Preparation of thermally and chemically activated charcoal from coconut shell and adsorption studies for iron removal from drinking water

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Abstract. Iron is one of the contaminants that reduces potability of water. As per the Safe Drinking Water Act Secondary Standards, permissible concentration of iron in drinking water is 0.3 ppm. Iron content in the water available in industrial area is too high. Natural cleaning methods can be adopted to reduce iron concentration. Activated charcoal prepared from coconut shell can be effectively used as an adsorbent for the removal of iron from drinking water. This project aims on the preparation of thermally and chemically activated charcoal from coconut shell and its characterization. This work also focused on the adsorption studies using the prepared charcoal by varying parameters such as adsorbent dosage, initial concentration and shaking time. Langmuir and Freundlich isotherms, as well as pseudo first order and pseudo second order has been studied. Adsorption data is well fitted with Freundlich isotherm and pseudo second order kinetics.

1. Introduction
Developing countries and third world countries are facing potable water supply problems because of inadequate financial resources [5]. The cost of water treatment is increasing, and the quality of river water is not stable. Water contains excessive amounts of iron and can stain clothes, discolor plumbing fixtures, and sometimes add a ‘rusty’ taste and look to the water. Surface water generally does not contain large amounts of iron, but iron is found frequently in water systems that use groundwater.
The Safe Drinking Water Act Secondary Standards (aesthetic, not health related) for iron in drinking water is 0.3 ppm. If water contains more than 0.5 ppm iron, proper remedial measures should be taken to reduce the concentration of iron to a safe level. Our body has a limited capacity to excrete iron, which means it can easily build up in organs like liver, heart and pancreas. This is dangerous because iron is a potent oxidizer and can damage your body tissues contributing to serious health issues including cirrhosis, liver cancer, cardiac arrhythmias, diabetes, Alzheimer’s disease, bacterial and viral infections [3,4].

2. Materials and Methods

2.1. Materials required
Chemicals such as HCl and HNO₃ were used for chemical activation of coconut shell. FeSO₄ is used for the preparation of stock solution. 1,10 Phenanthroline is used for analysis in UV-VIS spectrophotometer.
2.2. Pretreatment of raw material
Coconut shell is used as the raw material for the production of activated carbon[1,2]. The raw material is cleaned and then dried in a hot air oven at 110 °C for 6 hours to remove moisture (Figure 1).

![Figure 1. Production of powdered activated carbon](image)

2.3. Thermal activation
Dried coconut shell is heated in a muffle furnace at 900 °C for 4 hours. The product is cooled and powdered using ball mill. Sorting according to particle size is done by using a standard set of sieves and sieve shaker [6].

2.4. Chemical activation
Dried and cleaned coconut shell is soaked in 2 separate beakers containing HNO₃ & HCl respectively for 12 hours. The product is heated at a temperature of 450 °C in a muffle furnace. Charcoal obtained is pulverized using a ball mill. The product obtained is sieved in a standard set of sieves using a sieve shaker for 20 minutes. The particles from different sieves are retained and stored.

2.5. Preparation of stock solution
Accurately weighed 2.72 g of FeSO₄ is dissolved in distilled water in a 1000 ml standard flask to prepare 1000 ppm iron solution which acts as the stock solution. Solutions of required iron concentrations for adsorption study can be prepared using this stock solution.

2.6. Calibration data
Samples of known iron concentration (0 ppm, 10ppm, 20ppm, 30ppm, 40ppm, 50ppm) are prepared. These samples are analysed using a UV-VIS spectrophotometer to obtain corresponding absorptivity. A relation between iron concentration (in ppm) and absorptivity is deduced to form a calibration curve helps to find out the unknown iron concentration.

2.7. Effect of adsorbent dosage
The weight of thermally and chemically activated charcoal was varied from 0.2 to 1 g/L keeping pH at 7.0, 10 ppm as initial concentration and contact time as 60 minutes. Five samples of solutions were
prepared. Adsorption was done by adding different concentrations of thermally and chemically activated charcoal. Samples were filtered and the effect of adsorbent dosage was found out.

2.8. Effect of initial concentration
The effect of initial concentrations for the removal of total iron (in terms of percentage removal) on both thermally and chemically activated charcoal was studied by keeping pH of solution as 7.0 and then fixed dosage of adsorbent of 0.4g/l was added and shaking time was 60 minutes.

2.9. Effect of contact time
The experiments were carried out at different contact times 20min, 40min, 60 min, 80 min, 100 minutes. The adsorbent dose was 0.4 g/L, initial concentration was 10ppm at a pH of 7.0. The equilibrium is reached within the first 60 min of shaking time. The experiment was repeated for chemically activated (HCl and HNO₃) adsorbents to find the effect of shaking time.

3. Results and Discussion

3.1. Characterization of the prepared sample
3.1.1. Fourier transfer infrared spectroscopy of thermally and chemically activated sample. In case of thermally activated charcoal, it shows five major adsorption bands with peaks at 3450.02 cm⁻¹, 1555.85 cm⁻¹, 1388.88 cm⁻¹, 1005.07 cm⁻¹, 559.23 cm⁻¹. The band at 3450.02 cm⁻¹ is due to the absorption of water molecules as result of an O-H stretching mode of hydroxyl groups and adsorbed water. The bands at 1555.85 cm⁻¹ and 1388.88 cm⁻¹ are due to C-H bending bonds, while the remaining peaks represent the C-C bonds in the fingerprint region [7].

