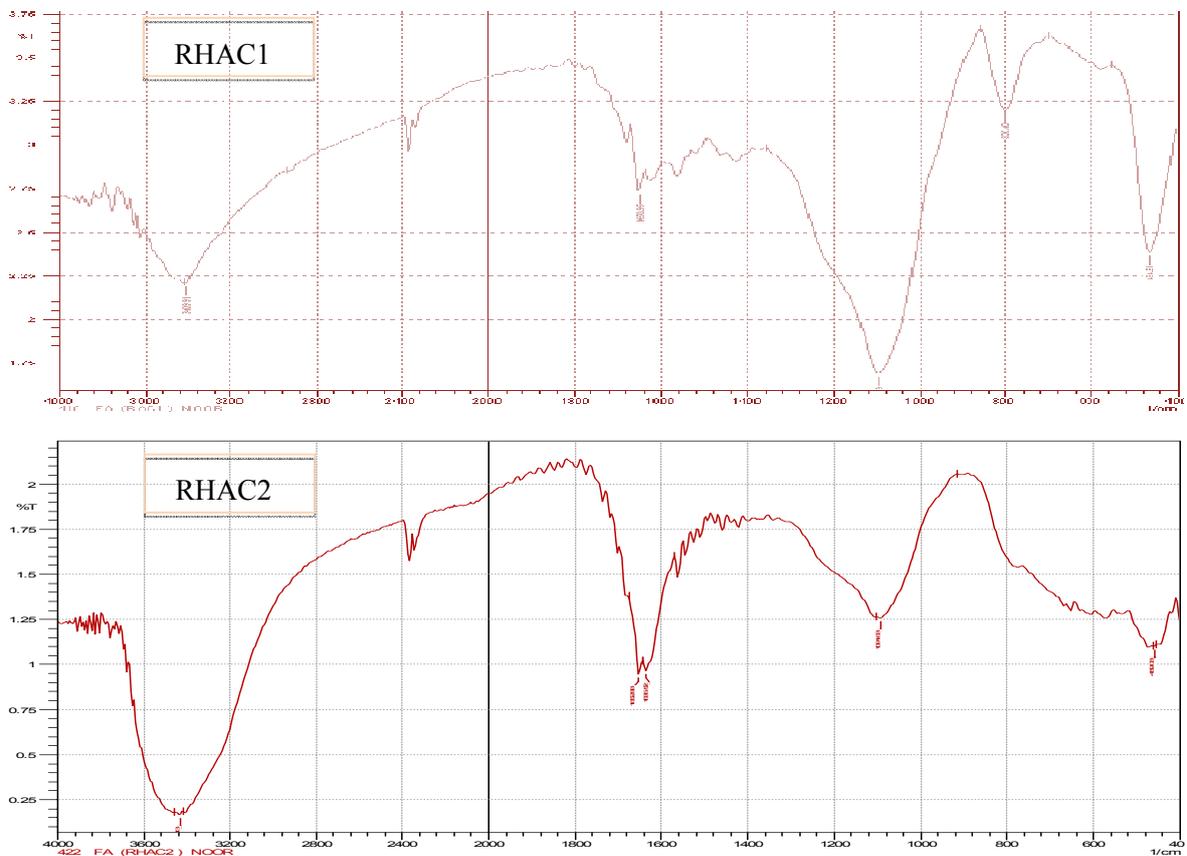


Figure 11. FTIR spectra of RHAC before (1) and after (2) adsorption processes

#### 4. Conclusions

The adsorption capacity increased with increases in contact time until reached the saturation time then remained constant. The pH parameter is very effected on the adsorption capacity which increased with increasing pH until reached the optimum pH value. The efficiency of adsorption increased with rotation speed due to provide a good contact between the H.M.I. in solution and the adsorbent active sites. The adsorption capacity decreased with increased in adsorbent size and initial concentration of heavy metal ions, while the adsorption capacity increased with increasing temperature, and adsorbent dose. The FTIR results showed that a different in functional groups was scattered in surface of adsorbent material, that causing the different in adsorption efficiencies of heavy metal ions.

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