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Review Article

Crystalline Structure Analysis of Bamboo and Coconut Coir–Based Activated Carbon for Supercapacitor Electrode Applications

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Abstract: Activated carbon has been developed for supercapacitor **e**lectrode material due to their high degree of micro porosity and large surface area. The carbon source, preparation conditions such as temperature and atmosphere, and preparation method strongly influence the crystalline structure and the properties of carbon materials. This article is focused on the crystalline structure analysis of bamboo and coconut coir activated carbon The bamboo and coconut coir carbon were fabricated by using pyrolysis method. The activated bamboo carbon and activated coconut coir carbon were produced using a chemical activation method where H_3PO_4 solution as activator agent. Characterization of the physical/crystalline structure of the bamboo carbon (BC), and coconut coir carbon (CCC) and bamboo activated carbon (BAC), coconut coir activated carbon (CCAC) was determined using XRD measurement. The XRD spectra of BC and BAC indicate that the percentage crystallinity are 29.1%, and 18.4% respectively. For CCC and CCAC the percentage crystallinity are 11.3% and 13.2%, respectively. The interlayer spacing (d_{hkl}) for BC is 4.05 Angstrom, and for BAC is 3,79 Angstrom. The crystallite height (Lc) for BAC is 6.64 Angstrom and for BC is 21.56 Angstrom. The interlayer spacing (d_{hkl}) for CCC and CCAC are the same 4.05 Angstrom. The crystallite height (Lc) for CCAC is 4.96 and for CCC is 2.83 Angstrom.

Keywords: Activated Carbon; Crystalline Structure; Pyrolysis Method; Supercapacitor Electrode; XRD Measurement

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1. Introduction

Activated carbon has been extensively developed as an electrode material for supercapacitor applications due to its high specific surface area, well-developed microporosity, chemical stability, and relatively low production cost. These characteristics enable efficient charge storage through electric double-layer capacitance (EDLC), making activated carbon one of the most widely used materials in commercial supercapacitors (Rajagopal et al., 2022; Lakshmi & Vedhanarayanan, 2023; Forouzandeh et al., 2020).

The electrochemical performance of supercapacitors is strongly dependent on the intrinsic properties of electrode materials as well as the fabrication processes employed. Factors such as carbon precursor type, preparation temperature, activation atmosphere, and activation method play a crucial role in determining the structural, textural, and surface chemical properties of carbon materials (Rehman et al., 2019; Ho & Kabbashi, 2020; Shahcheragh et al., 2023). In particular, the degree of crystallinity and the balance between amorphous and ordered carbon phases significantly influence electrical conductivity and charge transport behavior in supercapacitor electrodes (Kelesidis et al., 2022).

Biomass-derived activated carbons have attracted increasing attention as sustainable alternatives to fossil-based carbon materials. Agricultural wastes such as bamboo and coconut coir are considered promising precursors due to their abundance, renewability, low cost, and

high carbon content (Chew et al., 2023; Negara et al., 2023). Bamboo activated carbon (BAC) and coconut coir activated carbon (CCAC) have been reported to exhibit high specific surface area, long life cycle stability, and favorable pore structures for energy storage applications (Efeovbokhan et al., 2019; Maulina & Mentari, 2019).

During the carbonization process, biomass undergoes thermal decomposition in which moisture and volatile compounds are removed, leaving behind a carbon-rich char. This char generally exhibits a semi-crystalline structure composed of both amorphous carbon and turbostratic microcrystalline domains resembling graphite-like layers (Imammuddin et al., 2018). Further activation through physical or chemical methods enhances pore development and modifies the crystallinity of the carbon structure, which in turn affects electrochemical performance (Shahcheragh et al., 2023; Ho & Kabbashi, 2020).

Chemical activation using phosphoric acid (H_3PO_4) is one of the most effective methods for producing activated carbon with high porosity and improved structural characteristics. H_3PO_4 promotes dehydration, cross-linking reactions, and the formation of a porous network while suppressing excessive carbon burn-off (Chew et al., 2023; Negara et al., 2023). Several studies have demonstrated that H_3PO_4-activated carbon derived from biomass exhibits enhanced surface properties and suitable crystallinity for electrode applications (Maulina & Mentari, 2019; Negara et al., 2023).

