Characterization and property evaluation of low-cost electrically conductive bamboo charcoal from *Dendrocalamus sericeus* Munro.

Koranat Narkpiban¹ . Piangkhwan Kruapoo² . Rangsan Koodsamrong^{3,4} Chaiyasit Keawcharoon^{3,4} . Najiapak Sooksawat^{3,4,*}

Received: 17 May 2023/Accepted: 4 September 2023 Published online 23 January 2024

Abstract: Bamboo (Dendrocalamus sericeus Munro) is fast-growth plant with abundant biomass for conversion to fuel and various uses, including insulation and electronic applications. Bamboo charcoal produced in a low-cost furnace was separated into bamboo coal with electrical conductivity (bamboo coal 1) and without electrical conductivity (bamboo coal 2), subjected to proximate analysis and evaluation of adsorption and fuel properties. In addition, structural properties were studied based on surface area analysis using the Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods, Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). Of the two bamboo coal types, bamboo coal 1 with electrical conductivity had better

adsorptive properties, such as a greater iodine number value (240 mg g⁻¹), while the BET and the BJH methods showed that the surface area was 36.653 m² g⁻¹ with a pore volume of 0.052 cm³ g⁻¹ and a pore size of 15.292 Å. In addition, it was better for use as a deodorant and as a solid fuel for cooking with a longer total burning time (81 min). Bamboo coal 2 had lower moisture (5.75%), and ash (6.56%) contents, with a higher heating value (6913 kcal kg⁻¹), indicating that bamboo coal 2 was good for deodorant usage and could have potential for development as an insulator with a hydrophobic and fire-retardant coating material. Bamboo coal 1 had a graphite crystalline structure and could have potential with additional further graphene development for electronic uses.

*Corresponding Authors

Keywords: adsorption, bamboo, charcoal, electrical conductivity, fuel

Introduction

Bamboo has a short growth cycle, high strength, high toughness, is a renewable biomass that is easy to process with multi porosity, making it suitable for multifunctional applications. Among bamboo varieties, some species are drought-tolerant and flood-tolerant. For example, some species of bamboo in the genus Bambusa are flood-tolerant, with Bambusa beecheyana showing flood-tolerance for five months (Sungkaew and Teerawatananon, 2017). Bamboo's ability for successful colonization in varied environments results in bamboo wood varieties having both advantages and disadvantages, such as water absorption deformation cracking, light discoloration, easy to mold, easily contaminated with bacteria, easy to burn, and a good insulator (Wang et al., 2022). Applying a coating material of reduced graphene oxide and/or silver can

¹ Department of Innovation of Health and Beauty, Faculty of Science and Technology, Rajamangala University of Technology Tawan-Ok, Bangpra, Chonburi, 20110, Thailand

² Department of Science and Mathematics, Faculty of Science and Technology, Rajamangala University of Technology Tawan-Ok, Bangpra, Chonburi, 20110, Thailand

³ Department of Agricultural Engineering and Technology, Faculty of Agriculture and Natural Resources, Rajamangala University of Technology Tawan-Ok, Bangpra, Chonburi, 20110, Thailand

⁴ Center of Excellence in Agricultural Machinery, Faculty of Agriculture and Natural Resources, Rajamangala University of Technology Tawan-Ok, Bangpra, Chonburi, 20110, Thailand ⋈ najjapak so@rmutto.ac.th

improve the qualities of the bamboo surface making it antibacterial, mold-proof, corrosion resistant, photocatalytic, flame retardant, conductive, and having super hydrophobic properties (Wang *et al.*, 2022). Specific uses of bamboo are dependent on clarifying its properties for that use.

Bamboo is a traditionally used plant with a broad range of benefits to human. Native bamboo is distributed mainly in Asia, America, and Africa in both tropical and subtropical regions with various local types of soil, rainfall, temperature, and altitude (Ahmad et al., 2021). Almost all parts of bamboo are used in many processing systems of food and non-food products. Bamboo elements, such as strips, stands, flakes and fibers, can be manufactured into biocomposite products, such as bamboo strip and flattened culm sheets, stand based products, particle based products, fiber products and paper from bamboo (Chaowana and Barbu, 2017). The culms and rhizomes of Bambusa blumeana, Dendrocalamus asper, Phylostachys heterocycle, P. nigra, P. reticulata, P. makinoi, P. pubescens, and Schizostachyum zollingeri contain woody material composed of holocellulose (51.8–79.9) %) and lignin (21.4–28.5 %) (Chaowana and Barbu, 2017). Different mechanical properties, such as modulus of rupture, modulus of elasticity, shear strength, and compression strength can be evaluated to determine the appropriate use of bamboo wood for furniture and building involving delicate designs and architectural construction (Chaowana et al., 2015).

