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THE INFLUENCE OF H_3PO_4 CONCENTRATION ON THE YIELD, POROUS STRUCTURE, AND SURFACE CHEMICALS OF SARAWAK WILD BAMBOO ACTIVATED CARBON

(Pengaruh Kepekatan H_3PO_4 Terhadap Hasil, Struktur Berliang dan Bahan Kimia Permukaan Karbon Teraktif Buluh Liar Sarawak)

Mahanim Sarif[1,*], Hashim Bojet[2], Tumirah Khadiran[1], Rafidah Jalil[1], Puad Elham[1], Nicholas Andrew Lissem[2], Johari Zainudin[2], Wan Ching Ching[2] and Rickey Anak Dayus[2]

[1]*Wood Chemistry and Non Wood Utilization Programme, Forest Products Division, Forest Research Institute Malaysia, 52109 Kepong, Selangor, Malaysia*
[2]*Sarawak Timber Industry Development Corporation (STIDC), Jalan Stadium, Petra Jaya, 93050 Kuching, Sarawak*

[*]*Corresponding author: mahanim@frim.gov.my*

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Abstract

Activated carbon (AC) is a versatile porous material with numerous applications in various industries. In this study, ACs with both specific surface area and mesoporous developed structure were prepared using Sarawak wild bamboos, i.e., *Gigantochloa levis* (beting) and *Bambusa vulgaris* (aur), as a carbon precursor. The bamboos were chemically activated with phosphoric acid (H_3PO_4) at various concentrations, and then carbonized using a tubular furnace at 500 °C for 3 hr under an inert nitrogen gas flow. The proximate and ultimate analyses of AC were measured. The chemical and porous structure of AC were characterized using Brunauer, Emmett, and Teller (BET), Fourier transform infrared spectroscopy (FTIR), and field emission scanning electron microscopy (FESEM). The results show that by increasing the H_3PO_4 concentration, the surface area of bamboo AC was increased. The AC yield between 58% and 62% was obtained at 1%–9% v/v H_3PO_4 impregnations. The highest surface area of 1319 and 1285 m^2/g were obtained at 9% v/v H_3PO_4 impregnation of beting and aur bamboos, respectively. Therefore, ACs can be prepared using low H_3PO_4 impregnation concentration, but high quality ACs comparable to other ACs from different biomass with good yield and textural characteristics are also producible. This could lower the production cost of ACs from bamboo due to the use of cheap and novel raw materials from different species of bamboo by using only the minimum concentration of activating agent in the production.

Keywords: activated carbon, bamboo, chemical activation, surface area

Abstrak

Karbon teraktif (AC) ialah bahan berliang serba boleh dengan pelbagai aplikasi dalam pelbagai industri. Dalam kajian ini, AC dengan kedua-dua kawasan permukaan tertentu dan struktur liang meso telah disediakan menggunakan buluh liar Sarawak; *Gigantochloa levis* (beting) dan *Bambusa vulgaris* (aur) sebagai prekursor karbon. Buluh tersebut diaktifkan secara kimia dengan asid fosforik (H_3PO_4) pada pelbagai kepekatan, dan kemudian dikarbonisasi menggunakan relau tiub pada suhu 500 °C selama 3 jam dengan aliran gas nitrogen. Analisis proksimat dan unsur karbon, hidrogen, nitrogen, sulphur ke atas AC telah diukur. Struktur kimia dan keliangan AC dicirikan menggunakan, Brunauer, Emmett, dan Teller (BET), spektroskopi inframerah transformasi Fourier (FTIR) dan Mikroskopi elektron pengimbasan pelepasan medan (FESEM). Keputusan menunjukkan bahawa dengan meningkatkan kepekatan H_3PO_4, luas permukaan AC buluh meningkat. Hasil AC dalam julat antara 58% hingga 62% diperolehi pada impregnasi 1% - 9% v/v impregnasi H_3PO_4. Luas permukaan tertinggi 1319 m^2/g dan 1285 m^2/g diperolehi pada 9% v/v H_3PO_4 impregnasi buluh beting dan aur. Oleh itu, AC boleh disediakan menggunakan kepekatan impregnasi H_3PO_4 yang rendah, tetapi AC berkualiti tinggi setanding dengan AC lain daripada biojisim berbeza dengan hasil yang baik dan ciri tekstur juga boleh dihasilkan. Ini boleh mengurangkan kos pengeluaran AC daripada buluh kerana penggunaan bahan mentah yang murah dan baru daripada spesies buluh yang berbeza dengan hanya menggunakan kepekatan minimum agen pengaktif dalam pengeluaran.

