Characterization of Activated Carbon from Granulated Sugar

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Abstract

Activated carbon that potentially acts as an adsorbent was prepared from granulated sugar. The activated sugar was prepared through a dehydration process followed by a chemical activation process with a concentrated NaOH solution. To comprehend the properties of prepared ready carbon, characterization of the carbon was needed to be conducted. The conducted methods for prepared activated carbon were FTIR for detecting present functional groups attached to carbon, BET to identify the porous structure of synthesized carbon, SEM method as a morphological indicating method on synthesized carbon, and carbon thermal stability characteristic information from the TGA method. It was found that the prepared activated carbon contained various functional groups. The distinguished functional groups presented were carboxylic acid, free alcohol, NH$_2$, and SO$_3$. The readily activated carbon showed a rough surface to enhance the adsorption process on the synthesized carbon from the SEM characterization method. BET on the readily activated carbon revealed that the activated carbon exhibited type IV isotherm, consisting dominantly of mesopores and micropores as a minority. TGA characterization method showed that the thermal stability of carbon was significantly unstable in the range of 100°C and around 200°C. It implies that the prepared activated carbon contained OH as one of its functional groups. With all the characterization methods conducted on the readily activated carbon from granulated sugar, it was found that the prepared carbon had the potential to be utilized as an adsorbent. Moreover, it is suggested that the granular sugar-based carbon should be investigated in the following future study in the wastewater treatment process.

Keywords: Activated Carbon, Granulated Sugar, Mesopores, Micropores.

1. Introduction

Industrial waste is one of the severe problems in the era of industrialization. The industrial revolution, industrial and mining operations have been solved by many problems, including waste that may be toxic, flammable, corrosive, or reactive. This waste can have dangerous health and environmental consequences if not appropriately managed. It is produced at every stage in the production, use, and disposal of manufactured products.

Liquid waste treatment processes will vary for each waste; however, some general principles apply to the treatment process for aqueous wastes or wastes based on water. Wastes with high carbon loads (high BOD or COD) will often be treated with either aerobic or anaerobic biological functions depending on the trash. Non-aqueous wastes are treated, recovered for re-use, or destroyed depending on the type of the waste. Management of industrial liquid wastes is a very open-ended topic due to the vast variety of liquid wastes [1].

The waste from the area varied between solid, liquid, and gas according to different kinds of industries. Disposal of these wastes is a big problem affecting the site and its surroundings for a long time. Dumping these wastes was done randomly in neighboring areas that damaged the natural resources; there are many signs of environmental degradation like soil contamination and health problems that indicate a dangerous situation [2] [3].

The underlying reason for the waste is not only from the production process but also for survival. Therefore, waste treatment must be carried out as well as possible from upstream to downstream because if this is not done, then the pollution threat will be fatal [4], [5]. The fundamental problem of handling and managing waste is the lack of knowledge of business actors from small industrial business groups. This later became a justification for the low awareness of small industrial business actors on the management of handling and processing waste [6].

Activated carbon is a carbonaceous material that is predominantly amorphous and in which the process of manufacturing and treatment develops a high degree of porosity. Every activated carbon has a memory that largely depends on the source and the preparation conditions[7]. Activated carbon can be manufactured from virtually all carbonaceous materials. However, agricultural wastes offer the most available and cheapest of all the known raw materials. Activated carbon is inexpensive and hence very widely used adsorbent[8], [9]. This waste can be a renewable source of activated carbon. Several uses are attributable to activated carbon, ranging from removing undesirable odor, color, taste, and other organic and inorganic impurities from domestic and industrial wastewater. The vast internal surface and pore
volume are developed during the preparation stages. The preparation involves two main steps, which are: carbon: carbonization of the carbonized product. The carbon is usually charred at a temperature below 600°C during the carbonization [10]. Activated carbon is an amorphous carbon with a surface area ranging from 300-3500 m²/g. It has been treated with steam and heat to have a powerful affinity for absorbing (adsorption) various materials with ahh ability of activated carbon. This is related to the internal pore structure, which causes activated carbon to act as an adsorbent. Activated carbon can adsorb certain gases, and chemical compounds or their adsorption properties are selective, depending on the size or volume of the pores and surface area. Activated carbon can be used in various pharmaceutical and food industries for filtering, deodorization, and taste. Petroleum chemical industry, water purifier, shrimp farming, sugar industry, gas purification, catalyst and fertilizer preparation, and fertilizer bon (AC) has been widely used in wastewater treatment for a long time. However, the applications of the AC are limited by the difficulties associated with their regeneration process after usage. Fortunately, the samples in this study could not only be regenerated many times but also maintained their adsorption efficiency. Herein, highly porous carbon has been prepared from white sugar using the acid dehydration method. The prepared carbon was activated in a nitrogen environment, which resulted in the formation of the activated sugar-based carbon [11][12]. Sugar has been regarded as an available and sustainable source of carbon. It has short-chain and soluble carbohydrates which can be extracted from most plants, especially sugarcane. Generally, white sugar is obtained from raw sugar through a purifying process to remove the molasses [13].