The bamboo species Dendrocalamus sericeus Munro, is known locally as 'Pai sang-mon' in Thailand and is used to make furniture (Sungkaew and Teerawatananon, 2018). It has a clump height of 10–20 m and a clump diameter of 5–10 cm (Sungkaew and Teerawatananon, 2018). Lignocellulosic biomass from bamboo is a natural polymer composite material, a renewable resource composed of polysaccharide (cellulose and hemicellulose), while the lignin is an aromatic polymer (Wang et al., 2022). Dendrocalamus giganteus and Dendrocalamus asper have been used to produce charcoal in some countries (Subyakto et al., 2012; Park et al., 2020). The time and temperature of carbonization play important roles in the yield and properties of bamboo carbon associated with fixed carbon, volatile matter, ash content, and electrical conductivity (Subyakto et al., 2012). Materials with low electrical conductivity (<10⁻⁶ S m⁻¹) and higher electrical conductivity (10⁻⁶~ 10⁵ S m⁻¹) can be classified as

insulator and semiconductors, respectively, and those with $10^4 \sim 10^5$ S m⁻¹ can be classified as conductors (Subyakto et al., 2012). Park and et al. (2020) reported that D. giganteus and Passiflora edulis carbonization at 1000°C could enhance the electrical conductivity of bamboo carbon up to 0.41 and 0.54 mS cm⁻¹, respectively. The determination of the carbon property may indicate appropriate uses, such as a fuel for boiling or grilling, as an odor adsorbent or activated carbon, or as a semiconductor. The aim of the present research was to characterize bamboo (Dendrocalamus sericeus Munro) charcoal with and without electrical conductivity produced in a low-cost furnace wrapped with insulation. Proximate analysis, heating value, iodine adsorption, and fuel properties were characterized based on the Brunauer-Emmett-Teller (BET) and the Barrett-Joyner-Halenda (BJH) methods for surface area and also using Fourier transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD).

Materials and methods

Preparation of bamboo charcoal

The 3-years culms of bamboo (Dendrocalamus sericeus Munro) were collected from a farm in Kanchanaburi province, Thailand and cut into cross-sectional sizes of 10–30 cm long and 2–3 cm wide. The bamboo samples were carbonized at 300°C for 3 h and further carbonized at 700–1,000°C for 10 h in a furnace made from a 200 L cylinder-shaped cast iron container covered with ceramic fiber sheets for thermal insulation (Park et al., 2020). Then, the carbonized sample was cooled and removed from the container, screened, and then separated into pieces having an electrical conductivity using an LCR meter (Subyakto et al., 2012). The carbonized samples with the electrical conductivity were designated as bamboo coal 1, while those without the electrical conductivity were designated as bamboo coal 2. The electrical conductivity of the bamboo samples was measured using a 4-point probe method for determining the resistivity of semiconductor materials according to ASTM F43 (Lueang-on, 2011). The multimeter was used to determine the resistivity and convert to electrical conductivity by equation 1-3 (Lueang-on, 2011). The electrical conductivity (σ) can be converted by using equation 1.

$$\sigma = \frac{1}{\rho} = \frac{1}{2\pi sF} \frac{1}{V} - ---- equation 1$$

Where, σ is electrical conductivity (S/m), ρ is resistance

resistivity (Ω m), s is the distance between probes (m) I is current (A), and F is an accuracy factor which is generally obtained by testing against standard values, which vary according to the characteristics of the sample. F can be calculated from equation 2.

$$F = F_1F_3[\ln(2)F_2/\pi \quad ---- \quad \text{equation 2}$$

Where F_l is an adjustment for the thickness of the sample, that can be calculated from equation 3, F_2 is the adjustment for the size of the sample along the plane, that is in the range of 3.84-4.36, F_3 is the adjustment of the probe position relative to the edge of the sample, that is 1, and t is the thickness of the sample (m). The width of the sample and the distance from edge of sample to probes were considered during measurement.

$$F_1 = \frac{t/s}{2\ln[\sinh(t/s)/\sinh(t/2s)]} - - equation 3$$

The samples of bamboo coal 1 had electrical conductivity in the range of 3–790 S m⁻¹ and electrical resistivity in the range of $0.001-0.391 \Omega m$.