Kata kunci: karbon teraktif, buluh, pengaktifan kimia, luas permukaan

Introduction

Activated carbon (AC) is a unique and multifunctional substance due to its high porosity and large surface area, which leads to high adsorption capability. AC pores exhibit strong van der Waals forces, which are linked to its adsorption capabilities. ACs are widely utilized in various applications, including water purification, gas storage, water treatment, as a catalyst, and as a material for double layer capacitors [1-3], environmental remediation, air purification, food processing, medicinal uses, and recovery of metals in hydrometallurgy [4]. The demand for AC will continue to rise as its applications expand.

Commercial AC is often produced from coal, which is a non-renewable resource with limited availability. Biomass has emerged as an alternate raw material for the manufacturing of AC to circumvent this constraint. Approximately 80%~85% of the total AC production is derived and explored from non-renewable coal-based resources with the remaining from renewable sources such as agricultural residues, wood [4], palm, coconut shell [4], coffee endocarp, fine cone biomass [1], hazelnut shells, olive, peach, apricot and cherry stones, grape seeds, and eucalyptus leaves [3]. Bamboo is another biomass material that has a good potential to be used as a carbon precursor to produce AC [5-8]. Bamboo is a fast-growing plant, rich with lignin (32%–33%) and alpha cellulose (45%–46%), which makes it suited to be used as a carbon precursor to produce AC. The porous structure (micropores, mesoporous, and macropores) of AC from biomass is made up of cellulose and lignin. The chemical composition of the raw material and the parameters of the manufacturing process determine the characteristics of AC [1]. In other words, the structural properties of AC, i.e., porous structure (pore size and surface area) and surface chemistry depend on the carbon precursors and the manufacturing processes.

There are two manufacturing processes of AC known as physical activation and chemical activation methods. Physical activation is recognized as a two-step method because it involves carbonization followed by activation. Chemical activation is a single-step method in the presence of chemical activator, whereby carbonization and activation take place simultaneously. Nevertheless, chemical activation has shown more advantages over physical activation because it produces ACs with higher yield and higher surface area even at moderate temperatures with only one step activation [4, 9]. Basically, carbonization is the conversion of raw materials into charcoal and the formation of initial porosity through thermal decomposition resulting in a higher carbon content without non-carbon components in the substance. Meanwhile, activation is a process to create advanced porosity by opening previously inaccessible initial holes, forming new pores, and enlarging existing pores [10].

The most common chemical activators used in chemical activation method to produce AC are H_3PO_4, alkali metal compounds such as sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium carbonate (Na_2CO_3), and potassium carbonate (K_2CO_3), and zinc chloride ($ZnCl_2$) [2, 11-12]. Among the alkali metal compounds, KOH is often carried out at high temperatures of 700~900 °C with a high impregnation ratio (IR = KOH: biomass precursor) of 5:1 and very effective in creating micropores, small mesopores, and very high specific BET surface area of above 2000 m^2g^{-1} [3]. However, chemical activation using KOH activation has low carbon yield and has detrimental impacts on human health and environment. On the other hand, H_3PO_4 can be operated at low temperatures of 400~600 °C with a low IR (weight ratio of H_3PO_4 to biomass precursor) of 3:1, results in BET surface area of approximately 1000~1500 m^2g^{-1} consisting of mainly mesopores. As a dehydrating catalyst, H_3PO_4 does not only promote bond cleavage reactions but also facilitate crosslinking via cyclization, condensation, and forming a layer of linkage such as phosphate and polyphosphate esters, which could protect the internal pore structure, and thus prevent excessive burn-off in carbon activation. With the considerations of environmental impacts, energy cost, and carbon yield, H_3PO_4 is highly attractive and has been in demand for large-scale AC production in recent decades [4].

