Abstract: The idea of generating high-value practical materials, such as activated carbons, from agricultural wastes as a raw material has been a quite important trend recently due to its positive contributions to the environment and resource savings from biomass. In this paper, activated carbons prepared from durian husk waste by the KOH chemical activation method are studied. We focus on the effects of stages of the activating temperature on their properties. The optimum conditions for activation were a KOH/char ratio of 1:2 at the first and second activation process at the temperatures of 400 and 800 °C, respectively. The characterization results of Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and Brunauer–Emmett–Teller (BET) surface area showed that the obtained activated carbons have a high surface area and small pore size. The adsorption/desorption isotherms of the obtained activated carbons showed type I and type II isotherms. The chemical structure of obtained activated carbons did not show any variation in the surface functional groups. A feasible method to produce the activated carbons with a high surface area and high adsorption capability from durian husk waste was eventually demonstrated.

Keywords: activated carbons; durian husk; activation; waste utilizations; adsorption

1. Introduction

Durian is a one of most famous fruits consumed locally and exported in the Southeast Asia region, especially in Malaysia and Thailand. A large quantity of durian fruit is produced per year for commercialization [1]. However, it is well known that not the entire durian fruit is edible. The outer part of the durian skin normally weighs more than half of the total of this fruit, and this cannot be eaten and is thrown away. Large quantities of durian husk waste are being produced, which is becoming an environmental problem. Therefore, it is worthwhile to find a solution to this problem. Generally, durian husk waste is usually discarded at a landfill site and ends up in open burning, which can release harmful pollution into the air, increasing the air pollution to the environment [2,3]. In the past decade, the idea of utilizing biomass from agricultural wastes as a raw material for the production of valuable material has attracted the interest of global researchers.
Activated carbons, carbonaceous material with a large surface area and high porosity, are one of the most commonly obtained materials produced from agricultural wastes. In recent times, this material has received much attention from the scientific community as it is renewable, low-cost, and environmentally friendly. The surface area and pore size distribution are important factors in the determination of activated carbon’s performance, such as its adsorption and mechanical properties [4].

Recently, activated carbons with high surface area and pore volumes have been produced from various sources of agricultural wastes such as coal [5–7], coconut shell [8,9], sawdust [10,11], sugarcane bagasse [12], rice straw, and husk [13] plant materials [14–18], including agricultural industrial by-products such as coffee bean husk [19,20]. Among these precursors, durian husk waste is also a main source within agricultural industrial practice in the production of potential activated carbon due to its effectiveness and inexpensive cost as a raw material. In general, activated carbon is used in a wide range of environmental applications. In particular, it is a common material used as an adsorbent for adsorption or treatment in water purification, such as heavy metal removal in groundwater [21–23], wastewater treatment [24,25], and for the removal of volatile organic compounds (VOCs) [26], carbon dioxide [27], pigments, and odors [28]. It is well known that the potential efficiency of activated carbons depends on their surface area and active surfaces. Thus, it is very important to prepare activated carbon with a high surface area and high porosity in order to enhance its adsorption capacity due to these characteristics, which are particularly suitable for specific applications [29–31]. Currently, activated carbon is manufactured via physical or chemical stimulation processes, such as the physical activation of pyrolysis to produce char followed by steam gasification [32,33], and chemical activation, such as using solutions of zinc chloride [34], phosphoric acid [35], potassium hydroxide [36], sodium hydroxide [37], and so on. Besides this, the nature of raw materials has also commonly affected the pore structure and pore size distribution of activated carbon. A functional activated carbon will be obtained through a two-step process: (i) hydrothermal carbonization of the raw materials in an atmosphere without oxygen followed by the activation of the resulting char. In this stage, most of the non-carbon elements, such as hydrogen and oxygen, are removed in a gaseous form by pyrolytic decomposition, resulting in carbon with a fixed mass and a rudimentary pore structure; (ii) secondly, the activation step can be performed as follows: an appropriate amount of obtained char is mixed with an activating agent and then initiated by heating the samples in the furnace from room temperature until the desired temperature is reached. This step is employed to enlarge the diameter of fine pores and also to create new pores so that the adsorptive power of the carbonization product is enhanced [38–40]. It is well known that the activated carbon is not only used as an adsorbent for the removal of polluting molecules in the environment but is also widely used for supercapacitors’ electrode materials, catalyst carriers, and reinforcement materials [41–44]. Recently, several studies have reported about the method of activated carbon production, but there have been no studies into the effect of the activating temperature on the surface area and porosity structures of the obtained activated carbon. Therefore, it is worthwhile to further examine this parameter and how it affects the properties of activated carbon.

