

Synthesis and Characterization of Activated Carbon from Giant Sour Tamarind Fruit Shell by KOH Activation

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ARTICLE INFO	ABSTRACT
<p>Article history: Received 12 February 2024 Received in revised form 27 March 2024 Accepted 25 May 2024 Available online 30 June 2024</p> <p>Keywords: Activated carbon; giant sour tamarind; agriculture waste; chemical activation; potassium hydroxide</p>	<p>Activated carbon, recognized for its high porosity and adsorption capabilities, finds widespread applications in water purification, air filtration, and energy storage. This study investigated the synthesis of activated carbon from giant sour tamarind fruit shell, an agricultural waste by-product, employing a two-step chemical activation process with potassium hydroxide (KOH) at varying activation temperatures (600, 700, and 800 °C). The BET surface area, pore volume, adsorption average pore diameter, pore size distribution, and adsorption isotherm were examined to characterize the properties of the giant sour tamarind fruit shell activated carbon. Results indicate that the activated carbon obtained at 800 °C exhibited the highest BET surface area (572.61 m²/g) and total pore volume (0.2563 cm³/g), coupled with the smallest adsorption average pore diameter (1.79 nm). The adsorption isotherm displayed characteristics of Types I/IV, suggesting a micro-mesoporous carbon structure.</p>

1. Introduction

Activated carbon is a carbonaceous material with high porosity that has been processed to increase surface area and adsorption capacity [1-3]. With a large surface area and pore structure, activated carbon may adsorb a wide range of substances, including organic compounds, pollutants, and contaminants [4,5]. Activated carbon is commonly used in various applications, including water and air purification, industrial processes, medical treatments, energy storage devices, and even everyday products like face masks [6-9].

Principally, activated carbon can be produced by a two-step process known as carbonization and activation [10]. Carbonization or pyrolysis is the thermal degradation of carbonaceous material in an inert atmosphere or a vacuum to increase the content of the element carbon and remove volatile impurities [11,12]. The activation step can be activated by physical activation, chemical activation, or a combination of both activations. In the case of physical activation, the carbon materials are heated at the same or higher temperature of the carbonization process in an oxidizing gas such as steam or

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carbon dioxide [13]. However, this method has a long activation time, and the obtained activated carbon possesses low adsorption capacity [14]. In chemical activation processes, the precursor is treated with a chemical agent such as potassium hydroxide (KOH), phosphoric acid (H₃PO₄), zinc chloride (ZnCl₂), potassium carbonate (K₂CO₃), or sodium hydroxide (NaOH) at a temperatures range of 400-800 °C [15]. The two-step process has the advantage of providing a higher surface area and porosity, the first carbonization stage provides an initial porosity that is further improved with the activation process [16]. Among the chemical agents, potassium hydroxide (KOH) is commonly used as an activator to produce activated carbon due to its effective reaction with the carbonaceous material [17] and is considered environmentally friendly compared to certain other chemical activating agents [18,19]. The activating agent has been used on a variety of precursors, such as rubberwood [20], bamboo [21], coconut shell [22], palm kernel shell [23], rice straw [24], corn cob [25], and spent coffee grounds [26].

In recent years, agricultural waste has been widely used as raw materials for activated carbon production, aiming to mitigate waste generation, promote sustainable practices, and develop cost-effective adsorbents. Tamarind (*Tamarindus indica* L.) is an evergreen tree that belongs to the family of Leguminosae, grown in tropical regions and subtropical regions. The various components of the tree are used in a wide range of industries, including food, pharmaceuticals, textiles, cosmetics, and environmental science [27]. In Thailand, there are two main types of tamarind (sour tamarind and sweet tamarind) [28]. Giant sour tamarind fruit, a specific variety of tamarind that has been developed for larger fruit size and pronounced sour taste, has gained significant attention from Thai farmers. As a result, agricultural residues, derived from giant sour tamarind fruit shells, are abundant. However, the studies on the characteristics and products derived from giant sour tamarind fruit are still limited. Previous studies have reported that the general tamarind fruit shell has the potential to be used as a raw material for effective activated carbon that is used for adsorption in wastewater treatment and energy storage applications [29-32].

