Synthesis and Characterization of Activated Carbon from Water Hyacinth

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Abstract. Water hyacinth-based activated carbon (denoted as ACWH) with porous has the chemical and thermal activation of the material. The chemical and thermal activation were synthesized by chemical types used for activation process, activation time, temperature, carbonization time. Which the temperature for the thermal activation process was optimized to produce size activated carbon (AC). The crystal structure, microstructure and chemical composition of activated carbon was characterized by X-ray diffraction (XRD), field emission scanning electron microscope (FE-SEM) and Energy Dispersive X-Ray Spectroscopy (EDS), respectively. It was found that the performance of the activated carbon shows mixed phase of carbon (90%) and graphite (10%).

1. Introduction

Water Hyacinth (WH), one of the common aquatic. WH was utilized as a precursor to synthesize activated carbon. Activated Carbon (AC) is widely chosen with the consideration of its low-cost production. It is synthesized through the two-step process: carbonization of carbon sources and activation process [1]. AC has shown remarkable properties and has been widely applied in many research fields to produce a variety of products such as electronic devices, field emitters, hydrogen storages, composite materials, biosensors, fuel cells and capacitors. The process of carbon activation that can be regulated to produce large of specific surface area and specific pore size makes the activation process a topic of research for many experts [2]. Some carbon activation process parameters such as carbon sources, activator mass ratio, and activation temperatures contribute to the specific surface area [3]. AC can be synthesized from various carbon sources such as petroleum pitches and various kinds of organic waste (banana skin [4], watermelon [5], sorghum pith [6], and rice husk [7]). Availableness, production costs, and the potential of SSA are considered in the selection of carbon sources. WH is a water plant originating from the Nong Han Lake, Sakon Nakhon, Thailand. They are found in almost all freshwater ecosystems in the world. Its uncontrolled growth causes various ecosystem problems such as eutrophication and blockage of waterways. Although, behind its shortcomings, its abundant availability and low utilization make water hyacinth a suitable carbon source for batteries as its low-cost production. Consequently, AC synthesis from water hyacinth by
chemical and physical process, which is simple and convenient for mass production. This method is a cost-effective and user-friendly technique. With the progress of science and technology, it is found that AC seems to contain unlimited possibilities of development.

In this study, synthesis and characterization of activated carbon from Water Hyacinth was started with the carbonization and continued by the activation process using chemical activation with activator agent Hydrochloric acid (HCL). During the activation, the temperature was varied to obtain the optimal specific surface area. AC produced was characterized using XRD and FE-SEM.

2. Materials and methods

The water hyacinth stem (WHs) samples were taken from the Nong Han Lake, Sakon Nakhon, Thailand. The samples were washed the sludge off the surface in order to make the water hyacinth clean for about 7 days to ensure complete removal of surface water. Next, the sample was cut into small pieces 2 cm and dried in sunlight for a week and dried in an oven at 90 °C for 2 hours in Figure 1. The dried water hyacinth was then kept in a desiccator use for the preparation of activated carbon using physical and chemical methods [8]. Finally, the samples were stored in a desiccator until needed.

![Figure 1. Schematic illustration of the preparation process of dried Water Hyacinth](image)

The dried water hyacinth was treated with concentrated Hydrochloric acid (HCL) in the ratio 1:2 (w/w). The contents were kept in without air for about 24 hours. Then, the sample was washed distilled water to pH 7. The activation results were dried in an oven 90 °C for 2 hours. The dried material was then slow pyrolysis at 600 °C for 1 hour with a ramping temperature of 3°C/min under Argon (Ar) atmosphere at a flow rate of 0.3 L/min [9]. Finally, it was introduced into a milled and sieved into smaller sizes in the range of 3–5 mm. The proximate analysis and elemental analysis of water hyacinth are shown in Figure 3. Table 1 reveals the chemical element measurement mean score of Carbon Water Hyacinth (WHc) with SEM-EDS test. Those chemical substances including carbon (C), oxygen (O), potassium (K), chlorine (Cl), calcium (Ca), sodium (Na) and magnesium (Mg).

![Figure 2. Schematic illustration of the preparation process of Water Hyacinth Carbon](image)

| Element   | WHc Elemental Analysis (wt, %) | WHc Proximate Analysis (wt, %) |
|-----------|-------------------------------|-------------------------------|
| C         | 58.86                         | Ca                            |
| O         | 14.23                         | Pt                            |
| K         | 10.18                         | Na                            |
| Cl        | 8.64                          | Mg                            |
| Ca        | 4.60                          | 0.76                          |
| Pt        | 2.14                          | 0.59                          |
Figure 3. EDS elemental Mapping of the water hyacinth on dry basis (A) The corresponding elemental mapping of C, O, Na, Mg, Cl, K and Ca respectively. (B) SEM image and (C) EDS spectrum of WHc.