In this research, carbonized durian husk was used as a precursor for the production of activated carbon by chemical activation with potassium hydroxide; due to its cost effectiveness, it is used as an activating agent for producing activated carbon. The effects of the stages of the activating temperature on the properties of activated carbon were investigated. Then, a comparison of the iodine absorption capacity of activated carbon was performed to study the specific surface area and the amount of porosity in adsorption. In addition, the results of Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and Brunauer–Emmett–Teller (BET) surface area are investigated and discussed.
2. Materials and Methods

2.1. Materials

Durian husk (*Monthong varieties*) was collected from a local durian market in the Chonburi province of Thailand. The characteristics of durian husk waste are given in Table 1. Hydrochloric acid with a purity of 85% was purchased from Sigma–Aldrich (Germany). Potassium hydroxide and sodium hydroxide were purchased from Ajex chemical (Germany) and sodium thiosulfate of AR-grade was purchased from Loba Chemie (India). Other chemical agents were analytical grade purity and used as received without further purification.

Table 1. The characteristics of durian husk waste.

| Contents | Result (%) |
|----------|------------|
| Moisture | 30.34      |
| Ash      | 4.25       |
| Volatile | 76.70      |
| Carbon   | 21.10      |

2.2. Activated Carbon Preparations

As a first step, the durian husk waste was cut to 3–5 cm. Then, it was repeatedly washed with distilled water to remove dust and other inorganic impurities. After that, the clean durian husk was dried at a temperature of 100 °C for 24 h in the hot-air oven to reduce its moisture contents, and it was stored in a desiccator. In the carbonization process, the char was produced by carbonizing the samples of dried durian husk in a fixed-bed batch reactor in an atmosphere without oxygen. The reactor was filled with samples and was placed in a furnace (Model 30400, Barnstead Thermolyne, USA). The furnace was heated from room temperature to 600 °C, kept at that temperature for 3 h, and then cooled down to room temperature overnight. The obtained char was crushed in a crusher (Model ZM200, Restch, Germany) to obtain particles with sizes between 800 to 1000 µm and then stored in desiccators for further use. After the carbonization process, the mass yield of the char was calculated, and we found that the yield average was 73.58% of the total mass. The activation process of char was carried out as follows: the char was impregnated by soaking appropriate amounts of the char with different ratios of potassium hydroxide at room temperature for 24 h, as shown in Table 1. After the impregnation process was completed, we used into two processes for activations: (i) In Process I, denoted as DH1–DH3, the impregnated precursors were poured onto a porcelain disc and placed in the furnace for their first activation at a temperature of 400 °C for 1 h. Then, the activation process was carried out by adjustment to the second activation at a temperature of 600 °C for 3 h, and then the material was cooled down to room temperature. (ii) In Process II, defined as DH4–DH6 code samples, these activated carbons were heated to the second activation temperature of 800 °C for 3 h under the same conditions as in the Process I preparation. Details of the process conditions for preparing activated carbon are shown in Table 2.

Table 2. Sample identification, activation process conditions, and yield for activated carbon preparation.

| Samples | Carbonization Temperature (°C) | KOH:Char Ratio | First Activation Temperature (°C) | Second Activation Temperature (°C) | Yield (%) |
|---------|-------------------------------|----------------|----------------------------------|-----------------------------------|----------|
| DH1     | 600                           | 1:4            | 400                              | 600                               | 83.95    |
| DH2     | 600                           | 1:2            | 400                              | 600                               | 86.48    |
| DH3     | 600                           | 1:1            | 400                              | 600                               | 80.27    |
| DH4     | 600                           | 1:4            | 400                              | 800                               | 79.95    |
| DH5     | 600                           | 1:2            | 400                              | 800                               | 85.45    |
| DH6     | 600                           | 1:1            | 400                              | 800                               | 75.47    |

All activated carbons were washed sequentially with a 0.5 N HCl solution in order to eliminate excess KOH. Consecutively, activated carbon powders were repeatedly washed...
with distilled water until reaching a neutral pH. After that, these powders were dried in an oven at 110 °C for 24 h and stored in a desiccator before further use.

