Effect of Phosphoric Acid Concentration on the Characteristics of Sugarcane Bagasse Activated Carbon

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Abstract. Impregnation method is one of the crucial steps involved in producing activated carbon using chemical activation process. Chemicals employed in this step is effective at decomposing the structure of material and forming micropores that helps in adsorption of contaminants. This paper explains thorough procedures that have been involved in producing sugarcane bagasse activated carbon (SBAC) by using 5%, 10%, 20%, 30% phosphoric acid (H₃PO₄) during the impregnation step. Concentration of H₃PO₄ used in the process of producing SBAC was optimized through several tests including bulk density, ash content, iodine adsorption and pore size diameter and the characteristic of optimum SBAC produced has been compared with commercial activated carbon (CAC). Batch study has been carried out by using the SBAC produced from optimum condition to investigate the performance of SBAC in removal of turbidity and chemical oxygen demand (COD) from textile wastewater. From characteristic study, SBAC with 30% H₃PO₄ has shown the optimum value of bulk density, ash content, iodine adsorption and pore size diameter of 0.3023 g cm⁻³, 4.35%, 974.96 mg/g and 0.21-0.41 µm, respectively. These values are comparable to the characteristics of CAC. Experimental result from the batch study has been concluded that the SBAC has a promising potential in removing turbidity and COD of 75.5% and 66.3%, respectively which was a slightly lower than CAC which were able to remove 82.8% of turbidity and 70% of COD. As a conclusion, the SBAC is comparable with CAC in terms of their characteristics and the capability of removing contaminants from textile wastewater. Therefore, it has a commercial value to be used as an alternative of low-cost material in producing CAC.

Keywords: Sugarcane bagasse, activated carbon, impregnation method.

1. Introduction
Activated carbon is one of the adsorption agents that have best efficiency in removing contaminants from wastewater [1]. However, due to the non-affordable market price associated with intensive regeneration process, activated carbon is not favourable to manufacturer. This difficulty has led to search for the use of cheap and efficient alternative materials such as rice husk, bamboo, sugarcane bagasse and tamarind kernel powder [2]. Therefore, this study has been designed focusing on the use of sugarcane bagasse to produce activated carbon, as a means of replacing the expensive conventional activated carbon.
Impregnation method is one of the steps involved in chemical activation where raw materials are immersed fully or partially in certain acid. Chemicals employed in chemical activation (ZnCl$_2$, H$_3$PO$_4$, and H$_2$SO$_4$) are effective at decomposing the structure of the raw material and forming microspores [3]. Figure-1 shows the pores characterization of an activated carbon.

![Micro pores][1]

**Figure 1.** Pores characterization of an activated carbon.

Even though activated carbon from sugarcane bagasse has been investigated for removal of contaminants from wastewater by previous researchers, there are a few studies on treatment efficiency using different concentration of acid in producing activated carbon. Thus, the concentration of phosphoric acid used in impregnation method and its effect towards the performance of SBAC in removing turbidity and COD is highlighted in this study.

Activated carbon from sugarcane bagasse emerges as one of the alternatives that has been introduced to replace conventional activated carbon. Besides of its low cost, it is also easily available [4]. Crushed sugarcane produces 3 tonnes of bagasse for every 10 tonnes of it. This will consume one part of the landfill area. As landfill scarcity is one of the big issues that Malaysia confronting today, thus reuse of bagasse as raw material for producing filter media help in reducing production and disposal of organic waste at landfill.

2. Materials and Methodology

2.1 Material preparation

Sugarcane bagasse is taken from small-medium industries around Parit Raja, Johor. Raw bagasse was washed with tap water to remove contaminants and excess sugar and dried in dry oven at 105 °C overnight. Dried bagasse was impregnated with 5%, 10%, 20%, 30% phosphoric acid (H$_3$PO$_4$) for another 24 hours. In impregnation process, raw bagasse was fully immersed in acid with the ratio of 1g bagasse to 300 ml acid. After impregnated for 24 hours, impregnated bagasse is then carbonized at 500 °C in furnace for two hours. Produced activated carbon was fully immersed in acid with the ratio of 1g bagasse to 300 ml acid. After impregnated for 24 hours, impregnated bagasse is then carbonized at 500 °C in furnace for two hours. Produced activated carbon was washed with pure water until the pH of activated carbon reached 3-7. Activated carbon produced was then sieved to 60 μm to 2.00 mm by sieve analysis testing, referring to ASTM C136-06 Standard Test Method for Sieve Analysis of Fine and Course Aggregates [5]. A workflow of study is shown in Figure-2. Figure-3 shows activated carbon produced from sugarcane bagasse.
Figure 2. Workflow of study.

