Preparation of Activated Carbon by Using Chemical Treatment Method

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Abstract

Corn is a significant crop all around the world. Asia is the second major production region. The corncob is the waste generated during processing corn. It is proposed to convert corncob into activated carbon, which is very useful to treat the dye effluent from wastewater. In this project work activated carbon was prepared from corncobs. Fresh, air-dried, corncobs were first cracked and crushed and them the corncobs were carbonized in a muffle furnace at 500°C for about 10 minutes. The resulting carbonized corncobs were activated by using chemical treatment. In this research, "activation" is carried with sulphuric acid and nitric acid of (0.5 M, 1.0 M, 1.5 M, 2.0 M) concentrations were used. The corncob carbonized carbons were also activation with alkalis (NaOH, KOH) of (0.5M. 1.0 M and 1.5 M). And then, the physical and chemical properties of activated carbon were determined and used in removal of iron from water.

Keywords, Carbonization, Activation, Corncobs, Activated Carbon, Iron Content

Introduction

Activated carbons are carbons of highly microporous form with both high internal surface area and porosity, and commercially the most common adsorbents used for the removal of organic compounds from air and water streams. Any cheap material with high carbon content, low inorganic can be used as a raw material for the production of activated carbon. Basically, there are two different processes for the preparation of activated carbon: physical activation and chemical activation. Physical activation involves carbonization of a carbonaceous material followed by the activation of the resulting char at a temperature between 1073K and 1373K in the presence of suitable oxidizing gases such as carbon dioxide or steam.

Activated carbon is an excellent adsorbent and thus is used to purify, decolorize, deodorize, de-toxicate, filter or remove the salts. They are used as catalyst or catalyst supports. The adsorbent properties of activated carbon are essentially attributed to their large surface area, a high degree of surface reactivity, universal adsorption effect and favourable pore size. These unique characteristics are dependent on the type of raw material employed and method of activation. Basically, there are two methods of activation, Physical and chemical activation. Activated carbon can be made from many substances containing high carbon content such as coconut shells, walnut shells, coal, wood, bagasse etc. The raw material has a very large influence on the characteristics and performance of activated carbon and plays a major part in determining its ability to adsorb certain molecular species. In the present work corncobs were used to prepare activated carbon. Corncob is suitable for preparing micro porous activated carbon due to its excellent natural structure and low ash content. Preparation of Activated carbon was done by carbonization followed by activation which is described below. Carbonization is the process for the conversion of an organic substance into carbon or a carbon containing residue through pyrolysis or destructive distillation. Activation is a carefully controlled oxidation process to develop a porous carbon structure. The idea behind activation is not only to increase the diameter of the pores that were formed during carbonization process but also to create some new porosity [2]

The aims of this project work are

- (1) to prepared the activated carbon from waste corncobs
- (2) to study the properties of activated carbon
- (3) to use the resulting activated carbon in iron removal from ground water

Literature Review

Background of Study

In the recent times, there is the dear need and search for various methods of conversion of agricultural residues to more useful products like activated carbon. The activated carbons from the agricultural residues are subsequently used for various industrial applications such as in adsorption studies. Activated carbon with desired characteristics can be obtained from solid agricultural wastes if carefully controlled processes of dehydration, carbonization and activation of the material undertaken. Such activated charcoal is used in gas purification, medicine, sewage treatment, air filters in gas masks, filters in compressed air and many applications. They are also applied in vodka of organic impurities since the activated charcoal does not bind well to alcohols, the percentage of alcohol is not significantly affected, while the carbon will bind and remove many organic impurities which can affect taste, colour and odour. The activated carbon was obtained from these carbonaceous material or organic precursor have had their hydrocarbons removed to increase its powers of adsorption. There has been an increasingly high demand for activated carbon following the vast applications of activated carbon in industrial processes such as solvent recovery, gasoline vapour emission control canisters in automobile, clean-up of corn sugar solutions and removal of tastes and odours from water supplies, vegetable and animal fats and oils, alcoholic beverages chemicals and pharmaceuticals. As a decolourant, activated carbon, with its very large surface area and pore volume, is hundreds of times more efficient than charcoal and at least 40 times more than bone black. The amount of material adsorbed by activated carbon is surprisingly large, amounting frequently to from a quarter to an equal weight of such vapours as gasoline, benzene, carbon tetrachloride. Activated carbon is able to absorb practically any organic solvent at about 35 dC and release it when heated to 120 °C or higher for solvent recovery. Many reports have appeared on the development of activated carbon from cheaper and readily available agricultural materials. Throughout the world, agricultural by-products such as wood, coal, shells etc are some of the materials used in activated carbon production [4].