2.3. FTIR Spectroscopy

The chemical structure of the obtained activated carbon was verified by using a FTIR spectrometer (Invenio, Bruker, USA) in ATR mode equipped with a diamond crystal, which was used for the tests. All the spectra were recorded in transmittance mode with 4 cm⁻¹ resolution in the wavenumber range of 4000–400 cm⁻¹ under ambient conditions.

2.4. Iodine Number Analysis

The iodine number is a technique commonly employed to determine the adsorption capacity of activated carbons. In this paper, the iodine number was determined according to the standard test method ASTM D 4607–94 [45]. The procedure of the experiment can be briefly explained as follows: 1.0 g of dried activated carbon samples were placed in a 250 mL flask, and 10 mL of 5%wt. hydrochloric acid was added. The flask was swirled until the activated carbon became wet; then, the flask was boiled for 1 minute to degas the sulfur dioxide on the surface samples and cooled down to room temperature. Then, 100 mL of stock iodine solution (12.7 g of Iodine (Merck) and 19.1 g of potassium iodide (Merck) in 1 L of de-ionized water) was added into the sample flask, and the mixture was shaken for 1 minute in the magnetic stirrer (Model C-MAG HS7, IKA, Germany) and the samples were filtered. Then, 50 mL of filtrate was titrated with 0.1 N sodium thiosulfate until the solution become pale yellow. Then, approximately 1 mL of starch solution (1 wt.%), used as an indicator, was added, and titration slowly continued with sodium thiosulfate until the solution changed from blue color to colorless. The iodine number is the amount of adsorbed iodine per gram of samples (X/M), the value of which was calculated by Equations (1) and (2):

\[
\begin{align*}
\frac{X}{M} & = \left( \frac{(N_1 \times 126.93 \times V_1) - [(V_1 + V_{\text{HCl}})/V_F] \times (N_{\text{Na}_2\text{S}_2\text{O}_3} \times 126.93) \times V_{\text{Na}_2\text{S}_2\text{O}_3}}{M} \right) \\
C & = N_{\text{Na}_2\text{S}_2\text{O}_3} \times V_{\text{Na}_2\text{S}_2\text{O}_3}
\end{align*}
\]

where \(N_1\) and \(N_{\text{Na}_2\text{S}_2\text{O}_3}\) are the concentration of iodine solution and sodium thiosulfate solution (N), respectively. \(V_1\), \(V_{\text{HCl}}\), \(V_F\), and \(V_{\text{Na}_2\text{S}_2\text{O}_3}\) refer to the volume of iodine solution, hydrochloric acid, filtrate used in titration, and the consumed volume of sodium thiosulfate solution (mL), respectively. \(M\) is the mass of activated carbon (g).

2.5. Morphological Measurement

The morphological surfaces of the obtained activated carbon were examined via SEM (JSM-6610LV, JEOL, Japan) at an accelerating voltage of 10 kV. Each sample was attached to the stubs with double-sided carbon tape. Then, the samples were sputtered with gold under vacuum before observation. The average pore sizes for each activated carbon were investigated by using the image analysis software Image J (ver. 1.45, USA).

2.6. Specific Surface Area

The specific surface area, total pore volume \(V_T\), micropore volume \(V_a\), mesopore volume \(V_m\), and average pore size \(D_p\) of the obtained activated carbon were scrutinized via nitrogen adsorption Brunauer–Emmett–Teller (BET) analysis on a Micrometrics 3Flex Version 5.02 apparatus. The cell containing the sample was weighed before degassing. Approximately 0.2 g of sample powders were placed in a test tube in order to removes contaminants such as water vapor and adsorbed gases from the samples. The samples were degassed at 200 °C for 16 h before the measurements.
3. Results and Discussion