Crystallinity refers to the degree of structural order within a solid material and is a critical parameter in evaluating carbon-based electrode materials. The crystalline structure of activated carbon can be characterized using techniques such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and differential scanning calorimetry (DSC) (Efeovbokhan et al., 2019). Among these techniques, XRD is widely used to identify graphitic ordering, interlayer spacing, and crystallite size through methods such as the Scherrer equation (Fatimah et al., 2022).

Although numerous studies have investigated the surface area, pore structure, and electrochemical performance of bamboo- and coconut-based activated carbons, comparative studies focusing specifically on their crystalline structures remain limited. Understanding the crystallinity and structural ordering of BAC and CCAC is essential for optimizing their performance as supercapacitor electrode materials (Kelesidis et al., 2022; Rajagopal et al., 2022).

Therefore, this study focuses on the crystalline structure analysis of bamboo activated carbon (BAC) and coconut coir activated carbon (CCAC) prepared via pyrolysis followed by chemical activation using 20% H_3PO_4. The crystallinity of the resulting activated carbons was investigated using X-ray diffraction (XRD) to evaluate their structural characteristics and potential suitability for supercapacitor electrode applications..

2. Research Methods

Materials

Material used in this research were bamboo, coconut coir, phosphoric acid (H_3PO_4) 85% as activator, aquadest.

Fabrication of bamboo carbon (BC) and coconut coir carbon (CCC)

The bamboo carbon (BC) and coconut coir carbon (CCC) have been produced by pyrolysis method. The raw material bamboo and coconut coir used in this research are shown in Figure 1.

(a) (b)

Figure 1. (a). The Raw Materials Bamboo (*Bambu Andong, Gigantochola- verticillata*) from Manado. (b). The raw Materials Coconut Coir Carbon (*Cocos nucifer*) from Manado

The bamboo carbon (BC) and coconut coir (CCC) were fabricated using pyrolysis method. The raw material bamboo are cutting in small pieces (2x2 cm). The procedure of fabrication of bamboo carbon and coconut coir carbon is described as the following. The bamboo and coconut coir are cutting into small pieces (2x2 cm), and then put into pyrolysis reactor was made from stainless steel. The pyrolysis reactor is put on a Gas Stove (Rinnai Corp. Japan). The gas stove was on, and then the pyrolysis reactor was heated at temperature of about 400^{o}C for 90 minute. The set up experiment of the pyrolysis process is shown in figure 2.

Figure 2. The Pyrolysis Process for Fabrication Bamboo Carbon (BC) and Coconut Coir Carbon (CCC).

Fabrication of Bamboo Activated Carbon (BAC) and Coconut Coir Activated Carbon (CCAC)

Fabrication of bamboo activated carbon (BAC) and coconut coir activated carbon (CCAC) were carried out by chemical activation treatment using (H_3PO_4) 20% as activator. The chemical activation process using phosphoric acid solution of (H_3PO_4) 20% as activator. The purpose of this activation process in order to release the hydrocarbon, tar and other organic compounds that are still attached to carbonized charcoal. The activation procedure as the following:

a. Preparation of a phosphoric acid solution of (H_3PO_4) 20% by mixing 600 ml aquadest (distilled water) and 150 ml phosphoric acid (H_3PO_4) 85% . Then the phosphoric acid (H_3PO_4) 20% solution was stirred by using magnetic stirrer for 2 hours.
b. The measuring of the pH of aquadest (distilled water) was pH = 6-7, the pH of phosphoric acid (H_3PO_4) 85% pH =0.9 and the pH of phosphoric acid (H_3PO_4) 20% was pH = 1.2
c. The bamboo carbon (BC) powder or coconut coir carbon (CCC) powder was immersed into phosphoric acid (H_3PO_4) 20% solution. for 48 hours.
d. The bamboo carbon powder (BC) or coconut coir carbon powder (CCC) in H_3PO_4 20% is further washed with aquadest/distilled water until the washing water shows a constant pH of 6-7.
e. Then filtered using filter paper. The activation carbon powder was heated again in a pyrolysis chamber at temperature of about 300^{o}C for 2 hours.
f. The refinement process as manual was carried out using Agate Mortar and pestle. And then sieved by using 100 mesh stainless steel. And then the activated carbon was stored in cookies jar.

