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Effects on Morphology and Chemical Properties of Indonesian Bamboos by Carbonization

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ABSTRACT

A simple carbonization technique was applied to utilize Indonesian bamboo resources. Several bamboo species as betung (Dendrocalamus asper), andong (Gigantochloa pseudoarundinacea (Steudel) Widjaja), hitam (G. atroviolacea), tali (G. apus), kuning (Bambusa vulgaris var. striata), and ampel bamboo (B. Vulgaris Scharad) were selected for carbonization. Carbonization was conducted using a laboratory electrical furnace at 200, 400, 600, 800, and 1,000 1,000°C. The morphological and chemical properties of bamboos before and after carbonization were then analyzed. Betung, hitam, tali, kuning, and ampel bamboos had type IV structure which was the most common bamboo structure, while andong bambo had type III structure. Cracks in the fiber bundle were observed and became more visible with the increase of carbonization temperature. The pH showed a similar trend in all species. As a result of the electric conductivity test, carbonized andong and kuning bamboo showed higher value due to rich inorganic content. Kuning bamboo has a high value of potassium and silica content, which are suitable for use as fertilizer. The chemical structure transition of bamboo during the carbonization process was analyzed by FTIR spectroscopy, and significant changes were observed between 400 and 600°C. These results could be useful fundamental data for promoting high value-added bamboo utilization and improving research in Indonesian bamboo.

1. Introduction

Bamboo has been recognized as an important forest product of poor rural communities. It was called "poor man's timber" or "minor forest produce" for a long time (Buckingham et al. 2011). In Indonesia, bamboo occupies an area of 2 million ha, which is 5% of the world's bamboo population, consisting of 33 genera and 160 species. Bamboo has played a significant role in daily life in Indonesia. It has been used for architecture, household goods, furniture, musical instruments, textiles, and mats after simple physical processing (Febrianto et al. 2012). Timber resources occupy only 5% within Indonesian forests, while non-timber forest products account for 95%. In particular, bamboo is a significant part of non-timber forest products. Bamboo is an herbaceous plant with a very rapid growth rate. It can be harvested after 3 to 4 years and does not require expensive investment for production and maintenance. Therefore, bamboo has been

recognized as a potential resource to solve the problems of existing biomass resources with sustainability and high profitability. Bamboo is processed into various products such as charcoal, bioethanol, and biodiesel by different treatments (Asada et al. 2002; Sun et al. 2011). At one time, bamboo was simply used for poor man, but now it is called timber of wise man with the infinite possibility (INBAR 2010).

Although bamboo is an essential material in Indonesia, the usage of bamboo has been steadily decreased due to socio-economic reasons such as settlement plans, large-scale agriculture, forestry enterprises, and ranching in the large cities (Nair et al. 2021). Household items using bamboo have been replaced by new materials such as plastics on account of the overall improvement of living life. Recently, Indonesia's export of bamboo resources has been gradually declined behind Vietnam, Myanmar, and India (UN Comtrade 2014).

Nevertheless, the Indonesian government still regards bamboo as an important agroforestry product. Under the particular goal of promoting bamboo's usage in daily life and replacing wood resources, the bamboo industry has been developed around Central Java, West Java, East Java, Bali, and Yogyakarta. Several strategies have been established to improve the social images of bamboo and make bamboo more competitive products (Hardiani et al. 2014). The strategy could be summarized as promoting the use of bamboo and establishing the bamboo industry which local community participation. At first, the process of increasing market value on bamboo materials should be carried out. Currently, the bamboo industry is mainly concentrated on simple processing because most of the regions in Indonesia are lag behind except big cities. It is essential to make high value-added bamboo through simple development. Various studies on bamboo have been carried out, such as the use of bamboo for oriented strand boards (Febrianto et al. 2015; Maulana et al. 2018) and nanomaterials (Jang et al. 2013). Although Indonesia is the third-largest bamboo growing region after India and China, research and industrial investment have not been done to a greater extend. In addition, incentives for the local community involved in the bamboo site should be increased for the sustainability of bamboo resources.

In the past, the use of bamboo charcoal has been confined to fuels for household, iron melting, and fertilizer (Nurhayati et al. 2006). Currently, new utilizations of bamboo charcoal were discovered, and various products have been on the market. For example, bamboo charcoal has been considered to replace activated carbon. This is useful to dyeing and wastewater purification processes in paper mills due to its low production cost (Hameed et al. 2007). Bamboo charcoal is used to adjust the pleasing environment in daily life. When cook rice or boil water, adding bamboo charcoal can increase the taste and quality of water by soluble minerals (Park 2007).

