

A Simple and Rapid Preparation of Activated Carbons with Highly Surface Area from Durian Shell Ash

Tran Thi Bich Quyen^{1,*}, Nguyen Van Toan¹, Tran Chi Nhan¹, Doan Van Hong Thien¹,
Luong Huynh Vu Thanh¹

¹Department of Chemical Engineering, College of Engineering Technology, Can Tho University, 3/2 Street, Ninh Kieu District, Can Tho City, Vietnam

*Corresponding Author: Tran Thi Bich Quyen; E-mail: tbquyen@ctu.edu.vn

Abstract— In this study, activated carbons with high surface area prepared by a simple and rapid method from durian shell waste by a one-step carbonization-activation process. The activation process was performed by various activators (i.e. using acid, ethanol, water) and under supporting of microwave irradiation at 150 W for 1 hour. The morphology, structure, and composition of activated carbons were determined by scanning electronic microscopy (SEM), X-ray diffraction (XRD), and energy-dispersive X-ray (EDX) techniques. Moreover, the surface area of this prepared activated carbon was also observed using the Brunauer–Emmett–Teller (BET) analysis method. The results showed that activated carbons provided a high surface area about 683.24 m²/g more ~24.2 times that of durian shell ash with surface area ~26.18 m²/g. The results of this study present a relatively simple process for producing activated carbon made from durian shell waste.

Keywords— Durian shell (DS); durian shell ash (DSA); activated carbon (AC); surface area; microwave.

I. INTRODUCTION

Durian fruit production in 2017 in Vietnam reached 402.000 tons. Seventy percent of the durian fruit is its shell, which becomes waste after the meat is taken [1]. This waste is seen in many urban areas in Vietnam, especially during the fruit season. The utilization of durian shell waste offers a solution to urban waste distribution. One way of utilizing durian shells is to convert them into activated carbon to increase their economic value [2]. The production of activated carbon from biomass materials usually includes carbonization and activation processes [3].

The pollution control and environmental safety have got global attention and has opened new window for development in the adsorption process technology. In many industrial processes, for example, textile, washing, printing release pollutant into stream. Thus effluent treatment plant, a mandatory measure to recover them or reduce their concentrations in effluent streams, is employed due to environmental concerns, where adsorption method is used as it is one of the most effective and versatile methods. Activated carbons, are frequently used as adsorbent, remove a numerous species from waste stream as it has an excellent adsorption capacity, which is related to a large specific surface area and porousness. Several investigations have been found using activated carbon for removal of various species [4–13]. Despite its prolific use, activated carbons in water and wastewater industries, commercial activated carbons remain an expensive material. This has led to a search for low-cost, easily available materials as alternative adsorbent materials. Proper utilization of agro industrial by-product is very much important for economic development. A wide variety of activated carbons have been prepared from agricultural waste such as coconut shells, cotton stalk,

sugarcane bagasse, coir pith, straw [14, 15] and also from non-renewable materials like coal, wood, and peat [16–18].

Activated carbons adsorb a wide variety of substances including different types of dyes and heavy metals [19]. It is the porous structure and chemical nature of the surface of carbons that govern this property, both of which are related to the crystalline composition. Although the microcrystalline structure is dependent on the raw material, impurities and conditions of production, the basic structural unit is similar to that of graphite. Pore size is an important parameter for quality of activated carbon. Based on the effective radii and mechanism of gas adsorption, pores are classified to be macro-pores (>50 nm), meso-pores (2 to 50 nm) and micro-pores (<2 nm) [8, 20]. The main role of macro-pores in adsorption is to act as transport arteries that allow the meso- and micro-pores to become accessible to particles of the adsorbate. Meso-pores contribute significantly to the adsorption process and also provide the main transport arteries for the adsorbate. Adsorption occurs in micro-pores through the mechanism of volume filling. The energy of adsorption in micro-pores is much higher, resulting in a large increase in adsorption capacity for small equilibrium pressures of adsorbate. The molecular sizes of different pollutant species are different. Therefore, activated carbon having smaller pores are not able to adsorb pollutants having larger diameter. Hence, there is knowledge gap of developing different pore sized activated carbon warrants conducting study on variation of pore development in activated by various methods.