Figure 3. Sugarcane bagasse activated carbon.
2.2 Material characterization
All activated carbon produced from various concentrations of acid were undergone several tests to determine their bulk density, ash content, adsorption capacity (iodine), pore size diameter and morphology of the activated carbon produced. SBAC with the best properties was chosen to be continued in batch study.

2.3 Bulk density
Bulk density (expressed as g cm-3) is defined as the mass of a unit volume of the sample in air, including both the pore system and the voids among the particles [3]. Generally, an adsorbent with bulk density need to be generated frequently because it can hold adsorbate per unit weight [2]. In this test, 10 ml measuring cylinder was dried in over at 110 °C for 30 minutes. Powdered activated carbon was filled into cylinder with three layers and tapped about 300 times for each layer until its fully compacted and reweighed [5,6].

\[ \text{Bulk density, } D_b = \frac{1000 \text{m}}{\text{v}} \]  

Where,
- \( D_b \) = bulk density in g cm-3
- \( m \) = mass of sample in g
- \( v \) = volume of sample in measuring cylinder

2.4 Ash content
Ash content is a test that provide the total ash content although in some cases, a more detailed analysis of the ash is needed. It is the residue that remains when carbonaceous portion is burned off [7]. This test is carried out using method from ASTM [8]. Empty crucible is ignited in muffle furnace at 500°C for 15 minutes. Allow crucible to cool to room temperature in dessicator and weigh to nearest 0.1 mg. 1.0 g of activated carbon is transferred into crucible and ignite for another 1 hour. Crucible is allow to cool in dessicator to room temperature and reweighed again [5,6].

\[ A_c = \frac{F-G}{B-G} \times 100 \]  

Where,
- \( A_c \) = ash content in %
- \( F \) = Mass of crucible and ashed sample in g
- \( B \) = Mass of crucible and dried sample in g
- \( G \) = Mass of empty crucible in g

2.5 Iodine adsorption
Iodine adsorption indicates the internal surface area of carbon where in many cases, the result is close to Brunauer-Emmett-Teller (BET) surface area [3]. It has been established that, the iodine number in mg/g gives an estimate of the surface area in m2/g [9], and measures the porosity of pores with dimensions between 1.0-1.5nm [10]. According to method from AWWA [11,12], 10 ml of 5% hydrochloric acid is boiled with 1g of activated carbon. 100 ml of 0.10N iodine solution is added and shaken vigorously for 30 seconds. After filtration, filtrate is titrated with 0.10N sodium thiosulphate solution with starch as indicator.

\[ I_n = \frac{X}{M} A \]  

Where,
- \( I_n \) = iodine adsorption in mg/g
\[ I_n = \frac{v-x}{y} x \left( \frac{v}{w} \right) x M(126.9) \] (4)

Where,
- \( I_n \) = iodine adsorption in mg/g
- \( v \) = volume of iodine solution used for titration (ml)
- \( x \) = Volume of sodium thiosulphate used for carbon free aliquot (ml)
- \( y \) = Volume of sodium thiosulphate used for blank solution (ml)
- \( w \) = mass of activated carbon in g
- \( M \) = molarity of iodine solution used

2.6 Pore size diameter and morphological view
Morphological view and pore size is measured using Field Emission Scanning Electron Microscopy (FESEM). FESEM is microscope that works with electrons instead of light. FESEM was used to visualize very small topographic details on the surface area of SBAC. In this study, a range of pore size from selected surface area were used to observe whether the pore is well-developed or ruptured.