In this present study, the giant sour tamarind fruit shell was used as a new material to synthesize cost-effectiveness activated carbon by chemical activation with potassium hydroxide (KOH) at activation temperatures of 600, 700, and 800°C. The BET surface area, pore volume, adsorption average pore diameter, pore size distribution, and adsorption isotherm of activated carbon were investigated.

2. Methodology

2.1 Materials and Chemicals

The raw giant sour tamarind fruit shells were acquired as a precursor from a farmer in Dan Sai, Loei, Thailand. The raw giant sour tamarind fruits were boiled in water before removing the shell from the raw pulp by using a knife. Potassium hydroxide pellets (KOH) and hydrochloric acid (37%, HCl) used in this study were from KemAus and Qrec (AR grade).

2.2 Synthesis of Activated Carbon

The activated carbon was synthesized by a two-step process as shown in Figure 1. The first step is the carbonization process. The giant sour tamarind fruit shell was crushed to a particle size of 0.5-2 mm after it was cleaned and dried in an oven at 110 °C for 24 hours to eliminate impurities and moisture content. A dried precursor was placed in a 4-inch diameter tube furnace to produce char and heated up to 400 °C at a heating rate of 6 °C/min under a nitrogen gas flow of 500 mL/min for 1 hour. The second step is the chemical activation process, the derived giant sour tamarind fruit shell

char (TSC) was cooled down to room temperature and activated by using potassium hydroxide (KOH) in the impregnation ratio (w/w) of 1:3 (TSC: KOH). The 50 g of a giant sour tamarind fruit shell char (TSC) was mixed with 150 g of KOH in 300 mL of DI water by using a magnetic stirrer for 24 hours. Afterward, the impregnated sample was activated at the activation temperatures of 600, 700, and 800 °C at a heating rate of 6 °C/min under a nitrogen gas flow of 500 mL/min for 1 hour. Materials obtained after activation at 600, 700, and 800°C are named as K6-TSAC, K7-TSAC, and K8-TSAC, respectively. After activation, the derived activated carbon samples were cooled down to room temperature and soaked with 0.1M HCl for 2 hours and then washed with DI water until the pH reached neutral. Finally, the giant sour tamarind fruit shell activated carbon (TSAC) was dried in an oven at 110 °C for 24 hours then collected into a sample bag and stored in the dry cabinet for further analysis.

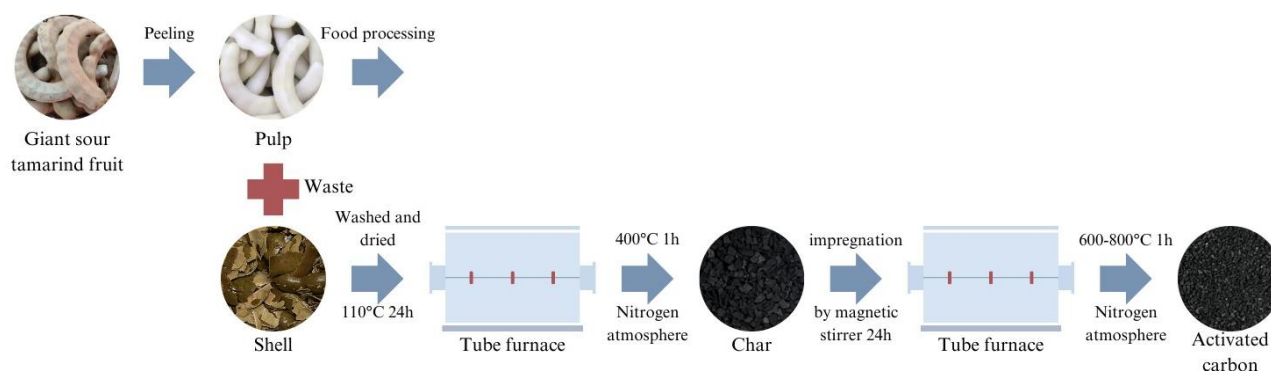


Fig. 1. The schematic of the synthesis of activated carbon

2.3 Characterization of Activated Carbon

The 4-digit analytical balance was used to weigh all the products in the preparation process. BET surface area, pore volume, adsorption average pore diameter, pore size distribution, and adsorption-desorption isotherm were determined by using static volumetric N₂ gas adsorption method with surface area and porosity analyzer (BET), ASAP2060, Micromeritics, USA from Office of Scientific Instrument and Testing, Prince of Songkla University, Thailand.