It was recognized that carbonization processing was selected as a high-value and simple method for the underdeveloped regions of the Indonesian community. In the previous work, several promising bamboo species from Indonesia have been selected for carbonization by simple carbonization processing, and the physical, morphological, and fuel properties of the bamboo charcoal were evaluated (Park et al. 2018, 2019, 2020). In this study, the investigations of chemical properties were performed to understand the carbonization mechanism of Indonesian bamboo.

2. Materials and Methods

2.1. Materials

Betung (Dendrocalamus asper), andong (Gigantochloa pseudoarundinacea (Steudel) Widjaja), hitam (G. atroviolacea), tali (G. apus), kuning (Bambusa vulgaris var. striata), and

ampel (*B. Vulgaris* Scharad) bamboos were selected in this study. These bamboo species were 4-5 years old, collected from the experimental bamboo plantation of IPB University, West Java, Indonesia (6° 20' 21" S, 106° 33' 58" E). Detailed information on the raw materials used in this study is presented in **Table 1**.

Davamatar	Species								
rarameter	Betung	Andong	Hitam	Tali	Kuning	Ampel			
Node length (cm)	37.1	42.5	59.5	34.4	24.3	33.3			
Diameter (cm)	15.5	14.0	12.1	8.3	8.1	7.9			
Thickness (mm)	16.5	22.4	12.6	10.9	9.7	17.4			
Density $(g \text{ cm}^{-3})$	0.56	0.71	0.71	0.60	0.67	0.77			

Table 1. General information of bamboo materia
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2.2. Methods

2.2.1. Sampling

During the procurement of the samples, the second node of bamboo culm from the ground level was taken and then air-dried for 3 months. The 10 strips of 25 mm widths were prepared by cutting nodes of bamboo according to the diagram shown in **Fig. 1**. Strips were cut at 40 mm intervals in the fiber direction and divided into 6 specimens. The specimens are almost similar in structure due to the homogeneity of the top and the bottom division of the bamboo stem. Sixty specimens were prepared for each bamboo species. Ten specimens were collected from each strip, and they were carbonized at different target temperatures.



Fig. 1. Diagram of sample preparation and visual appearance of carbonized bamboo samples.

2.2.2. Carbonization of bamboo

The samples were prepared with a size of 25 mm wide and 40 mm long. Following the methods of Park et al. (2009), the samples were wrapped in paper and aluminum foil and placed in a small rectangular heat-resistant container with a cover to prevent oxidation during the carbonization process. The target temperature was varied from 200 to 1,000°C, and samples were carbonized in a laboratory electrical furnace (TST BT-F724, Korea). Carbonization was carried out for 2 h at the target temperature. At the end of the carbonization process, carbonized samples

were taken out of the electric furnace and cooling down for several hours at room temperature. The carbonized bamboo samples were pulverized and sorted into 150 mesh sieves.

2.2.3. Evaluation of morphological properties

Sample preparation was conducted according to the method described by Kwon et al. (2006). The cross-section of the raw bamboo sample was prepared with a sliding microtome (WSL lab microtome, WSL Startseite, Swiss) after softening it in water for 2 weeks. After the carbonization, the specimens were sliced to 5 mm \times 5 mm (radial \times tangential), and the central part was broken to obtain a clean and parallel cut surface. These specimens were coated with gold and palladium and then observed with a scanning electron microscope (SEM, COXEM-30, COXEM, Korea).

2.2.4. Evaluation chemical properties

The hydrogen ion concentration (pH) was determined according to TAPPI 435 method (TAPPI 2006). The mixed solution was prepared from 1 g oven-dried powder sample and 100 ml distilled water. The solution was then boiled for 10 minutes and cooled down to room temperature. The pH was measured with a pH meter (HM-30R, SECHANG, Korea).

The electric conductivity of raw and carbonized samples was measured according to KS M ISO 6587 (KSA 2020). The electric conductivity indicates the strength of electrolyte ions in the solution and is expressed as a reciprocal of resistance. As inorganic content in the solution increased, the electric conductivity increased. The mixed solution of 1g oven-dried sample powder and 100 ml distilled water at room temperature was measured by an electric conductivity analyzer (CM-30R, TOA DKK, Japan). The electric conductivity of the solution as distilled water, clean tap water, and seawater was $0.5 \,\mu\text{m.cm}^{-1}$, $50 \,\mu\text{m.cm}^{-1}$, and $53 \,\mu\text{m.cm}^{-1}$, respectively.

The inorganic contents of raw and carbonized bamboo samples were determined using an energy dispersive x-ray analyzer (410-EDS, Bruker, Germany). The EDS analyzer was operated by the interaction between sources of x-ray stimulation and unique electromagnetic emission spectra of elements in samples. The samples processed to a size of 10 mm \times 10 mm (radial \times tangential \times longitudinal) were coated with gold. After setting the electric stimulation of the equipment to 10 kV, the contents of silicon, potassium, calcium, and magnesium were measured.