2.7 Batch study
Batch study was conducted to measure performance of activated carbon produced. Here, 12.5g of activated carbon with the best properties was chosen to neutralize pH, COD and turbidity from 250 ml textile wastewater with contact time of 24 hours. A comparable study is conducted towards CAC. Figure-4 shows detail on the batch study.
3. Results and Discussion

3.1 Bulk density
Determination of bulk density depends on the shape, size and density of individual particles and are useful in estimating tank or packing volume [3]. Figure-5 shows bulk density of activated carbon for impregnation using 5%, 10%, 20%, 30% of H3PO4. The trend shows that bulk density was decreased with the increasing of acid concentration. According to Yakout and El-Deen[13] the trend indicates the increasing of carbon particle in activated carbon along with increasing of acid concentration. As the lowest bulk density indicates a good adsorbant, thus it can be said that 30% H3PO4 has the lowest bulk density from the others with 0.3023 g cm\(^{-3}\).

3.2 Ash content
Ash content indicates the quality of activated carbon in terms of mechanical strength. Having a large number of ash in activated carbon, indicates the poor strength of carbon produced. Ash content is also related to carbon yield percentage. The increment of ash content in activated carbon will decrease the carbon yield. Figure-6 shows ash content from SBAC. From figure-6, 30% H\(_3\)PO\(_4\) has the lowest ash contents with 4.35%, hence having the best mechanical strength.
3.3 Iodine adsorption
Practically, iodine number is the amount of iodine in milligrams, adsorbed per gram of carbon when the equilibrium concentration (Ce) of iodine is 0.02M. Figure-7 illustrates iodine number for various concentration of H₃PO₄. According to AWWA [12], activated carbon recommended for water treatment must have iodine number ranging from 600 to 1100 mg/g. From the figure-7, it shows that all produced AC comply with the standard set by AWWA [12]. Moreover, iodine number of SBAC from 30% H₃PO₄ is comparable to commercial activated carbon.

![Figure 7. Iodine number of SBAC and CAC.](image)

3.4 Pore size diameter and morphological view
Generally, observing pore size diameter is conducted to see how well the chemicals employed in impregnation method decompose raw material and develop micropores. Porosity is one of the important properties in activated carbon. The more micropores form in activated carbon, the bigger the surface area, hence the performance of activated carbon in absorbing contaminants from wastewater is more efficient. Table 1 shows morphological view and pore size diameter of SBAC and CAC. It can be concluded that all SBAC produced having a comparable pore size diameter with CAC (0.14-0.92 mm).

4. Conclusions
From this study, SBAC with 30% H₃PO₄ has shown the optimum value of bulk density, ash content, iodine adsorption and pore size diameter of 0.3023 g cm⁻³, 4.35%, 974.96 mg/g and 0.21-0.41 µm, respectively. It was chosen to have the best performance in removing contaminants from textile wastewater. This study proves that SBAC performance is comparable to CAC. This is shown when SBAC is able to achieve 75.5% and 66.3% in removal of turbidity and COD, respectively which was a slightly lower than CAC which able to remove 82.8% of turbidity and 70% of COD. This study also will allow a reappraisal of optimizing acid used in impregnation method of sugarcane bagasse activated carbon production.

The production of activated carbon by using agricultural waste is more economical compared to commercially activated carbon. This research will also assist nation guidelines and educational drives to alleviate the water pollution awareness. Indirectly, this study will immerse a degree of awareness of the “waste to wealth” concept as well as pleasant drainage could invite eco-tourism in Malaysia.
Table 1. Morphological view and pore size diameter.

| Morphological view | Pore size diameter (µm) | Morphological view | Pore size diameter (µm) |
|--------------------|-------------------------|--------------------|-------------------------|
| Raw sugarcane bagasse | no pores was detected | 10% H₃PO₄ | 0.15 – 0.66 |
| 5% H₃PO₄ | 0.28 – 0.92 | 0.34 - 0.85 |
| 30% H₃PO₄ | 0.21 – 0.41 | CAC | 0.14 – 0.92 |

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