In this work, we investigated the effect of activation agents on variation in pore size of activated carbon; and determined them by BET method (surface area, pore size), and SEM image.

II. EXPERIMENTAL SECTION

2.1 Materials

Durian shells were collected from a garden at Binh Minh of Vinh Long Province in Vietnam. Sulfuric acid (H_2SO_4), nitric acid (HNO_3) and ethanol (C_2H_5OH) were purchased from Acros. All solutions were prepared with deionized water from a MilliQ system.

2.2 Methods

2.2.1 Preparation of durian shells ash

Fresh durian shells were washed, sliced and dried for 24 h at $80^\circ C$. Then the sample was burned inside a muffle furnace to give a required heat treatment under N_2 gas flow. After ashing at temperature of $700^\circ C$ for 1 h, the durian shells ash sample was obtained. Then the burned samples are preserved in desiccators to avoid further absorption of moisture. Similar procedure was followed to prepare activated carbon with various activations by acid, ethanol and water.

2.2.2 Preparation of activated carbons

0.5 g of durian shell ash was stirred in 30 mL of various activators (i.e. acid solution of H_2SO_4 (10 M)/ HNO_3 (10 M) solution with ratio = 1:1; or ethanol; or deion (DI) water, respectively) for 30 min at room temperature. After activation, the samples were washed with distilled water until the pH value reached 7.0. Next, the samples were dissolved in deion water (DI water) and put into the microwave for heat treatment at 150 W for 1 h. After that,

the samples are filtered and dried in an oven at $80^\circ C$ for 24 h. Final, the samples are calcinated at $350^\circ C$ for 2 h to obtain activated carbons for the investigation of next steps.

2.2.3 Characterization techniques

The phase structure of the activated carbons was examined using an X-ray diffractometer (Rigaku Dmax-B, Japan) with $Cu K_\alpha$ source operated at 40 kV and 100 mA. A scan rate of 0.02 deg^{-1} was used for 2θ between 10° and 90° . The surface morphology of the activated carbon was characterized by scanning electron microscopy (SEM) and Energy-dispersive X-ray spectroscopy (EDX or EDS) by HR-SEM Hitachi s-4700 equipment. The pore structure of activated carbons was characterized by physical adsorption of nitrogen, in an automatic Micromeritics ASAP-2010 volumetric sorption analyzer at 77.35 K over a relative pressure (p/p^0) range from approximately 10^{-5} –0.995. The specific surface area (S_{BET}), micropore volume (V_{mic}), and micropore surface area (S_{mic}) of the samples were determined by means of Brunauer–Emmett–Teller (BET).

III. RESULTS AND DISCUSSION

3.1 Characterization and Morphology of the Activated Carbons from Durian Shells Ash

As shown in Figure 1, the SEM images in Figures 1a, b, and c represent the activated carbon/ethanol (AC/EtOH), activated carbon/acid (AC/Ac), activated carbon/water (AC/Wt) samples, respectively.