2. Literature Review

2.1. Activated Carbon
Carbon activation is usually achieved naturally or synthetically by the precursor carbonization process, followed by activation. During the carbonation process, carbon-based material is thermally degraded in an inert atmosphere; in which tottorolar devolatisation takes place. Activation is aimed to increase porous volume and its diameter size enhanced in the carbonization process and creating new porosities [14].

Activated carbon is usually applied in potable water production and wastewater treatment. The use of activated carbon in those processes is as adsorbent related to its large surface area, porous structure, and pore distribution that enhance adsorption rate[15].

2.2. Adsorption
The adsorption process is the phenomenon of enrichment from the gaseous or liquid phase at the interphase layer of solid material. The adsorbed material is called adsorbate, and the media where the adsorption process takes place is adsorbent [16]. There are two methods in the adsorption process: batch and dynamic methods. In the batch process the adsorbent is placed in solution and stirred in a range of time the the solution is separated from the adsorbent. In dynamic method, an adsorbent filled column is passed with solution containing adsorbate and then the adsorbent is separated by flowing the solution out of the column[17].

In general, there are two types adsorption, which are physical and chemical adsorption. In physical adsorption, it is reversible process that the binding between adsorbate and adsorbent is driven by Van der wall's force. Moreover, since the free adsorbate substance always exist, the reaction last for long time[18]. Chemical adsorption utilizes activation energy for its chemical reaction. The process is irreversible process, in which the bond between adsorbate and adsorbent is much stronger than in physical adsorption.

Adsorption process is dependent on several factors, such as agitation process, adsorbent characterization, adsorbate solubility, adsorbate molecular size, pH and temperature. All the factors affecting adsorption control the adsorption capacity, its adsorption rate, and the adsorption effectiveness[19].

3. Methods

3.1. Preparation of Activated Carbon
Granulated sugar, which is an easily accessible carbon source, was prepared for carbon preparation mixed with concentrated 96% (v/v) H₂SO₄ solution, which is chemical activation process[20], in stoichiometric amount. The mixing process was aimed to dehydrate the sugar from hydrogen and oxygen atoms. The dehydration process took place by stirring the solution at room temperature in a beaker where granulated sugar loses H and O atoms, left with C atoms that have properties of being in solid phase and black colored. The dehydration reaction was exothermic reaction and took place in a relatively short period of time[11]. Having obtained the carbon, it was then washed with NaOH in order to neutralize the residues of acids left in with carbon. The neutral carbon was then carbonized in oven at more than 100 °C for 8 hours for the activation process. Fig. 1 shows the activated carbon from granulated sugar as raw material.

![Fig 1. Activated carbon from granulated sugar](image)

3.2. Activated Carbon Characterization
In order to characterize the prepared activated carbon, there were several methods conducted. The aim of characterization of the material was to understand the properties of the prepared activated carbon. The characterization methods applied were TGA, FTIR, BET and SEM. The BET method characterization was done by using Micromeritics BET instrument that would give the information regarding activated carbon’s porous volume and its surface area. Besides, the types of pores existing in the carbon would also be revealed. The other method is SEM, in which the prepared activated sugar surface morphology would be presented. From this method, the shape and structure of
prepared activated carbon would be accessible so that one would know the adsorption could take place on the prepared carbon more effectively.