Carbonization

Carbonization (or carbonisation) is the term for the conversion of an organic substance into carbon or a carbon-containing residue through pyrolysis or destructive distillation. It is often used in organic chemistry with reference to the generation of coal gas and coal tar from raw coal. Coke is produced commercially by carbonization of coal, either at high or low temperatures. The main purpose in the carbonization of coal is to produce coke, and any chemicals produced are of secondary importance. Carbonization is a process in which a fuel is heated without air to leave solid porous carbon. Initially it was obtained from the carbonization (heating) of coal, which produces coke, combustible gas, and a number of by-products, including benzene.

The carbonization stage may be decisive in charcoal production even though it is not the most expensive one. Unless it is carried out as efficiently as possible, it puts the whole operation of charcoal production at risk since low yields in carbonization reflect back through the whole chain of production as increased costs and waste of resources [5].

Activated Carbon Development

Activated carbon is nothing but carbon produced from carbonaceous source materials like corncob, nutshells, peat, wood, coir, lignite, coal and petroleum pitch. It can be produced by any one of the following described processes:

Physical Reactivation

Physical activation was carried out at 650 °C in a horizontal tube furnace for 3 hours. By this process precursor is developed into activated carbons using gases.

This is generally done by using one or a combination of the following processes:

Carbonization: Material having appreciable carbon content is pyrolyzed at temperature ranging between 600–900 °C, in the absence of oxygen (usually in inert atmosphere with gases like argon or nitrogen) using a furnace.

Activation/Oxidation: in this process raw material or carbonized material is exposed to oxidizing atmospheres (carbon monoxide, oxygen, or steam) at temperatures above 250°C, usually in the temperature range of 600–1200°C ^[5].

Chemical Reactivation

Before carbonization, the raw material can be impregnated with certain chemicals. The chemical needs to be typically an acid, strong base, or a salt (ammonium sulphate, phosphoric acid, potassium hydroxide, sodium hydroxide, zinc chloride, respectively). The role of the activating agent is to improve the pore size of the activated carbon in order to improve its adsorption capacity. After impregnation, the raw material needs to be carbonized at lower temperatures (450–900 °C). It is believed that the carbonization / activation step proceeds simultaneously with the chemical activation. Chemical activation is preferred over physical activation owing to the lower temperatures and shorter time needed for activating material^[5].

Steam Activation

The use of steam for activation can be applied to virtually all raw materials. Varieties of methods have been developed but all of this shares the same principle of initial carbonization at 500°C to 600°C followed by activation with steam at 800°C to 1100°C. Since the overall (converting carbon to carbon dioxide) is exothermic, it is possible to utilize this energy and have a self-sustaining process. Initial, gasification of the carbonize material with steam occurs and is shown in the following reaction known as water-gas reaction^[5].

$$C + H_2O = CO + H_2$$
 (- 31 Kcal)

This reaction maintains temperature by partial burning of the CO and H_2

$$CO + 1/2O_2 = CO_2 (+ 67 \text{ Kcal})$$

 $H_2 + 1/2O_2 = H_2O (+ 58 \text{ Kcal})$
 $C + O_2 = CO_2 (+ 94 \text{ Kcal})$

Materials and Methods

Raw Materials

Waste corncobs were purchased from the market, Hmawbi Township, Yangon Region. The required chemicals such as H_2SO_4 and HNO_3 , NaOH and KOH (Analar grade , England made) were purchased from

Academy Chemical shop, 28^{th} Street , Pabedan Township, Yangon Region.