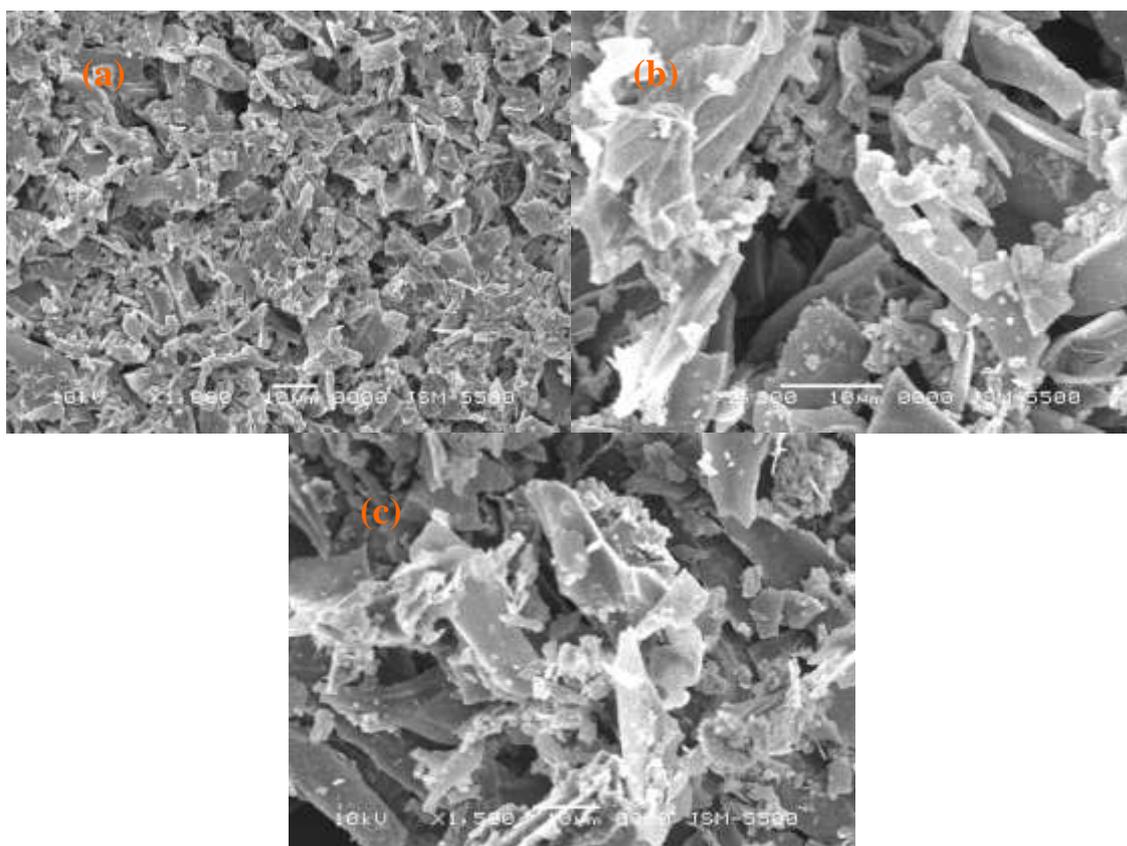


Fig. 1. SEM images of activated carbons from durian shells ash at room temperature for 30 min and at 150 W for 1 h in the microwave with various activations of (a) ethanol; (b) acid; and (c) water.

Figure 1a shows that the morphology of the activated carbons (AC) at the cross-sectional area was dominated by particles with a relatively small, finer size, and large spaces (macro pores) was observed between the particles. Figures 1b and 1c show that the SEM micrograph was dominated by larger particles. Based on the variations in the activation process, different sample morphologies were observed. Samples with ethanol activation had pore sizes between the particles ranging from 10.96 μm to 13.26 μm , while the pore sizes between the particles for the DI water and the acid activation samples ranged from 3.86 μm to 8.12 μm . The presence of these flat pores indicated that samples with ethanol activation had good porosity properties.

An energy dispersive X-ray (EDX) analysis was conducted to determine the compositions of the activated carbons. The elements of the durian shell ash-based activated carbon samples are shown in Figure 2. Based on the data in Figure 2, the material consisted of carbon (C), oxygen (O), magnesium (Mg), silicon (Si), phosphorus (P), and potassium (K). Carbon was the dominant element with a range from 79.4% to 80.3%. These results prove that the sample predominantly contained carbon as desired. The presence of potassium, with the largest amount after carbon, indicated that the composition of the durian shell was also mostly dominated by potassium.

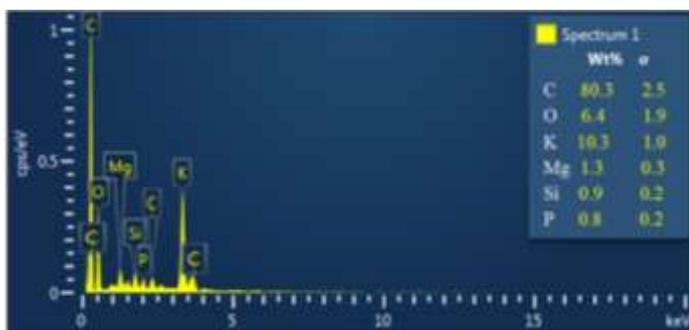


Fig. 2. EDX profile of activated carbon with activation of ethanol from durian shell ash and their quantitative analysis.

