

# Characterization and Comparative Adsorption Studies of Activated Carbon and Silica Prepared From Rice Husk

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**Abstract** – Due to industrialization there is an excessive release of toxic compounds to the environment. Since these substances are responsible for the health hazards in the bio system it is inevitable to remove them from the system. However expensive technologies have failed to get adapted. The research has brought rice husk in its different forms which is locally available as well as an effective adsorbent. In the present study rice husk is heated at different temperatures and two different adsorbents were obtained. Both of them can be used as adsorbents. Batch studies are carried out as functions of dosage, contact time, concentration and pH. Equilibrium data obtained for the dosage used in hexavalent chromium adsorption shows that Freundlich isotherm is the best fit isotherm for the adsorbent. The column studies were also conducted which shows as the rate of flow is kept low and bed depth high the length of used adsorbent bed is greater. The characterization of the adsorbent in its raw, as carbon and ash are obtained. The SEM and EDS studies were carried out.

**Key Words:** Adsorption, Bioadsorbent, Rice husk carbon, Rice husk ash, Characterization, Batch studies, Adsorption isotherm, Column studies

## 1. INTRODUCTION

The present century has seen rapid industrial growth. May that be metal plating, mining operations, tanneries or alloy industry the effluent from manufacturing industries consist of heavy metal. Such heavy metals are non-biodegradable hence can cause severe health disorders. Therefore the effluents being generated must be treated before discharging it into any water body.

Chromium occurs in two forms, trivalent and hexavalent chromium. Trivalent chromium is most stable and is less dangerous compared to hexavalent chromium. Hexavalent chromium is the most toxic form of chromium. It is with +6 oxidation state. Its application lies in electroplating, leather tanning, textile manufacturing and others. Studies show that there is an increase in lung cancer rate among the people exposed to high levels of chromium. The National Toxicology Program conducted at California, USA conducts research on male and female rats. They showed malignant tumours in the oral cavity.

Adsorption is the process of adhesion of atoms, ions, or molecules from a gas, liquid, or dissolved solid to a surface.

This creates a film of the adsorbate on the surface of the adsorbent. This is different from absorption, where in the adsorbate is dissolved by or permeates a liquid or solid respectively. However the commercially available adsorbents are still expensive to be used by a small scale industry. Hence agricultural byproducts are used as adsorbents in this study.

Rice husk is an agricultural waste generated in the areas of rice production. The annual rice production provides 20% of husk. Dry rice husk contains 70-85% of organic matter (lignin, cellulose, sugar) and the remainder contains silica which is available in the membrane. The chemical composition of rice husk differs from sample to sample. On an average, husk contains 20% silica with some metallic impurities which increases to 90% on ashing.

## 2 EXPERIMENTAL

### 2.1 Adsorbent preparation

The available rice husk was washed to remove suspended particles, dust and dirt. Further it is dried for 24 hours in an oven for 100°C. Then it is leached in the presence of HCl of 0.3% dilution. The dry husk is taken in a crucible and placed in the furnace at 500°C for 1 hour 15 minutes. The golden brown husk would have turned black (Rice husk carbon) retaining the longitudinal structure of the husk. Then dry husk is taken in a crucible and placed in the furnace at 900°C for 1 hour 15 minutes where the carbon content is lost and the silica content increases (Rice Husk Ash). The material is powdered and sieved to obtain material less than 75µm size.

### 1.2 Adsorbate

The experiments demand synthesized stock of hexavalent chromium. A stock solution of such demand with 1000mg/l was prepared by dissolving accurately 2.826g of A.R. potassium dichromate crystals in one litre of double distilled water. The batch studies were carried out using 10mg/l concentration of hexavalent chromium. Working solutions of 50mg/l, 100mg/l, 150mg/l and 200mg/l is prepared by diluting the 1000mg/l stock as per requirement. To obtain 100ml of different concentrations 5ml, 10ml, 15ml and 20ml of the 1000mg/l is diluted in 100ml of double distilled water which gives 50mg/l, 100mg/l, 150mg/l and 200mg/l of solution.

### 2.3 Batch Studies

After the selection of the adsorbent, the adsorption studies were carried out for various parameters like dosage of adsorbent, contact time, initial concentration, pH. To begin with the batch studies stock solution was prepared for chromium and zinc. The rice husk carbon and rice husk ash prepared are sieved and only particles below 75 microns are chosen for experimental purpose. The adsorption percentage was determined by

$$\%R = \frac{C_o - C_f}{C_o} * 100$$

Where  $C_o$  is initial concentration of metal ion and  $C_f$  is the final concentration of metal ion after adsorption. The results are plotted on a line graph.