Since all compounds of organic and inorganic molecules show different energy absorption patterns in the infrared region, a unique spectrum appears according to respective materials. It is used for quantitative analysis and qualitative analysis of material or components through Fourier transform spectroscopy (FTIR). The FTIR spectrophotometer (Nicolet 6700, Thermo Scientific, USA) was used to observe the chemical structure changes of raw and carbonized bamboo samples. The spectrum was obtained at the mid-infrared region of 4,000 to 650 cm⁻¹. Spectral analysis was conducted by using OMNIC 9.2 software.

3. Results and Discussion

3.1. Morphological Properties

The vascular bundle that is the main structure of bamboo was investigated to represent the cross-section anatomical characteristics of bamboo species in this study. SEM images of carbonized bamboo samples were shown in **Fig. 2**. Grosser et al. (1971) classified the types of the

bamboo vascular bundle into four types. Type I consists of only the vascular center of the vascular system, and type II consists of the vascular center of the vascular system in which the rear wall cells adjacent to the protoxylem are expanded. Type III is composed of one fiber located in the center of the tube and the inner direction. Type IV takes the form of fiber in the outer direction in the form of type III. As a result, vascular bundle type IV was observed at Indonesian bamboo species, excluding andong bamboo.



Fig. 2. The SEM images of bamboo samples according to carbonization temperature.

Andong bamboo mainly consisted of type III and rarely in type IV. The fiber bundles located in the bottom part of the vascular bundle seemed to form a crescent shape. On the other, the fiber bundles located in the upper part of the vascular bundle were slightly different depending on species. The fiber bundles of tali and kuning bamboos were often observed to be squashed round shape. Liese (1980) observed the same tendency in Indonesian bamboo. Even though 40% or more reduction of bamboo volume occurred during carbonization, the bamboo structure was still preserved. The elements in the vascular bundle, such as vessel, xylem, and intercellular space, were maintained. No particular trend was observed in all bamboo species used in this study. However, it was found that the tali bamboo has a somewhat smaller vascular center than other species. In addition, with the increase of carbonization temperature, the fiber bundles of betung and kuning bamboos were often cracked or depressed. It was confirmed that the starch contained in the parenchyma cell did not appear to be lost. It was preserved as a hardened form.

3.2. Chemical Properties

3.2.1. pH and electric conductivity

The pH and electric conductivity of carbonized bamboo samples were shown in **Fig. 3**. It was observed that control bamboo samples changed from acid to alkaline as increasing carbonization temperature. The pH is expressed as a logarithm of the number of grams of hydrogen ions contained in a liquid. It was confirmed that hydrogen was discharged in various forms during pyrolysis that brought about the increase of pH value. The trend of increasing pH was classified into two types. The rapid changes from 200 to 600°C carbonization occurred on betung, andong, tali, and kuning bamboos. This phenomenon seems to be related to the increase of ashes due to the intensive decomposition of bamboo components during the carbonization process (Park et al. 2020). Ampel bamboo showed a similar tendency, but the increase interval was slightly shorter. The pH of hitam bamboo steadily increased. At the carbonization temperature of 1,000°C, the pH of carbonized bamboos reached 9 or more. At the carbonization temperature of 600°C, the pH of all specimens was above 7, which is considered as the minimum carbonization temperature for using carbonized bamboo as a neutralizer. Park (2007) reported that the pH of Indonesian carbonized bamboo is generally higher than that of carbonized Moso bamboo.



Fig. 3. The pH and electric conductivity of carbonized bamboo samples.

The electric conductivity is an index of the strength of electrolyte ions in solution and is expressed in terms of inverse of resistance. Therefore, the greater the number of electrolyte ions in the solution, the higher is the electric conductivity. Bamboo was known to contain a large amount of water-soluble inorganic substances, and an electric conductivity test was conducted to compare that Park (2007). Moreover, the parenchyma cell of bamboo has abundant starch. However, starch was assumed to have little effect on electric conductivity because it was melted or burned out during the carbonization process. It was observed that the electric conductivity of all bamboo species increased until carbonization temperature of 800°C, and then slightly decreased after carbonized at 800°C. It is highly related to the increase of ash content, which is mainly composed of inorganic matter. In addition, at 800 and 1,000°C carbonization, the amount of silica content dissolved in the solution was not significant due to the hardening process. Especially, andong and kuning bamboo samples seemed to have a relatively higher inorganic content, which may be attributed to the high inorganic content observed by the EDX test. The electric conductivity of betung, hitam, tali, and ampel bamboos showed the same tendency within the range of 0 to 1,000°C.

3.2.2. Inorganic contents

The inorganic substance of carbonized bamboo was investigated. The inorganic substance is one of the main valuation matters for biomass resources. Since it is possible to supply nutrients that help plant growth, it increases the potential utilization as a fertilizer. However, the inorganic substance has a negative effect on fuel use due to the decrease of fixed carbon (Basu 2018). The content changes of potassium, silica, magnesium, and calcium according to carbonization temperature are shown in **Fig. 4** and **Fig. 5**.