FTIR results of the durian shell ash and activated carbon samples as shown in Figure 3. Results also denote the increase of peak energy of the symmetrical and assymetrical bands at wavenumber ranges of 935 cm^{-1} , 1100–1200 cm^{-1} , 1400–1680 cm^{-1} , and 3200–3500 cm^{-1} for carbons modified with activation of ethanol. These bands may correspond to C–C stretch of carboxylic groups, C=O stretch of lactone group, and O–H stretch of phenolic groups, respectively [21].

The structure of the activated carbon from the durian shell ash was determined by X-ray diffraction (XRD). As shown in Figure 4, two broadening peaks at 2θ values of 24.17 and 43.56 in the X-ray diffraction curves show the presence of carbon materials within amorphous structures [22]. The 2θ diffraction angles in the reflection planes of 002 and 100 for the activated sample by ethanol were 26.79° and 43.69°; 26.55° and 43.51° for the activation sample by acid; and 26.35° and 43.39° for the original durian shells ash sample, respectively. These data show that the samples exhibited good peaks for carbon materials [22].

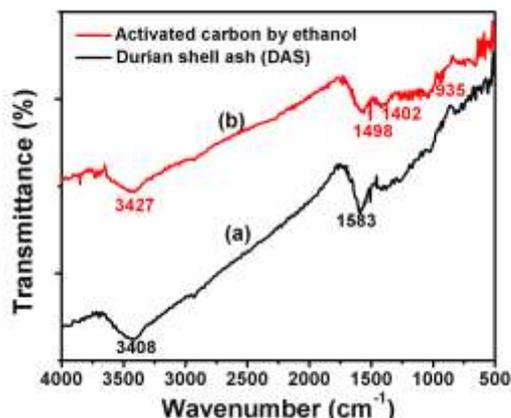


Fig. 3. FTIR spectra of activated carbon with activation of ethanol from durian shell ash and original durian shell ash.

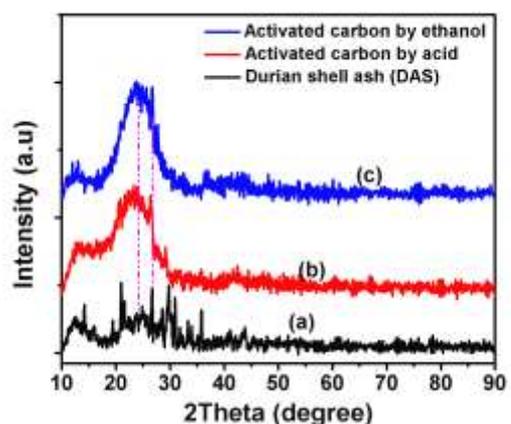


Fig. 4. XRD patterns of (a) original durian shell ash; modified carbon with different activators of (b) acid solution; and (c) ethanol, respectively.

IV. CONCLUSIONS

A rapid, single-step carbonization and activation method for producing activated carbons from durian shell waste was successfully demonstrated. This rapid, friendly-environmental, single-step method clearly reduces the time required for processing activated carbon materials and certainly reduces the production costs. The activated carbons from durian shell ash waste produced using the single-step method demonstrated good physical and chemical properties. The acid and ethanol activation resulted in slightly different physical and chemical properties, from which the ethanol activation produced activated carbon with better pore (i.e. surface area $\sim 638.24 \text{ m}^2/\text{g}$ using ethanol for activation, and $\sim 163.72 \text{ m}^2/\text{g}$ for using acid, respectively) and highly catalytic activity. Therefore, the activated carbons it could be very interesting in the field of catalysis and energy. In future work, the catalytic activity of this material and application in the Lithium ion batteries will be investigated.

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