### 2.4 Column Studies

The column studies were performed in a dynamic system. The adsorbent used was rice husk ash. The adsorbent was sieved and sizes above 200mm were considered for the experiments. Rice husk ash was filled in a glass column with internal diameter of 2.5cms and height of 20cms. The waste water collected from a small scale industry was used in the column study. This was basically the wash water collected and diluted to 30ppm. The waste water was filled in the container kept as a certain height above the glass column. The rate of the flow was set to 4ml/min. The bed height is varied to know the effect of the bed depth on adsorption of chromium. For study purposes two different bed depths are considered.

## 3. RESULTS AND DISCUSSIONS

### 3.1 Characterization before adsorption

Rice husk carbon has greater surface area compared to raw rice husk. The graph represents the presence of carbon in highest amount. The pore size of the ash is smaller and the number of pores are more than those in rice husk carbon. The highest element is silicon which is present by 72%.

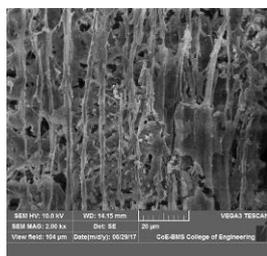
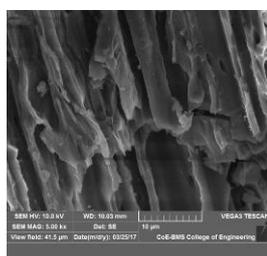


Fig -1: SEM of RHC

Fig-2 : SEM of RHA

### 3.2 Batch Studies

#### 3.2.1 Effect of adsorbent dosage

The effect of dosage of each of the adsorbent was studied by taking dosage from 0.2g to 2.2g of adsorbents in 50ml of 10mg/l of adsorbate run on a mechanical shaker at 150rpm. The effect is shown graphically as in Chart 1. The percentage

removal increases to 62% using 2g of RHC whereas using 2g of RHA the percentage increases to 75%.

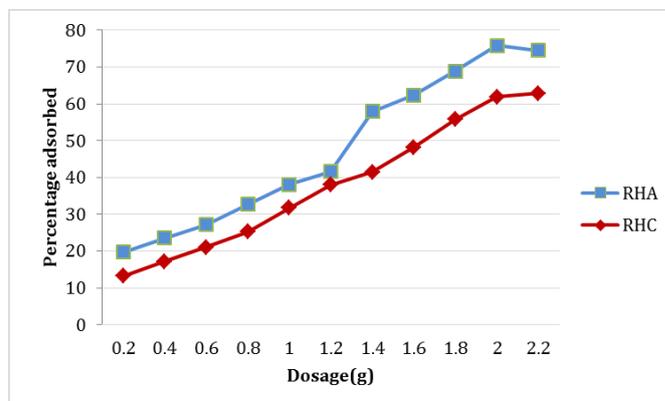


Chart- 1: Variation of adsorption efficiency with varying adsorbent dosage using (a) RHC (b)RHA

#### 3.2.2 Effect of contact time

As the contact time increases from 30minutes to 150 minutes there is an increase in adsorption. At the end of 150 minutes the RHA would have adsorbed 90% of the metal ion which is higher than that by RHC. This is represented as below

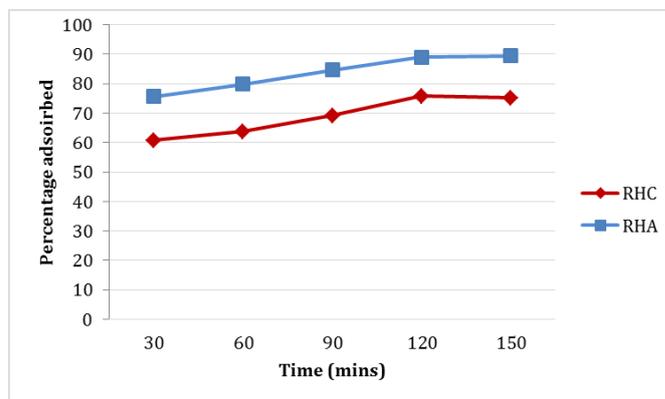


Chart-2: Variation of adsorption efficiency with varying contact time using (a) RHC (b)RHA

#### 3.2.3 Effect of concentration

As the concentration increases the adsorptive capacity decreases. The concentration ranging from 50mg/l to 200mg/l is tried. Both the adsorbents adsorb below 16% of the adsorbate after the concentration reaches 200mg/l.