Figure (1) Raw Corncobs

Experimental Procedure Carbonization of Corncob

Corncobs were cleaned thoroughly and dried in sun for a week. All these cleaned corncobs were dried in an oven at 70 °C for one hour. Then, the dried corncobs were crushed, ground and sieved by 35 mesh screen.

100 g of ground corncobs were placed in a preweighed crucible and carbonized in a muffle furnace at 500 °C for 10 minutes. The carbonized corncob was ground into powder and sieved by 200 mesh screen. The powder carbonized corncob was stored in a airtight container.

Activation of Carbonized Corncob Carbon

Acid activated carbon was prepared by treating 100 g of powder carbonized carbon (at 500 °C) with 75 ml of sulphuric acid (0.5 M, 1.0 M,1.5 M and 2.0 M) in the 100 ml beaker for 24 hour. After that the content were filtered through the Whatmann filter paper No.41and the carbon residue was washed with water until no more acid was left and dried. The same procedure was also carried by using nitric acid (0.5 M, 1.0 M, 1.5 M and 2.0 M) and both carbons were termed as acid activated carbon.

Corncob carbon was also activated by using alkali such as sodium hydroxide and potassium hydroxide of (0.5 M, 1.0 M,1.5 M and 2.0 M). Then, the yield percents of the two acid activated carbons were recorded.

Effect of Acid Concentration on Yield Percent of Activated Carbon

The yield percent of activated carbon were determined by using different concentrations of acid (0.5 M, 1.0 M, 1, 5 M, 2.0 M) and the resulting yield percents were recorded in Table (1).

Effect of Alkali Concentration on Yield Percent of Activated Carbon

The yield percent of activated carbon were determined by using different concentrations of alkali (0.5

M, 1.0 M, 1, 5 M, 2.0 M) and the resulting yield percents were recorded in Table (2).

Determination of Characteristics of Activated Carbon

- 1. Moisture Content
- 2. Ash Content
- 3. Volatile Matter Content
- 4. Fixed Carbon Content
- 5. Iron Adsorption Activity
- Surface Morphology



Fig (2) Scanning Electron Microscope (SEM)

Results and Discussions

In this project paper, the corncobs were obtained from the Hmawbi Township as the waste from the local markets. The activated carbon was prepared from the waste corncobs. In the preparation of activated carbon, firstly the corncobs were carbonized at the temperature 500°C for 10 minutes and the carbonized carbon was activated by using sulphuric acid. The same procedure was also carried out by using nitric acid both the procedures were carried out with different concentration of acids and alkalis.

The characteristics of the resulting activated carbons were determined. Table (1) represents the characteristics of activated carbon and it was found that the fixed carbon contents of activated carbons were higher than the literature value. There were little ash content in activated carbon and the other characteristics were also in acceptable range.

The effects of acid and alkali concentrations on yield percent of activated carbons are shown in Tables 2 and 3. It was found that yield percent became lower at the higher concentration. The yield percents of activated carbon were obtained by activation different acids and alkalis, it was found that 36.4 % was the optimum yield percent for sulphuric acid and 37.5 % was the optimum yield percent for nitric acid and 39.58 for NaOH and 38.5 for KOH.

In this project paper the resulting activated carbons were used in removal of iron from ground water and Table (4) shows the resulting iron contents of before and after treatment with activated carbon.

The surface morphology of activated carbons were also determined and the results were represent on Figures (3 - 6). In the results, the pore sizes were more clear and wider. The resulting products acid activated carbons of H_2SO_4 and HNO_3 are shown in Figures (7 and 8) receptively and the Figures (9 and 10) represent the alkali activated carbons of NaOH and KOH receptively.