Characterization of bamboo charcoal

Proximate analysis

Proximate analysis was used to determine moisture, volatile matter, fixed carbon, and ash contents of the bamboo samples (ASTM D7582, 2015; ASTM D5373, 2016).

Heating values

The thermal properties were analyzed using a bomb calorimeter for determination of higher and lower heating values of the bamboo samples (PARR 6300; USA) according to ASTM D5865 (2013).

Iodine number

The adsorption property of the bamboo samples was determined according to ASTM D4607 (2021) based on iodine number.

Fuel property

The water boiling test procedure for solid fuel was modified from Du *et al.*, (2013) and Khampha *et al.*, (2020). Distilled water (1,200 mL) was used in the test in a 2,500 mL metal container. The boiling test of the bamboo samples (150 g) was used to determine the boiling time, rolling boil time, and total burning

time (Du et al., 2013; Khampha et al., 2020).

Surface area analysis

The surface area analysis, pore volume, and pore size of the bamboo samples were determined using a modified method from Mahanim *et al.*, (2011). The bamboo samples were measured using the N_2 adsorption method with a surface area analyzer (Quantachrome; Autosorb iQ-C-XR-XR-XR; Austria) at a temperature of 77 K. Prior to measurements, the bamboo samples were outgassed at 300 °C under a nitrogen flow for 2 hr. About 0.03 g of sample was used in each adsorption experiment. The nitrogen adsorption isotherm was measured over a relative pressure (P/P₀) range of approximately 10^{-2} to 1. The BET and BJH methods were used to determine the surface area, pore volume, and pore size of the bamboo samples (Kikuchi *et al.*, 2018).

FTIR analysis

The functional group components in the bamboo samples were detected using an FTIR spectrometer (Nicolet 6700; Thermo Scientific, USA). A mixture of 1 mg of each bamboo sample and 200–300 mg of KBr were pressed to form a translucent pellet. Then, the IR spectra of the bamboo samples were scanned at 4000–400 cm⁻¹ (Buson *et al.*, 2018; Phothong *et al.*,2021).

XRD analysis

The X-ray diffracto-grams of the bamboo samples were obtained using a high resolution X-ray diffractometer (Rigaku: SmartLab, Japan). The method was adapted from Zhou *et al.* (2018). The samples were irradiated with a CuK α source (λ = 1.54Å) and the X-rays were generated at 40 kV and 30 mA. The samples were scanned at 2q in the range from 10° to 60° at a 0.02° s⁻¹ scanning rate.

Statistical analysis

All experiments were done in triplicate and all experimental data was analyzed using the SPSS program (version 18, SPSS, Inc. USA) using an independent sample T-test, with significant differences determined at P<0.05.

Result

Proximate analysis and heating values of bamboo charcoal

The proximate analysis of the bamboo charcoal carbonized in a low cost furnace is shown in table 1.

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Table 1. Proximate anal	lysis and	heating va	lues of bam	boo charcoal

Proximate Analysis	Bamboo coal 1	Bamboo coal 2
Moisture (%)	8.00±0.02a	5.75±0.04b
Volatile matter (%)	15.54±0.38b	23.61±0.77a
Fixed carbon (%)	$68.26 \pm 0.34a$	64.07±0.75b
Ash (%)	$8.20\pm0.06a$	6.56±0.06b
High heating value (kcal kg ⁻¹)	6702.13±9.28b	$6913.22 \pm 10.04a$
Low heating value (kcal kg ⁻¹)	6586.43±9.28b	6746.23±10.04a

Mean±SD in the same row with different lowercase letters are significantly (p<0.05) different (a>b).