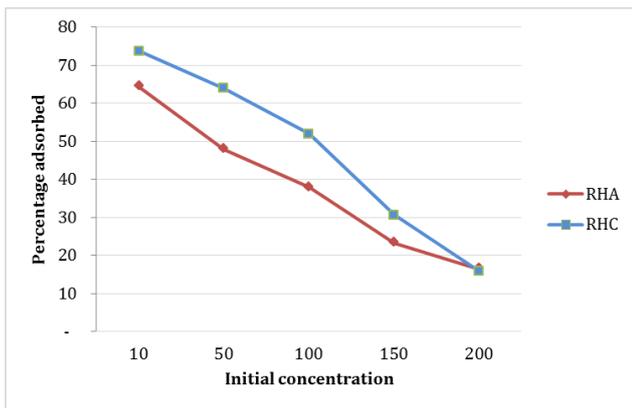


Chart-3: Variation of adsorption efficiency with varying initial concentration using (a) RHC (b)RHA

### 3.2.4 Effect of pH

The acidic environment increases the adsorptive capacity of the adsorbents. The adsorption increases to 95% when RHA was used at a pH goes below 4. However RHC also being a good adsorbent is capable of adsorbing upto 86% of the metal ion from the adsorbate.

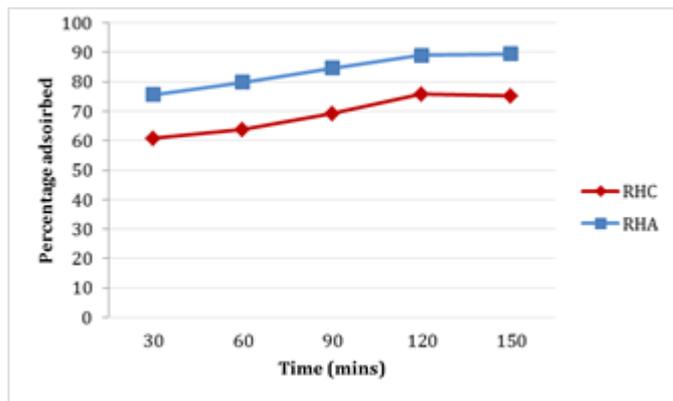


Chart-4: Variation of adsorption efficiency with varying pH using (a) RHC (b)RHA

### 3.3 Adsorption Isotherms

These are the graphs plotted for the amount if adsorbate adsorbed on the surface of adsorbent and the pressure at constant temperature. The types are Langmuir and Freundlich isotherms.

#### 3.3.1 Langmuir isotherm:

The assumption made in the isotherm is that all the sites on the adsorbent have equal affinity for the adsorbate and one adsorbate particle binds to a single site on adsorbent. Based on this theory following equation is derived

$$\theta = \frac{KP}{1 + KP}$$

where  $\theta$  is the number of site covered, K is the equilibrium constant for distribution and P represents the pressure.

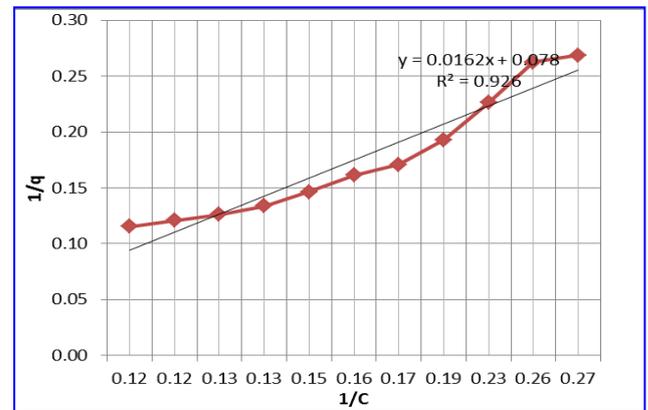


Chart-5: Langmuir adsorption plot of Cr adsorption by RHA

#### 3.3.2 Freundlich isotherm:

This isotherm is derived from the Langmuir isotherm. It states that there exists difference in affinities for different adsorbates.

$$\frac{x}{m} = k_f P^n$$

Where x is the mass of the gas adsorbed and m is mass of adsorbent at pressure P and k is measure of affinity of adsorbate n describes the affinity for the adsorbate.