3.1. FTIR Spectroscopy

FTIR spectra were collected for the characterization of the surface functional groups of prepared activated carbon. Figure 1 depicts the FTIR spectrum of all activated carbons obtained after the chemical treatment with KOH followed by the different conditions, as shown in Table 1. The IR spectrum of the char sample was used to compare the spectra of the obtained activated carbons. We found that the weaker broad band around 3380 cm\(^{-1}\) revealed the stretching vibrations of the O-H bonds of the mixtures that were adsorbed onto the surface of activated carbon. A peak at around 2100 cm\(^{-1}\) shows the presence of a triple bond between carbon–carbon atoms (alkynes group) with stretching vibration [46]. Compared to the char spectrum, it was found that some changes could be noted in the position and intensity of some peaks in the activated carbon spectrum. A new strong peak appeared around 1030–1100 cm\(^{-1}\), which was because of the C-O stretching vibrations of carboxyl or carbonyl groups. This probably indicates the formation of an oxidation reaction during treatment with KOH in the activation process. Furthermore, we also found that the absorption peak in the regions between 1500 and 1600 cm\(^{-1}\) could be ascribed to C = C aromatic ring stretching vibration, which is generally found in carbonaceous material [47].

![FT-IR analysis of the obtained char and activated carbons.](image)

**Figure 1.** FT-IR analysis of the obtained char and activated carbons.

3.2. Iodine Number Analysis

It is well known that the indirect value of the iodine number also indicates the porosity of activated carbon, which is correlated with the micropore volume. A higher iodine number meant a higher porosity on the surface determined by iodine absorption. Therefore, iodine number analysis was used to analyze the porosity of DH1–DH5, and char was also used for comparison. The iodine adsorption capacity of all the activated carbon is illustrated in Figure 2. It is confirmed that the process parameters affected the porosity of the obtained activated carbon, which was found to be higher than char. Besides this, we found that the impregnation ratio between KOH and char has a profound effect on the adsorption properties of activated carbon. At the given activation temperatures, the iodine numbers of the activated carbons increased with an impregnation ratio increased from 1:4 to 1:2 and decreased with the KOH:char ratio increased to 1:1. From our experimental results, the highest iodine number was achieved at the impregnated ratio of 1:2, as shown in Figure 2. The iodine numbers of char, DH1, DH2, and DH3 are 717, 959, 971, and 933 mg/g, respectively. In addition, the effect of the activation process on the adsorption capacity was studied. It was found that the iodine numbers increased with increases in the temperature of the second activation process, which can be compared with DH2 and DH5 in Figure 2. At the given impregnation ratio, DH5 showed the highest values, at 1037 mg/g, of its iodine number compared with the other activated carbon. This is probably due to the effect of pore structure formation within the char structure during the activation process.
As mentioned above, the iodine number is commonly used in industries as a measure of adsorption capacity, reflecting the number of micropores in the carbon. This value indicates the approximate internal surface area of activated carbon. The relationship between the iodine number and the internal surface area of activated carbon has been well-established in the literature for various precursors [48–50]. It is well known that the iodine number has a direct proportional relationship with the specific surface area. Comparing recent studies, the iodine number of activated carbon from agricultural wastes has been reported to be up to 1000 mg/g, which is in accordance with the previous studies by Saka [51], Toledo et al. [52], and Anderson et al. [53]. Thus, the results of this study can suggest that the biochar from durian husk can be used as a candidate as a precursor in the manufacture of activated carbon with a highly porous structure for specialized industrial sorbents.

![Figure 2](image_url)

**Figure 2.** Effect of various preparation conditions on iodine number of activated carbon.

### 3.3. Morphological Measurement

An SEM instrument was used to observe the surface morphology of the materials obtained. Based on the iodine numbers, DH2 and DH5 were selected as desired activated carbons due to their high exhibited iodine number values. Therefore, we focused on investigating their surface morphologies, and the char was also compared in this study. Figure 3 shows the SEM micrographs of char, DH2, and DH5, respectively. From the image, it is clear that the durian husk can produce char and activated carbon composed of porous structures, with the appearance of roughly similar structures from all samples. However, the images suggest that the average pore diameters of the samples are quite different. As mentioned above, the pore sizes of activated carbon would be directly affected in terms of several of their properties, such as adsorption and mechanical properties. Therefore, it is worthwhile to further examine the pore sizes of the obtained activated carbon. Figure 4 shows the SEM images and the surface porous diameter distributions for the char and activated carbon materials. DH5 shows that the surface porosity is smaller than DH2 and char, respectively. In Figure 4, it can be seen that the average surface porous diameters of char, DH2, and DH5 are 6150 ± 2.86, 5060 ± 2.01, and 3790 ± 2.37 nm, respectively. The averages diameter of the pore structure decreased with an increasing activation temperature. This investigation qualitatively confirms in the same trends the results of the iodine number discussed earlier. Thus, we suggest that the surface structures of obtained activated carbon depend on the temperature and method of the activation process.
An SEM instrument was used to observe the surface morphology of the materials. Figure 3 shows the SEM micrographs of char, DH2, and DH5, respectively. From the image, it is clear that the durian husk can produce char and activated carbon composed of porous structures, with the appearance of roughly similar structures from all samples. However, it is notable that the surface morphologies of the materials vary, indicating differences in their porosities and structural characteristics.