Bamboo coal 1 had better properties in terms of higher fixed carbon (68.3%) and lower volatile matter contents (15.5%) in compared with bamboo coal 2 (Table 1). However, bamboo without the electrical conductivity (bamboo coal 2) had better properties for fuel use with greater high and low heating values (6,913 and 6,746 kcal kg⁻¹, respectively) and lower moisture and ash contents (5.75% and 6.56%, respectively; Table 1). Moisture content can be used to classify the quality of charcoal according to the product standard for wood charcoal for cooking (<10%) or grilling (<8%), for deodorizing charcoal (<8%), or for activated carbon (<8%, Ministry of Industry, 2004a–2004c, 2017).

Adsorption property of bamboo charcoal

The result of adsorption property showed that the bamboo coal 1 had an iodine number of the activated carbon higher (240 mg g⁻¹) than for the bamboo coal 2 (200 mg g⁻¹), suggesting that bamboo charcoal 1 with the electrical conductivity had more adsorption than bamboo charcoal 2 without the electrical conductivity. The iodine number in the bamboo charcoal from

Dendrocalamus sericeus Munro in the present study was comparable to that of D. giganteus (175 mg g⁻¹, Park et al., 2020). The iodine number of charcoal varies with the carbonization temperature. Increasing the carbonization temperature from 600°C to 1,000°C, decreased the iodine number of the charcoal in both D. giganteus and P. edulis charcoal (Park et al., 2020). In the present study, the range in the carbonization temperature (700– 1000°C) during the charcoal drying process in the furnace might have affected the adsorption properties of the bamboo charcoal samples. Bamboo coal 1 and bamboo coal 2 in the present study could qualify as deodorizing charcoal with higher level of 150 mg g-1 iodine number (Ministry of Industry, 2017). Further improvement in the iodine number to >600 mg g-1 (for powder, granules, pellets, and briquettes) or to >1,000 mg g-1 (for premiumgrade granule) could qualify the product as activated carbon (Ministry of Industry, 2004c). Activated carbon is normally used in applications such as drinking water treatment and as a pharmaceutical product for digestive disorders.

Table 2. Fuel properties of bamboo charcoal

Fuel Properties	Bamboo coal 1	Bamboo coal 2
Boiling time (min)	27.73±3.61	21.52±1.37
Rolling boil time (min)	20.90±3.35	19.99±1.51
Total burning time (min)	80.60±4.07a	69.96±3.83b

Mean±SD in the same row with different lowercase letters are significantly (p<0.05) different (a>b).

Surface characterization	Bamboo coal 1	Bamboo coal 2
BET Surface area (m²/g)	56.889	49.878
BJH Surface area (m²/g)	36.653	26.199
BJH Pore volume (cm ³ /g)	0.052	0.043
BJH Pore size (Å)	15.292	21.564

Table 3. Surface characterization of bamboo charcoal from BET and BJH methods

Fuel property of bamboo charcoal

A water boiling test was used to evaluate the fuel property of the bamboo charcoal, with the results shown in Table 2. Bamboo coal 1 had a longer total burning time (80.60 min) than bamboo coal 2 (69.96 min; Table 2). A longer burning time and rolling boil time are good fuel properties, whereas a shorter boiling time is good for solid fuel heating in cooking (Du *et al.*, 2013). The longer total burning time for bamboo coal 1 might have been due to the greater amount of fixed carbon in the fuel than in bamboo coal 2.

Characteristics of bamboo charcoal

Surface area analysis of bamboo charcoal

The electrochemical properties of activated carbon depend upon the porosity at the carbon surface. The structural changes in bamboo coal 1 and bamboo coal 2 are summarized in Table 3. The results showed that the bamboo coal 1 with the electrical conductivity had greater BET and BJH surface areas (56.889 m² g⁻¹ and 36.653 m² g⁻¹, respectively) than bamboo coal 2 without the electrical conductivity (49.878 and 26.199 m² g⁻¹, respectively). In addition, the bamboo coal 1 also had larger BJH pore volume (0.052 cm³ g⁻¹) than for bamboo coal 2 (0.043 cm³ g⁻¹). Notably, the pore size results for the bamboo coal types showed that bamboo coal 1 had a smaller BJH pore size than bamboo coal 2 (15.3 and 21.6 A, respectively).