Fig. 4. The potassium (left) and silica (right) of carbonized bamboo samples.



Fig. 5. The calcium (left) and magnesium (right) of carbonized bamboo samples.

The potassium content was the highest rate among inorganic content in this study. Moreover, it was observed to be abundant in andong and kuning bamboo. Potassium content increased until carbonization temperature of 600°C and then decreased as increasing temperature. The other bamboo samples showed a similar change tendency. The potassium content in other species was small, and it was hard to find out significant change according to carbonization temperature. The highest value of silica content was detected in kuning bamboo. The other bamboo species had similar silica contents. The magnesium and calcium content of betung bamboo showed higher contents than others. The other species showed a higher rate of magnesium and calcium after carbonization at 400°C. On the other hand, betung bamboo represented the higher rate after carbonization at 600°C. Ampel bamboo, which had the higher calorific value in previous studies (Park et al. 2019), was confirmed to have a lower content of inorganic content. Hitam and tali bamboo showed low value regardless of carbonization temperature and type of inorganic substance, and the cause is analyzed to be due to differences in species.

3.2.3. FTIR Spectroscopy

The ATR-FTIR spectroscopy was performed to investigate the change in functional groups of bamboo components, and the spectrums of all bamboo samples were shown in **Fig. 6-8**. Information on the binding structure assigned to detected peaks was according to **Table 2**.



Fig. 6. FTIR spectra of betung (left) and andong (right) carbonized bamboo samples.



Fig. 7. FTIR spectra of hitam (left) and tali (right) carbonized bamboo samples.



Fig. 8. FTIR spectra of kuning (left) and ampel (right) carbonized bamboo samples.

Band position (cm ⁻¹)	Band assignment	Range (°C)		
3346-3332	O-H stretching (hydrogen bond)	0-200		
2896-2891	C-H stretching in methyl- and methylene group	0-200		
2359	Weak additional band, CH ₃ of alkane	0-1000		
1732-1727	C=O stretching in unconjugated ketone	0-600		
1602-1601	C=C stretching mode unconjugated alkene	0-600		
1510-1506	C-C in aromatic skeletal vibration	0-200		
1455-1453	C-H deformation in methyl- and methylene group	0-200		
1422-1421	C-H in-plane deformation with aromatic ring stretching	0-200		
1367	In-plane deformation vibration of phenolic-OH	0-600		
1323-1317	C-O stretching of syringyl ring	0-200		
1158-1155	C-O-C stretching in pyranose rings	0-200		
1031-1023	C-H in-plane bending mode of phenylalanine	0-200		
896	C-H stretching out of plane of aromatic ring	0-200		
832	β (CH) ring, γ (CC) with aromatic ring	0-200		

Table	2.	Band	positions	and	assignments	in	the	infrared	spectra	of	carbo	nized	bamboo	samples
I abit		Duna	positions	unu	assignments	111	une	mmulou	spectru	O1	curoo	mzou	oumooo	Sumpter

Source: Chen et al. (2010); Muller et al. (2009).

Various peaks were found in all of the raw bamboo samples. For example, the major peaks which contribute to cellulose concentrated at n(C-O) are around 1,100 cm⁻¹, and n(OH) of hydroxyl groups is around 3,300 cm⁻¹. Furthermore, the aromatic peak of lignin between 1,400 cm⁻¹ and 1,700 cm⁻¹ were detected. The results showed that the interval of carbonization temperature remarkably changed the chemical structure of bamboo, and the tendency of all species was very similar. Most of the measured peaks of raw bamboo samples were remained up to 200°C. Some peaks related to cellulose were rarely found at 400°C. At 600°C, it was challenging to detect peaks associated with lignin. The flat spectrum curve with most peaks completely disappeared was observed at 800 and 1000°C. According to results, the carbonization temperature of 400 and 600°C is regarded as a critical change point of bamboo charcoal. The tendency of the spectrum curves in this study is similar to Chen et al. (2015). They studied the carbonization mechanism of bamboo in the range of 300 – 700°C and mentioned that most bonds were disappeared at 400°C.

4. Conclusions

Fundamental data of carbonized bamboo manufactured by simple carbonization technique were collected in this study. The carbonized Indonesian bamboo is expected to be advantageous for soil improvement and water purification due to its high inorganic content and electric conductivity. Furthermore, carbonized bamboo could be simply applied in an underdeveloped area that requires water supply facilities. The results of the carbonization mechanism on bamboo charcoal can be used to establish a suitable manufacturing plan. Although this study is in the foundational research stage, it is hoped that this study will contribute to the research activity and high value-added projects on the utilization of Indonesian bamboo.

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