Table (1) Effect of Acid Concentration on the Yield Percent of Prepared Activated Carbon

Sr. No	Acid Concentration (Molarity)	Yield Percent of Activated Carbon(% w/w)		
		Activated with H ₂ SO ₄	Activated with HNO ₃	
1.	0.5	35.8	35.3	
2.	1.0	35.2	36.0	
3.	1.5 *	36.4	37.5	
4.	2.0	35.6	37.2	

^{*} The optimum concentration of acid

Table (2) Effect of Alkali Concentration on the Yield Percent of Prepared Activated Carbon

Sr. No	Alkali Concentration (Molarity)	Yield Percent of Activated Carbon(% w/w)		
		Activated with NaOH	Activated with KOH	
1.	0.5	36.7	35.9	
2.	1.0*	39.58	38.5	
3.	1.5	36.5	36.8	

^{*} The optimum concentration of alkali

Table (3) Characteristics of Prepared Activated Carbon Carbon

Sr.	Characteristics	Activated Carbon (Activating with various acids and alkalis)			Literature	
No		H ₂ S O ₄	H N O ₃	Na OH	КОН	Value
1.	Yield (% w/w)	36.4	37. 5	39.5 8	38.5	24.3*
2.	Moisture Content (% w/w)	4.5	4.7	4.5	4.8	3-9.8**
3.	Volatile Matter Content (% w/w)	49.3	48. 7	49.8	49.7	63.5**
4.	Ash Content (% w/w)	9.8	9.7	9.2	9.6	15.6**
5.	Fixed Carbon Content (% w/w)	36.4	36. 9	36.5	35.9	11.2**

^{*} http://www.scienta-atricana uniportjournal-info

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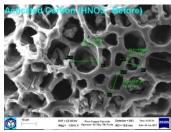


Fig (3) Surface Morphology of Activated Carbon Using Nitric Acid (Before Adsorption)

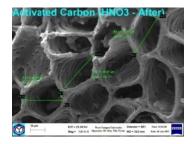


Fig (4) Surface Morphology of Activated Carbon Using Nitric Acid (After Adsorption)

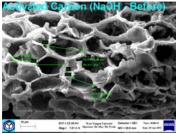


Fig (5) Surface Morphology of Activated Carbon Using Sodium Hydroxide (Before Adsorption)

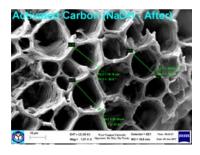


Fig (6) Surface Morphology of Activated Carbon Using Sodium Hydroxide (After Adsorption)

Table (4) Comparison of Iron Content between Before and After Using Activated Carbon

		Iron Content (mg/L)		
Sr. No	Type of Activated Carbon	Before Treatment with Activated Carbon	After Treatment with Activated Carbon	
1.	Activated Carbon (Using Sulphuric Acid)	3.75	0.45	
2.	Activated Carbon (Using Nitric Acid)	3.75	0.43	
3.	Activated Carbon (Using Sodium Hydroxide)	3.75	0.44	
4.	Activated Carbon (Using Potassium Hydroxide)	3.75	0.42	



Fig (7) Activated Carbon (Using Sulphuric Acid)



Fig (8) Activated Carbon (Using Nitric Acid)



Fig (9) Activated Carbon (Using Sodium Hydroxide)



Fig (10) Activated Carbon (Using Potassium Hydroxide)

Conclusion

In this project work, carbonized carbon was prepared from waste corncobs and the resulting carbon was activated by using sulphuric acid and nitric acid. It was found that the acid concentration (1.5 M) was the most suitable content for H_2SO_4 and HNO_3 . The optimum yield percent were 36.4 % and 37.5 % respectively. . It was also found that the alkali concentration (1.0 M) was the most suitable content for NaOH and KOH. The optimum yield percent were 39.58 % and 38.5 % respectively. The characteristics of the acid activated carbons were within in acceptable range. It was also found that the adsorption property of active carbons was effectively used in removal of iron from ground water.

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