The powder XRD patterns of the WHc and the composite of WHc are shown in Figure 4. The XRD pattern of pristine composite shows diffraction peaks at 2θ degree of 24.48°, 28.34°, 40.50°, 50.16°, 58.64°, 66.38° and 73.73° can be indexed as (111), (200), (220), (222), (400), (420) and 422 planes of standard KCl (JCPDS Card No. 00-041-1476). Thus, WHc heat treatment 600 °C has cubic structure with lattice parameter a = 0.6287 nm. By without evident diffraction plane of carbon, when increasing heat treatment temperature over 900 °C, the planarity increases and the imperfection density within the layers decreases. The planarity of the layers begin to be established leading to the production of (hkl) diffraction lines.

Figure 4. Powder XRD of WHc at 600 °C for 1h in an argon flow 0.3 L/min.
The unique microstructure of WHc bodes well for the preparation of large porous sheet-like carbon materials and the preparation procedures that are schematically illustrated in Figure 5 are simple, economical, and environmentally friendly. Then the sample was smoothed with a grinder and sifted using a Shaker 75 No. 200 mesh sieve and then fast pyrolysis at five different holding temperatures, such as, 600 °C, 800 °C, 1000 °C, and 1200 °C for 1 hour with a ramping temperature of 3°C/min, under argon atmosphere at a pressure under 0.05 MPa. Argon (Ar) atmosphere at a flow rate of 0.3 L/min. The water hyacinth derived activated carbon (ACWH), then denoted as WHc600, AC600, AC800, AC1000, and AC1200, respectively, according to its activation temperature. The sample was treated with concentrated Potassium Hydroxide (KOH) in the ratio 1:2 (w/w) in air for about 2 hours. Then, the sample was washed deionized (DI) water to pH 7 to obtain Activated Carbon Water Hyacinth (ACWH).

The morphology consideration and quantitative of samples were observed using scanning electron microscopy (SEM-EDS: JSM-7610F Plus) with an accelerating voltage of 15 kV. X-ray Diffractometer (XRD) of ACWH samples was performed on an X-ray Diffractometer (XRD: SHIMADZU-6100, Cu k radiation voltage 40 kV current 30 mA).

3. Results and Discussion

The powder XRD patterns of the ACWH are shown in Figure 6. The XRD pattern of pristine ACWH shows two weak diffraction peaks at 2θ degree of 23.40°, 44.02° and 77.42° which can be attributed to plan (002), (100) and (110) crystal planes of partially activated carbon graphitized with the amorphous character [10]. The XRD spectrum of WHc600, AC600, AC800, AC1000 and AC1200 also show a broad peak located around 2 theta 20° ~ 30°, which originated from the graphene structure [11] [12]. The broad (002) diffraction peak found on WHc600, AC600, AC800, AC1000 and AC1200 also indicates the presence of the amorphous structure of carbon.

To examine the morphology of activated carbon particles from different treatments, a field emission scanning electron microscope (FE-SEM) test was conducted. Figure 7 shows the morphology of ACWH samples with 1200 °C. The proximate analysis and elemental analysis of ACWH are shown in Figure 7. Table 2 reveals the chemical element measurement mean score of ACWH with FE-SEM test. Those chemical substances including carbon (C), oxygen (O) and potassium (K).
Figure 6. XRD pattern of sample (a) WHc600 and AC600 (b) AC800, AC1000 and AC1200 (c) AC1200
Table 2. Shows the elemental analysis and proximate analysis of ACWH.

| Elemental analysis (wt, %) |
|---------------------------|
| C                        | 94.64 |
| O                        | 1.77  |
| K                        | 0.55  |
| Total                    | 100   |

Figure 7. EDS elemental Mapping of the water hyacinth on dry basis (A) The corresponding elemental mapping of C, O and K respectively. (B) SEM image and (C) EDS.

4. Conclusion

We can synthesize the AC from water hyacinth using slow-fast pyrolysis and chemical activation with activator agent hydrochloric acid. The crystallography of AC shows the main phase of carbon and small phases of graphite. The microstructure of AC has size and shape of small, thin and aggregate closely related to each other and the reduced percentage of oxygen elements in carbon and graphite.

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