FTIR analysis of bamboo charcoal

The electrical properties of activated carbon depend upon the porosity as well as the chemical reactivity of the functional groups at the carbon surface. The FTIR analysis identified that the carbonization process caused changes in the chemical constituents of the bamboo charcoals (Fig. 1). The peak at 1590 cm⁻¹ was ascribed in both samples to the vibration of aromatic C=C. This peak is typically found in carbonaceous materials and was consistent with the research by Phothong et al. (2021). The decrease in the intensity of the peak at 1590 cm⁻¹ for bamboo coal 1 indicated that the aromatic structure (C=C bond) of lignin fractions in the bamboo raw material was changed to a different type of aromatic compound by carbonization. A very broad peak due to the vibration of the C-O stretching was observed at 1030 cm⁻¹ in both samples. Lastly, the peak at 740 cm⁻¹ in both samples after carbonization indicated the presence of the aromatic ring in activated carbon (Nandiyanto et al., 2019).

XRD analysis of bamboo charcoal

The x-ray diffraction patterns of bamboo coal 1 and bamboo coal 2 are presented in Fig 2. Their patterns had similar diffraction peaks, but with different intensities. Fig. 2 shows there are two broad diffraction peaks in the spectrum at $2\theta = 24^{\circ}$ and 43° , respectively, corresponding to the diffraction of (0 0 2) and (1 0 0), respectively. The diffraction patterns of (0 0 2) show broad but weak reflection, indicating their disordered nature. The weak (100) reflection located at 43° suggests small islands of coherent and parallel stacked graphene sheets. The XRD results indicated the crystallinity index values for bamboo coal 1 and bamboo coal 2 were 89.03% and 88.7%, respectively.

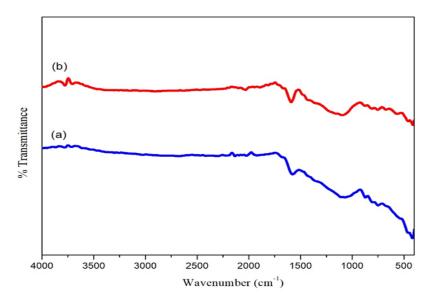


Fig.1 FTIR spectra of (a) bamboo coal 1; (b) bamboo coal 2

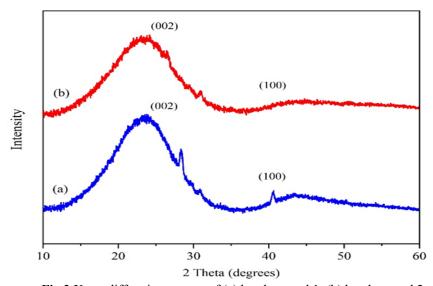


Fig.2 X-ray diffraction spectra of (a) bamboo coal 1; (b) bamboo coal 2

Discussions

In proximate analysis, the bamboo charcoal 2 in the present study was good quality (Table 1), based on the moisture content and qualified as charcoal for cooking with moisture content (<8%), volatile matter (<25%), ash content (<8%), and heating value (>6,000 kcal/kg). A better qualification with lower volatile matter (<8%) and ash content (<3%) and with greater heating value (>7,000 kcal/kg) would improve the charcoal for grilling purposes and it could then be suitable as a cleaner industrial solid fuel with steam (Ministry of

Industry, 2004a-2004c).

The results of bamboo charcoal characteristics of surface area, pore volume and pore size (Table 3) were consistent with the trends observed in the iodine adsorption of the bamboo charcoals (240 mg g⁻¹ for bamboo coal 1 and 200 mg/g for bamboo coal 2). In addition, the large surface area was correlated with increases in the pore volume. The pore size is used to classify porous materials as micropore (less than 2 nm), mesopore (2–50 nm), and macropore (greater than 50 nm; Wang *et al.*, 2022). Thus the present study

showed that bamboo coal 1 was clearly in the micropore category, with bamboo coal 2 tending to have slightly larger micropores (~2 nm).