XRD Measurements

The physics structure i.e; crystallinity of the materials used in this research was carried out using a XRD Bruker DB Advance diffractometer. This diffractometer is equipped with a CuK α X-ray source with a wavelength of 1.54060 Angstrom. The XRD spectra were recorded in range of $2\theta = 10^{o}$ to $2\theta = 90^{o}$. The X-ray tube was set at 40 kV and 40 mA. The crysrallinity study of the sample is based on the XRD spectra. The relative percentage crystallinity of the sample can be calculated by using equation (1):

$$\% \text{ Crystallinity} = (Ic/Ic + Ia) \times 100\% \quad (1)$$

The interlayer spacing (d_{hkl}) is calculated using Bragg equation as stated in equation (2) [11]:

$$d_{hkl} = \frac{n\lambda}{\sin\theta} \quad (2)$$

Where: n is diffraction order, $d_{(hkl)}$ is the interlayer spacing, θ is the diffraction angle, and λ is the wavelength of the X-ray used.
For the crystallite height (Lc) is estimated by using Scherrer's formula: as stated in equation (3).

$$Lc = \frac{0.89\lambda}{\beta_{(002)} \cos\theta_{(002)}} \quad (3)$$

where: Lc is the crystallite height, K is the shape factor = 0,89 ; $\theta_{(hkl)}$ is diffraction angle at maximum intensity, CuKα with λ = 1.54060 Angstrom, (is the wavelength of X-ray. β is Full width at half Maximum (FWHM) in radian (1 degree = 0.0174 radian).

3. Results and Discussion

The results of fabrication of BAC powder and CCAC powder are shown in figure 3.

Figure 3. BAC Powder & CCAC Powder

The Results of XRD Measurement

The XRD Spectra of bamboo carbon (BC) pyrolysis and bamboo activated carbon (BAC) is shown in Figure 4 and 5 respectively.

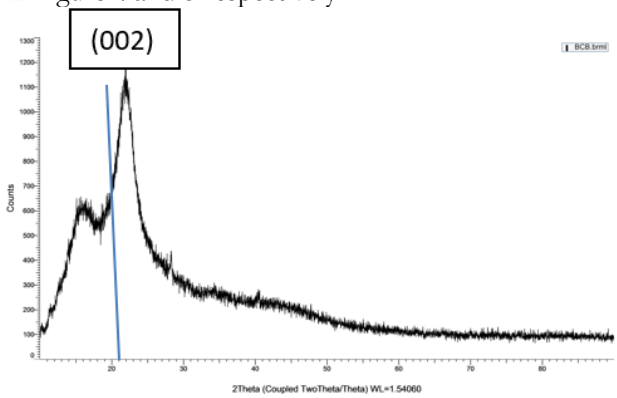

Figure 4. The XRD Spectra of Bamboo Carbon (BC) Pyrolysis

From the XRD spectra (figure 4) can be obtained that the bamboo carbon (BC) is semi-crystalline materials which consists of crystallinity phase: 29.1%, and amorphous phase: 70.9%

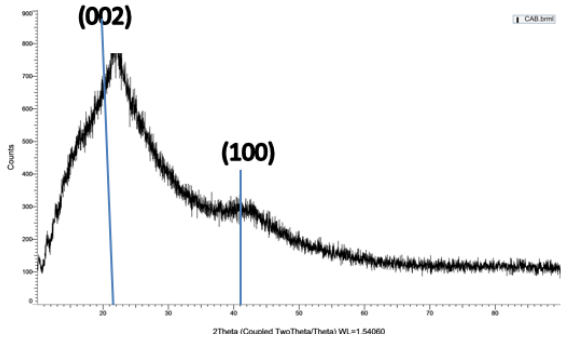

Figure 5. The XRD Spectra of Bamboo Activated Carbon (BAC)

From the XRD spectra (figure 5) can be obtained that the bamboo activated carbon (BAC) is semi-crystalline materials which consists of crystallinity phase: 18.4%, and amorphous phase: 81.6%. The characteristics of BC and BAC as stated in Table 1

Table 1. The Crystalline Structure Characteristics of BC and BAC

Sample	2θ (degree)	Plane diffract ion	Interlayer spacing (d_{hkl}) Angstrom)	Crystallite height (Lc) (Angstrom)
BC	16	–	5.60	–
	22	(002)	4.05	21.5582
	42	(100)	2.15	–
BAC	24	(002)	3.785	6.6365
	44	(100)	2.217	–

The XRD spectra of coconut coir carbon (CCC) and coconut coir activated carbon (CCAC) are shown in Figure 6. and 7 respectively.