3. Results

3.1 The Yield of Activated Carbon

The yield of activated carbon was calculated by using Eq. (1), which involved the weight of the final activated carbon product and the weight of the raw materials on a dry basis. The yields of activated carbon from giant sour tamarind fruit shell are in a range of 23.01-28.31% as shown in Figure 2, the lowest yield was 23.01%, which was activated at the activation temperature of 800 °C. In contrast, the activated carbon with an activation temperature of 600 °C has the highest yield of 28.31%. Based on the result, it seems that when the activation temperature is increased, a greater quantity of small molecules is released [33].

$$Yield (\%) = \frac{Weight\ of\ activated\ carbon}{Weight\ of\ raw\ material} \times 100\% \quad (1)$$

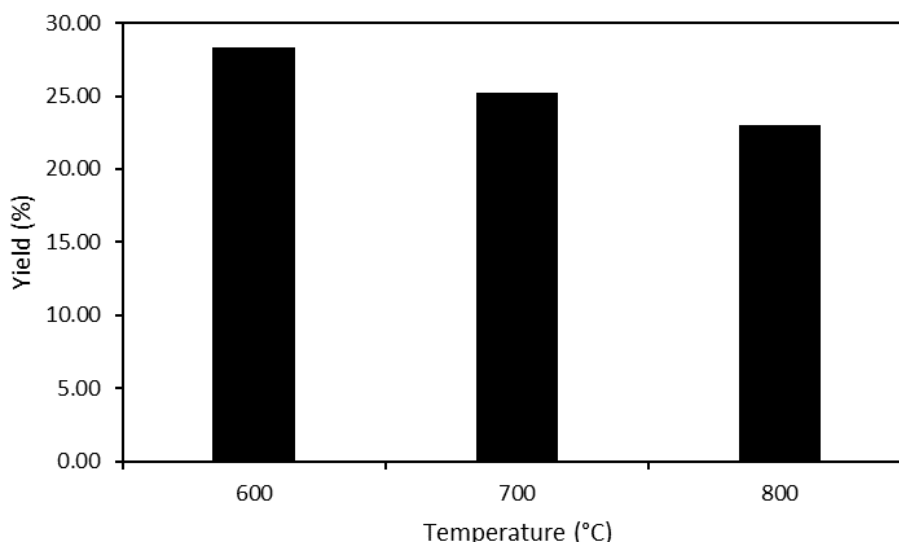


Fig. 2. The yield of activated carbon from giant sour tamarind fruit shell

3.2 Textural Characteristics

The textural properties of activated carbon derived from the giant sour tamarind fruit shell (TSAC) were assessed using the Brunauer, Emmett, and Teller (BET) method, which measures the quantity of physically adsorbed N_2 gas. The determination of total pore volume was based on the adsorbed amount, providing an indication of the overall porous spaces within the activated carbon, irrespective of pore width. Table 1 summarizes the textural characteristics of activated carbons derived from the giant sour tamarind fruit shell (TSAC) at different activation temperatures. BET surface areas of K6-TSAC, K7-TSAC, and K8-TSAC are 4.43, 375.27, and 572.61 m^2/g , respectively. Furthermore, the total pore volumes of K6-TSAC, K7-TSAC, and K8-TSAC are 0.0085, 0.1597, and 0.2563 cm^3/g , respectively. Among the samples, activated carbon produced at 800 °C exhibited the highest BET surface area and total pore volume, which included a micropore volume of 0.0967 cm^3/g , constituting 37.71% of the total pore volume. Both BET surface area and total pore volume increased with the elevation of the activation temperature. Adsorption average pore diameters of K6-TSAC, K7-TSAC, and K8-TSAC are 7.71, 1.79, and 1.79 nm, respectively.