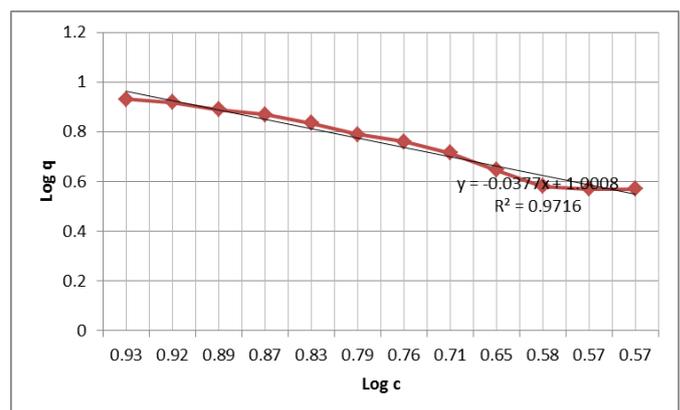


Chart-6: Freundlich adsorption plot of Cr adsorption by RHA

### 3.4 Column studies:

The operating flow rate was taken as 4ml/min based on Mass Transfer Zone. The bed depth was varied. The graph below shows that as the bed depth increases the time available for adsorption increases. Thus there is an increase in adsorption. The adsorption decreased after 150 minutes of contact time. As the bed is made acidic the adsorption has increased again by almost 20%.

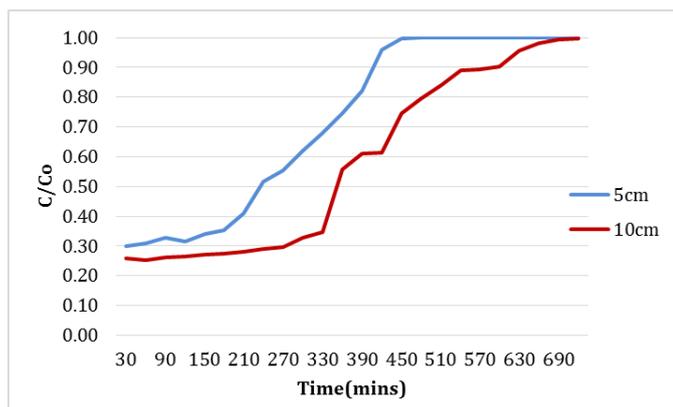


Chart-7: Variation of adsorption using RHA with increase in time and bed depth

### 3.5 Characterization after adsorption:

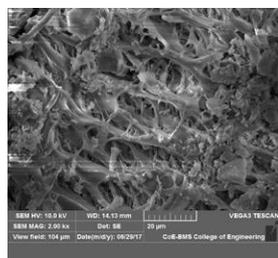


Fig-3: SEM of RHA after Cr adsorption

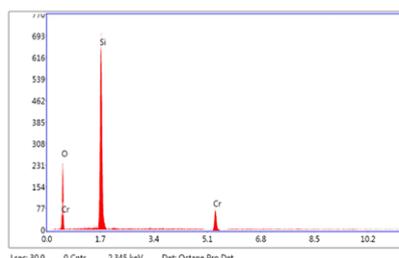


Fig-4: EDS of RHA after Cr adsorption

The SEM and EDS analysis after adsorption shows the presence of chromium due to adsorption.

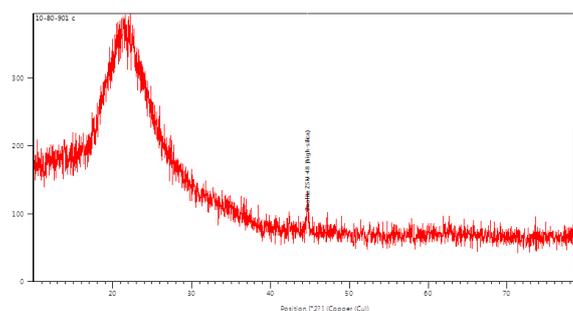


Fig-5: XRD graph of RHA showing the peak of silica

When the XRD analysis were carried out for the RHA sample it was found that the peak obtained was for SILICA. The EDS composition and XRD analysis shows that the material is made up of silica for about 98%. Thus the ash formed which consists of 98% of silica in it can also be termed as rice husk silica.

### 4. CONCLUSIONS

The leaching of adsorbent helped the hydrolysis of the epidermis. The adsorbents heated at 500°C and 900°C produced RHC and RHA respectively. The characterization of the adsorbent shows the porous structure and the changes in the structure as the temperature increases. Optimization of the operational parameters was conducted through batch studies, after which 2g of adsorbent was determined as optimum dosage which exhibited highest adsorption at 150 minutes when the pH was below 4. The Freundlich isotherm seems to fit. For a low rate of flow it was found that higher the bed height better is the adsorption. However the bed length lasted for 250 hours efficiently.

The comparative studies have shown that the rice husk is an economical adsorbent both as carbon and silica. In a lower pH environment RHA was capable of removing 92-95% of the heavy metal ion.

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