Figure 4 illustrates the nitrogen adsorption/desorption isotherms of the char, DH2, and DH5. The adsorbed volume of DH5 is higher than DH2 and char, respectively. However, it was found that the isotherm curves for the char sample showed that N_{2} adsorption/desorption exhibited a pattern of a type II isotherm, according to IUPAC classification of isotherms [54]. This type of isotherm is a general characteristic of porous carbons that have large-sized pores. On the other hand, the N_{2} adsorption/desorption isotherms of DH2 and DH5 were exhibited as types I and II, indicating that there were many micropores in the materials. The rapid rise of adsorption/desorption isotherms in the low relative pressure region also indicated that there were microporous or small mesoporous structures. The textural characteristics of char and activated carbon prepared under different activation conditions...
are listed in Table 2. The $S_{\text{BET}}$ and $S_{\text{Langmuir}}$ increased significantly compared to char. Additionally, it can be observed from the data that the BET and Langmuir surface area increased with increases in the temperature of the second activation process when comparing DH2 and DH5. On the one hand, the pore size of DH5 was found to be the smallest of the three materials, which corresponded with similar trends in the results for the surface area. This is probably due to the increase in the activation temperature indicating the increase in extent of the reaction between KOH and char. Thus, the increased extent of the reaction aids in the enhancement and generation of new pores in the char, resulting in a higher surface area and pore volume, as presented in Table 3. It is well-known that the activation process can contribute to the carbon structure, allowing the process to produce a more ordered structural skeleton. Typically, the pore creation process would occur in four stages: (i) the opening of previously accessible pores, (ii) creation of new pores, (iii) widening of the existing pores, and (iv) combination of the existing pores due to pore wall breakage [55].

Figure 5. Nitrogen adsorption–desorption isotherms for char, DH2, and DH5, respectively.

| Samples | $S_{\text{BET}}$ (m$^2$/g) | $S_{\text{Langmuir}}$ (m$^2$/g) | $V_{T}$ (cm$^3$/g) | $V_{m}$ (cm$^3$/g) | $V_{p}$ (cm$^3$/g) | $D_p$ (nm) |
|---------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------|
| Char    | 68.10           | 13.97           | 0.07            | 0.07            | 0.07            | 4.63      |
| DH2     | 359.98          | 398.30          | 0.26            | 0.04            | 0.18            | 4.10      |
| DH5     | 608.03          | 666.22          | 0.34            | 0.12            | 0.08            | 3.96      |

4. Conclusions

We successfully produced activated carbons with a high porosity and high surface area to volume structures from durian husk waste via a chemical activation method. The condition of the activation process directly affected the physical properties of the obtained activated carbons. The porosity and surface areas of activated carbons increased with the increases in temperatures during the second activation process. The average iodine number was exhibited to be up to 1320 mg/g. FTIR spectra also indicated only characteristic peaks of activated carbons without any new sign of functional groups. SEM images also revealed that all the activated carbons with various pore sizes and porosities were related to the temperatures in the second activation process. The specific surface area, pore volume, and pore size were further confirmed by BET investigations. It was found that the DH5 exhibited the highest surface area and pore volume of the three samples, which agreed with the results of iodine numbers and SEM investigations. The adsorption/desorption isotherms of activated carbons produced within the range of temperatures and impregnation ratios were a mixture of type I and II isotherms. In summary, we demonstrated the advantages of the utilization of such an inexpensive material through a simple preparation method. This work opens up a new approach to producing activated carbons for applications in the environment and industries, such as for adsorbents and battery electrodes.
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