The activated carbon by the inclusion of HCl shows five major peaks at different frequencies. New bonds were formed due to the addition of chemical similar to the thermally activated sample. Both the chemically activated shows O-H stretching bonds at 3441.27 cm⁻¹. Also C-C stretching bonds were formed by chemical activation which is more predominant in HCl activated sample 3015 cm⁻¹. By chemical activation C=C stretching bonds were formed in HCl activated samples at 1609.32 cm⁻¹. Also C-H stretching bond were present in these sample without much deterioration. The different peaks between the 1000 cm⁻¹ and 1300 cm⁻¹ represents the C-O stretching in acids which present in the chemically activated sample (Figure 2 and 3).

![Figure 2](image-url) FTIR images of thermally activated sample
3.1.2. **Scanning electron microscopy of thermally and chemically activated sample.** The images magnified at 10μm and 2μm shows that number of pores and size of pores are greater in case of chemically activated charcoal than the thermally activated one. Since the number of pores plays a vital role in the adsorption process, it is evident that the chemically activated charcoal will be more effective in treating drinking water for iron removal than the thermally activated one.

![Figure 4. 2μm magnified image of thermally activated charcoal](image)

3.1.3. **X-Ray diffraction of thermally and chemically activated sample.** X-ray diffraction pattern of the thermally activated and chemically activated samples are shown in Figure 6 and Figure 7. The diffraction peaks are observed at the diffraction angle of 2θ = 29.5°, 34.6° and 39.4° respectively which corresponds to carbon. While the rest of the other peaks that are observed at the diffraction angle of 44.5° corresponding to sodalite, and sodium silicate, respectively. The two active carbon samples showed two broad diffraction peaks located at 2θ = 30°-40° and 40°-50° which revealed an amorphous structure that was irregularly stacked by carbon rings and useful for generating an adsorbed gap. Here a sharp peak observed at 44.5° may be due to the presence of HCl used during the carbon activation process. Samples contain two broad diffraction peaks and can be attributed to the presence of carbon.

![Figure 5. 2μm magnified image of thermally activated charcoal](image)
3.2. Calibration data

The calibration data obtained which relates the absorptivity and iron concentration can be fitted into a calibration curve as shown in Figure 8. This calibration curve can be utilized to find the unknown iron concentration corresponding to the known absorptivity readings.

3.3. Effect of adsorbent dosage

For thermally activated and chemically activated adsorbents, maximum percentage removal of iron is found to be 0.4 g/L. HCl activated sample are more efficient than HNO$_3$ and thermally activated
sample. At a dosage of 0.4g/l, HCl removal is about 78.13 % of iron while the thermally activated shows an efficiency of 67.41 %.

![Graph showing adsorbent dosage vs removal percentage](image1)

**Figure 9.** Effect of adsorbent dosage

### 3.4. Effect of contact time
From the graph (Figure 10) the coconut shell charcoal is very effective to remove the iron content in the water. Increasing the initial concentration, the amount of iron removal decreases. It is observed from the figure that the concentration above 10 ppm of charcoal can marginally decrease the removal of iron from the system. Hence 10 ppm of coconut shell charcoal can be taken as the initial concentration. As the initial concentration increases the iron removal decreases. This is due to less adsorption of iron by the adsorbent by increasing the concentration between adsorbent and adsorbate.

![Graph showing initial concentration vs removal percentage](image2)

**Figure 10.** Effect of Initial concentration

### 3.5. Effect of initial concentration
Figure 11 indicates the variation of adsorption with constant time and it indicates the efficiency of HCl activated charcoal over HNO₃ and thermally activated charcoal. Maximum adsorption is attained at 60 minutes while further increase in contact time doesn’t affect the adsorption process. Maximum percentage removal of iron from drinking water.
4. Conclusions
Charcoal prepared from coconut shell can be used as a natural iron removal agent for water treatment. SEM, XRD and FTIR analysis shows that adsorbent become more suitable for iron removal by chemical activation process. Chemically activated charcoal is found to be more effective in iron removal than the thermally activated charcoal for potable water treatment. HCl is found to be more effective activating agent than HNO$_3$ for chemical activation process. Adsorbent dosage, initial concentration and contact time are found to be three important factors affecting the adsorption process. For the maximum adsorption, contact time between sample and the adsorbent is obtained as 60 minutes. The percentage removal of iron is higher at lower initial concentration and with an adsorbent dosage of 0.4g/L. The chemically activated charcoal can reduce the iron concentration to a safe limit. Freundlich isotherm well fits for this adsorption. The equilibrium data well matches with pseudo second order kinetics. Production of charcoal can be achieved at low cost and this can be forwarded as a profitable business idea in future. Coconut shell is inexpensive and readily available. Thus, this study provides a cost-effective means for removing iron from drinking water.

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