The x-ray diffraction patterns of bamboo coals indicated that the activated carbon had the characteristics of a crystalline structure graphite (Jena *et al.*, 2021). However, the presence of the broad diffraction background and the absence of a sharp peak indicated that there was still an amorphous carbon structure (Shamsuddin *et al.*, 2016). Activated carbon, generally regarded as an amorphous carbon, has a large surface area and porosity. This can be explained by the heating process arranging the carbon into a planar layer of graphite. During carbonization, there is a random distribution of graphite structures in activated carbon. As a result, there is no integrity in the carbon crystal structure. In addition, increasing the carbonization temperature could increase the crystallinity percentage.

The present study examined the properties of bamboo charcoal separated into samples with and without electrically conductive properties. In bamboo D. sericeus culm, after the first carbonization at 400 °C for 300 min, further carbonization at 800 °C for 60 min were the optimal conditions with high levels of yield with high fixed carbon of the bamboo and with low levels of volatile matter of the bamboo carbon. However, increasing the carbonization temperature to 900 °C increased the crystallinity percentage and electrical conductivity of the bamboo (D. asper) carbon (Subyakto et al., 2012). In the present study, bamboo coal 1 had a crystalline structure like graphite (Fig.2). The functional group of activated carbon had organic compounds where most of the elements were amorphous carbons mixed with graphite crystals, which was consistent with the x-ray diffractogram results. Bamboo coal 1 had electrical conductivity in the range 3–790 S m⁻¹; bamboo carbon with electrical conductivity of 10² S m⁻¹ may be used as a load sensor for smart concrete (Subyakto et al., 2012). The electrical resistivity can be used to classify material properties as a conductor (less than $10^{-5} \Omega m$), an insulator (more than $10^{8} \Omega m$), and as a semiconductor $(10^{-5}-10^{8} \Omega m, Chand et al., 2006)$. Bamboo coal 1 in this study had electrical resistivity in the range of $0.001-0.391 \Omega m$ that could be classified as a semiconductor. Natural raw bamboo or wood-based material has an equilibrium moisture content of 15% and can be used as an insulating material or insulator (Du et al., 2002). In addition, a low moisture content of bamboo or wood-based conductive composites can be

synthesized for use in many applications, including as an organic semiconductor, organic lasers, and organic memories (Zhu et al., 2020). Carbon improvement for application as graphene has been an emerging novel use in electronic and optoelectronic devices (Wang et al., 2022). The physical structure of bamboo cellulose could be a substrate for supercapacitors electrode materials leading to water absorption, a swelling effect, and conduction to absorption electrolyte. This may include as an interval mesopore structure channel for ion diffusion to electrochemical energy storage materials (Gui et al., 2013). Recently, Wang et al. (2022) reported on biochar containing graphene synthesized from bamboo biomass and K2CO3, as an activating agent, using microwave heating with a short duration. The amorphous carbon was converted into graphene-like carbon and the graphene structure showed abundant micropores with extremely high surface areas of up to 1,565 m² g⁻¹. The process requires a catalyst and hightemperature heating. The present study investigated a low-cost preparation of a conductive bamboo sample with potential for further improvement by activating and converting it into graphene composting batteries or electronic devices.

Conclusions

The low-cost electrically conductive bamboo charcoal had a highly heterogenous heat distribution from the conversion of bamboo wood. Bamboo coal 1 with electrical conductivity performed as a cleaner solid fuel in use due to its low volatile matter compared to bamboo coal 2. Bamboo coal 1 had better adsorptive ability due to its higher iodine number to bamboo coal 2. The fuel properties of bamboo coal 1 confirmed that it had a superior total burning time compared to bamboo coal 2. The surface characterization of bamboo coal 1 indicated a higher surface area and pore volume, ut a smaller pore size than bamboo coal 2. This confirmed the greater adsorptive ability of bamboo coal 1 over bamboo coal 2. The FTIR analysis showed that both bamboo coal types were carbonaceous materials and the XRD analysis showed that they both had a crystalline graphite structure. Bamboo coal 1 appeared to have superior properties in terms of solid fuel and adsorption. Further improvement of the bamboo coal graphite structure to graphene may lead to its use as a renewable source for batteries and electronic material.

Acknowledgements

The bamboo charcoal samples were supplied by the

Khun Noi Bamboo Garden in Kanchanaburi province, Thailand.

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