Table 1
 Textural characteristics of activated carbon

Sample	T (°C)	S_{BET} (m^2/g) ^a	V_T (cm^3/g) ^b	V_{mic} (cm^3/g) ^c	D_p (nm) ^d
K6-TSAC	600	4.43	0.0085	-	7.71
K7-TSAC	700	375.27	0.1597	0.0443	1.79
K8-TSAC	800	572.61	0.2563	0.0967	1.79

^a BET surface area

^b Total pore volume

^c Micropore volume

^d Adsorption average pore diameter

3.3 Pore Size Distribution

Figure 3 depicts the pore size distributions of activated carbon within a range of 0-10 nm, indicating two pore size regions: micropores (pore width: <2 nm) and mesopores (pore width: 2-50 nm). The pore size distribution of K7-TSAC and K8-TSAC show similar results as shown in Figure 3(b)

and Figure 3(c). Zero pore volume is observed with pore widths smaller than 1.0901 nm for K7-TSAC and 1.795 nm for K8-TSAC. Both graphs have the same noticeable peak of pore volume at 1.2688 nm, indicating the presence of a significant number of micropores. Figure 3(a), K6-TSAC exhibits zero pore volume for pore widths smaller than 1 nm and displays a peak of pore volume at 1.0901 nm. However, this peak remains significantly lower compared to that of K7-TSAC and K8-TSAC. Additionally, there is a notable increase in pore volume for widths above 7.3986 nm, attributed to the mesoporous structure, corresponding to the adsorption average pore diameter in Table 1.