TGA method for prepared activated carbon characterization was conducted by flowing nitrogen above the material at temperature range from 100°C to 600°C. The percentage of mass loss of observing material would reveal the components in the material following its thermal stability information. In FTIR method characterization, the sample was analyzed by using FTIR instrument, which was SHIMADZU FTIR Spectrophotometer. Through this method, the existing functional groups are revealed based on the wave length.

4. Results and Discussion

4.1. TGA Characterization
The characterization of prepared activated carbon through TGA method was done and the result is shown in Figure 2. From Fig. 2, it can be seen that the material being observed exhibited significantly thermal instability in the range of 30 °C to 100 °C. This phenomenon implies the presence of OH[21].

![Fig 2. Thermal stability analysis of activated carbon from granulated sugar](image)

The presence of OH functional group would support the binding of other chemicals in adsorption process for activated carbon, such as Methylene Blue and Congo red that are usually used as waste water indicator [22].

4.2. FTIR Characterization
Prepared activated carbon was characterized with FTIR in order to comprehend all functional groups that were present in the prepared activated carbon. The results of FTIR characterization are shown in Fig. 3.

![Fig 3. Functional group analysis of activated carbon from granulated sugar](image)

From Fig. 3, it can be seen that there were several functional groups existed in the prepared activated carbon. It shows some essential functional groups such as SO, amino, carboxylic acid and OH groups. These functional groups imply potential application of prepared activated carbon for next studies for waste water treatment as adsorbent material, which are usually applying Methylene Blue and Congo Red as indicators of waste water [23].

4.3. BET Characterization
The BET surface area, mesopore volume and micropore surface area of the activated carbon were determined by the application of the Brunauer-Emmett-Teller (BET). The adsorbent pores are classified into three groups: micropore (diameter<2nm), mesopore (2–50 nm), and macropores (>50 nm). Fig. 4 shows that prepared carbon exhibited type IV isotherm as it resembled in [24]. Moreover, as it is show in Fig.5, the pore size distribution of the activated carbon exhibit one main peak. One of the peak is at 18.586 nm and the other peak at 27.2 nm; 34.3 nm; 50. 3 nm. Furthermore, the pore size distributions after 50.3nm are broadened.

![Fig 4. Nitrogen adsorption–desorption isotherms for activated carbon](image)
The effect of acid concentration on the BET surface area, micropore, mesopore volume, and total pore volume are given in Table 1. The total pore volume, \( V_{\text{total}} \), was calculated from nitrogen adsorption data as volume of liquid nitrogen at a relative pressure of 0.95.

![Fig 5. Pore size distribution from \( \text{N}_2 \) adsorption of activated carbon](image)

The micropore volume, \( V_{\text{micro}} \), was determined by DR method, and the mesopore volume, \( V_{\text{meso}} \), was obtained by the subtraction of micropore volume and total pore volume. As can be seen, the BET surface area and total pore volume increase with the increasing acid concentration.

| Parameters | \( S_{\text{BET}} \) (m\(^2\)/g) | \( V_{\text{Total}} \) (cm\(^3\)/g) | \( V_{\text{Micro}} \) (cm\(^3\)/g) | \( V_{\text{Meso}} \) (cm\(^3\)/g) |
|------------|-------------------------------|---------------------------------|-------------------------------|-----------------|
| Values     | 0.2911                         | 0.001393                        | 0.000189                      | 0.001204        |

### 4.4. SEM Characterization

Besides the previous characterization methods, the morphological properties of prepared activated carbon were determined with SEM. Fig. 6 shows the SEM images of carbon granulated sugar samples.

![Fig 6. SEM image for activated carbon](image)

It can be observed that the surface structure of carbon granulated sugar is like pores with a rough surface and relatively large pores. This means that the activated carbon under the nitrogen condition has significantly improved the porosity of the adsorbent.

### 5. Conclusion

Granulated sugar as it is relatively accessible to everyone has other potential to be used as activated carbon. Through the chemical and thermal activation process, activated carbon was synthesized from the granulated sugar. From its characterization process, the prepared activated carbon from granulated sugar showed the material has potential to be used as adsorbent in adsorption process, which is applicable in adsorbing Methylene Blue and Congo Red as commonly used materials in resembling waste water model for its treatment study.

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