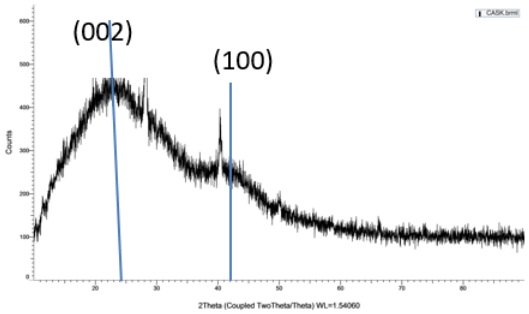

Figure 6. The XRD Spectra of Coconut Coir Carbon (CCC)

From the XRD spectra (figure 6) can be obtained that the coconut coir carbon (CCC) is semi-crystalline materials which consists of crystallinity phase: 11.3%, and amorphous phase: 88.7%.

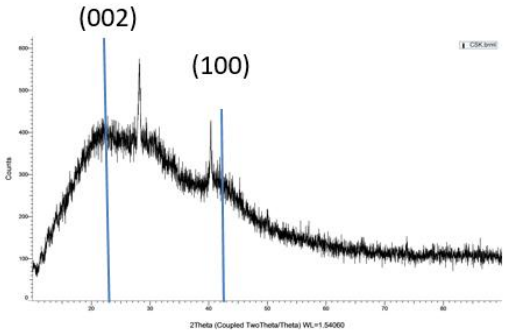

Figure 7. XRD Spectra of Coconut Coir Activated Carbon (CCAC) (Crystalinity: 13.2%, Amorphous: 86.8%)

From the XRD spectra (figure 7) can be obtained that the coconut coir activated carbon (CCAC) is semi-crystalline materials which consists of crystallinity phase: 13.2%, and amorphous phase: 86.8%.

The crystalline structure characteristics of CCC and CCAC as stated in Table 2

Table 2. The Crystalline Structure Characteristics of CCC and CCAC

Sample	2θ (degree)	Plane diffraction	Interlayer spacing (d_{hkl}) (Angstrom)	Crystallite height (Lc) (Angstrom)
CCC	22	(002)	4.05	2.8329
	28	–	3.19	–
	42	(100)	2.15	–
CCAC	22	(002)	4.05	4.9624
	28	–	3.19	–
	42	(100)	2.15	–

Figure 4 and 5 show the XRD spectra of BC and BAC indicate that the percentage crystallinity were 29.1 %, and 18.4% respectively. Table 1 shows that the interlayer spacing (dhkl) for BC is 4.05 and for BAC is 3.785 Angstrom. The crystallite height (Lc) for BAC is 6.6335 Angstrom and for BC is 21.558 Angstrom. The XRD spectra of bamboo carbon (BC) pyrolysis as shown in Figure 4 has the diffraction peak at the angle Bragg 2θ= 160. This peak

might be originated from the microcrystalline Selulosa. However, in the XRD spectra of BAC this peak disappear, due to the activation treatment using H3PO4 solution and then washing treatment using distilled water. And this also indicates that there is a structural changing in the carbon material from BC sample to BAC sample. Next, figure 6 and 7 show the XRD spectra of CCC and CCAC indicate that the percentage crystallinity were 11.3% and 13.2%, respectively the interlayer spacing (dhkl) for CCC and CCAC are the same that is 4.05 Angstrom, the crystallite height (Lc) for CCAC is 4.9624 Angstrom and for CCC is 2.8329 Angstrom as sown in Table 2.

Figure 6 and 7 show the diffraction peak at the angle Bragg 2θ= 280 and 400 might be originated from the Platinum (Pt) within the sample of BAC of about 2.048% as reported from EDX measurement results [15]. Figure 4 and 5 also figure 6 and 7 show a differences in intensity and widening band of diffraction peak at (002) because of the differences in crystalline structure for BC and BAC and also for CCC and CCAC samples.

4. Conclusion

The powder bamboo activated carbons (BAC) and coconut coir activated carbons (CCAC) can be fabricated by using H_3PO_4 as activator. The percentage crystallinity structure of BC is more higher then CCC and also the percentage crystallinity structure of BAC is more hgher then CCAC. The interlayer spacing (d_{hkl}) for BC is the same as for CCC. However, the interlayer spacing (d_{hkl}) for BAC is smaller then of CCAC. The crystallite height (Lc) for BAC is more higher then of CCAC. And also the crystallite height (Lc) of BC is more higher then of CCC. The differences in intensity and widening band of diffraction peak at diffraction plane (002) because of the differences in crystalline structure for BC and BAC and also for CCC and CCAC samples.

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