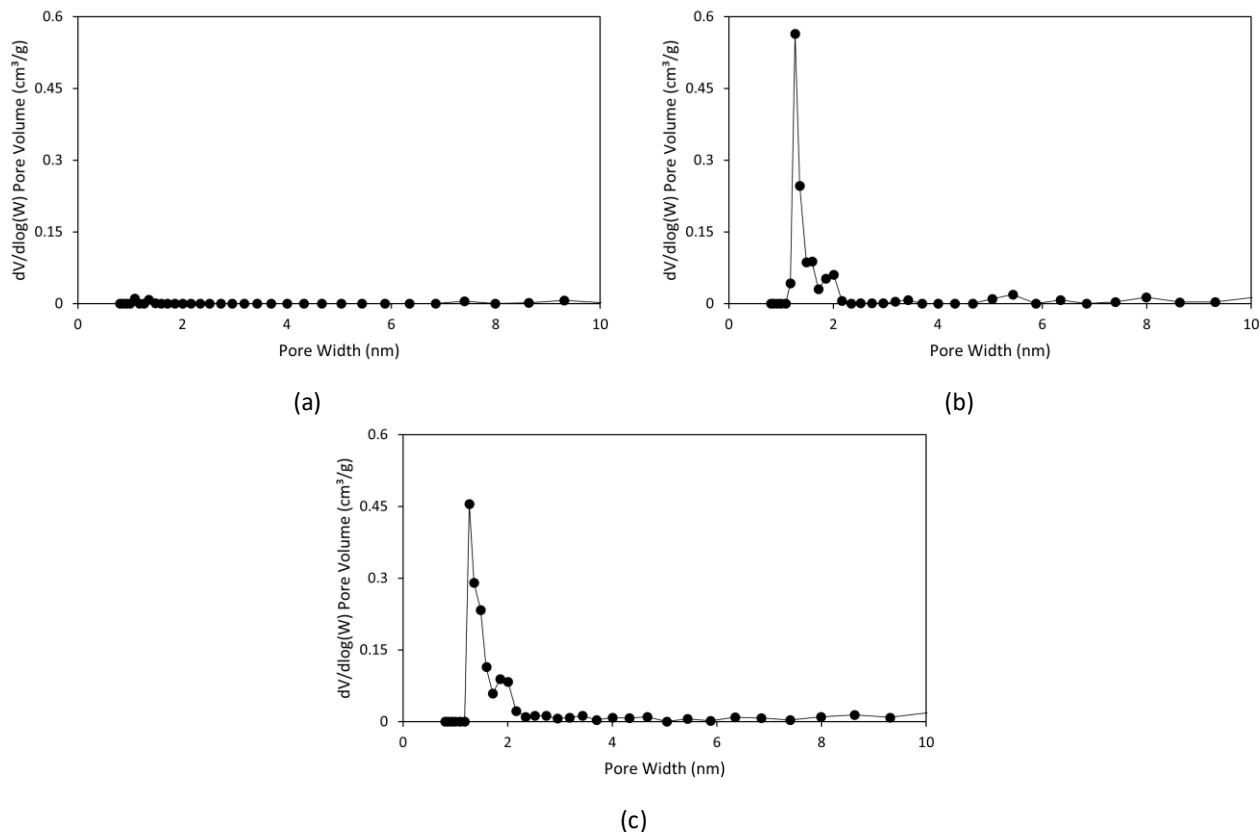


Fig. 3. Pore size distribution of (a) K6-TSAC, (b) K7-TSAC, and (c) K8-TSAC

3.4 Adsorption Isotherm

The adsorption-desorption isotherms conducted with N_2 at 77 K on activated carbon at different activation temperatures are illustrated in Figure 4. According to The International Union of Pure and Applied Chemistry (IUPAC) isotherms classification, the isotherm curves of K7-TSAC and K8-TSAC exhibit similar shapes and might be classified as a combination of Types I and IV isotherms [34,35]. The adsorption branch displays characteristics akin to a Type I isotherm, with a sharp uptake at low p/p° , indicating the filling of micropores. The appearance of a hysteresis loop within the medium range, above p/p° of 0.4, categorizes it as an H4-type according to IUPAC, commonly observed in micro-mesoporous carbons. Additionally, a slightly limitless increase is observed at p/p° near 1, confirming the presence of mesopores. However, the isotherm curves of K6-TSAC might be classified as Type II isotherms, associated with the physisorption of most gases on nonporous or microporous adsorbents.

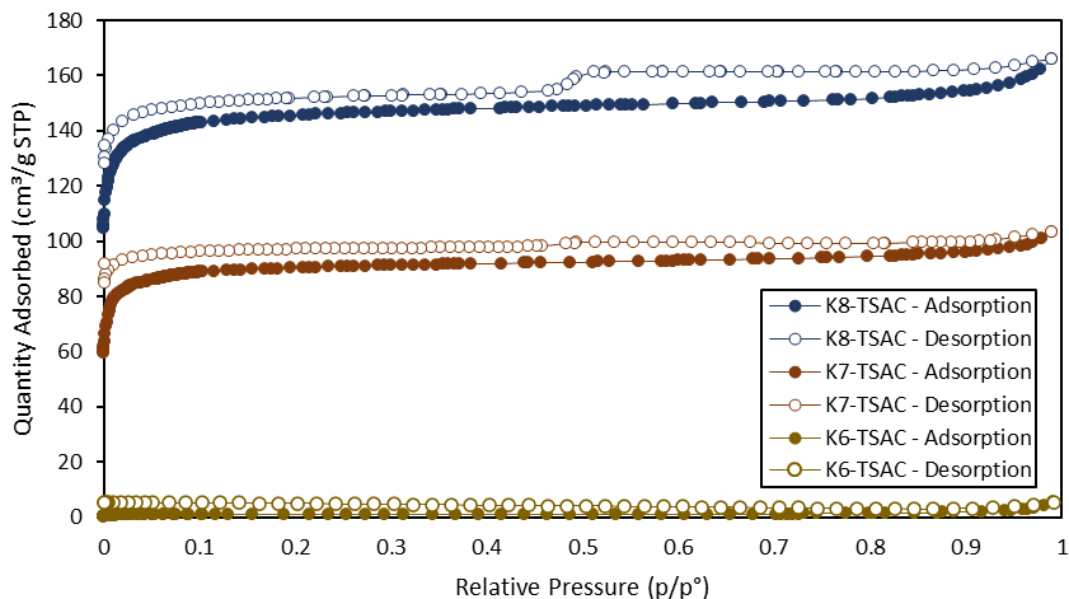


Fig. 4. Adsorption-desorption isotherm of activated carbons

4. Conclusions

In this study, activated carbon was synthesized from the giant sour tamarind fruit shell by chemical activation with KOH at various activation temperatures. The activated carbon obtained at 800 °C exhibited the highest BET surface area, total pore volume, and micropore volume of 572.61 m²/g, 0.2563 cm³/g, and 0.0967 cm³/g, respectively. These values showed a significant increase with the rise in activation temperature and tended to further increase at temperatures over 800 °C. The adsorption average pore diameter, pore size distribution, and adsorption isotherm collectively indicate that the activated carbon obtained at 600 °C tends to exhibit a nonporous or mesoporous structure, while the activated carbons obtained at higher temperatures display a micro-mesoporous structure. The well-developed porous activated carbon derived from giant sour tamarind fruit shell is capable of being potentially used as an adsorbent in various applications.

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