In this paper, AC was prepared using two different Sarawak wild bamboo species, i.e., *Gigantochloa levis* (Beting) and *Bambusa vulgaris* (Aur) through the H_3PO_4 chemical activation. They can easily be found in almost all Sarawak regencies. To the best of our knowledge, there are no previous reports on the preparation of AC from Beting) and Aur bamboos using low concentration H_3PO_4 chemical activation. There is a shortage of data, particularly regarding the properties of AC. Therefore, the effects of H_3PO_4 concentration on the AC yield, porous structure, and surface chemicals of beting and aur bamboos were examined in this study.

Materials and Methods

Materials

Two different species of Sarawak wild bamboo, i.e., *Gigantochloa levis* (beting) and *Bambusa vulgaris* (aur), approximately aged 4 years old were supplied by Sarawak Timber Industry Development Corporation (STIDC). Both bamboos were originated from Kota Samarahan, Sarawak, Malaysia. Mixed portion of bamboos (top, middle, bottom) were used as the carbon precursor throughout the study. The bamboo internodes were cut and chipped into small size and oven-dried at 103±2 °C for 48 hr to remove water content. The dried bamboos were ground and sieved through a 1.0-mm sieve. The ground bamboo was known as dried bamboo particle. The analytical grade of H_3PO_4 was used as a chemical activator. The chemicals and reagents used in this study were purchased from Sigma-Aldrich.

Preparation of bamboo activated carbon

The method used to prepare the bamboo AC was adopted from Tumirah et al. [12]. Approximately 5g of dried bamboo particle was impregnated with 50 mL of different concentrations (1%, 3%, 6%, and 9% v/v) of H_3PO_4. The samples were shaken overnight using an orbital shaker at 100 rpm at 70±3 °C. Then, the samples were heated using sand bath until all the excess water was evaporated. The chemical activation of bamboo was carried out using a tubular furnace at 500 °C for 3 hr under an inert nitrogen gas flow. The resulting bamboo ACs were obtained after washing with hot water and oven-dried at 103±2 °C for 24 hr. The experimental set up for chemical activation is shown in Figure 1.

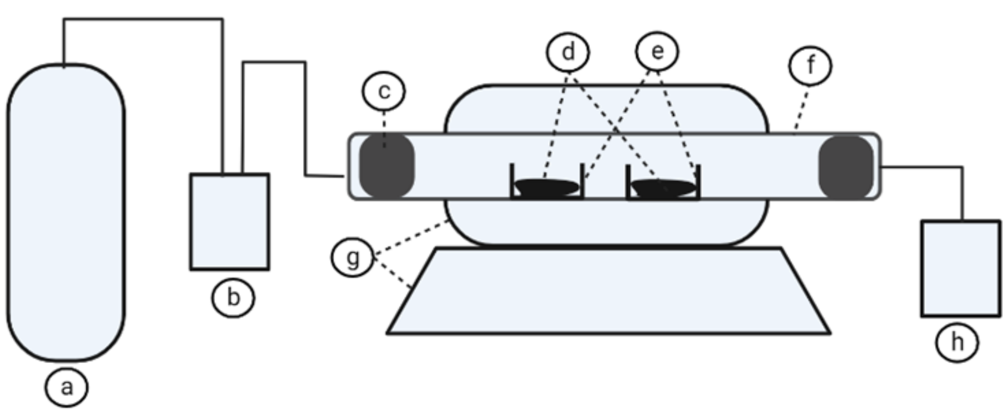

Figure 1. Experimental set up for chemical activation. Notes: (a) N_2 gas tank (b) Flow meter (c) Glass Block (d) Activated Sample (e) Sampling boat (f) Glass tube (g) Furnace (h) Water bottle (N_2 flow indicator)

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Characterization of bamboo activated carbon: Chemical composition

The internodes of bamboos were ground into 250 µm size and used for the chemical analysis. The resulting material was placed in a sealed plastic container and labelled for the chemical analysis which comprises holocellulose, alpha cellulose, and lignin analysis using the test method of Wise et al. [13], TAPPI 203 om-93 [14], and TAPPI T222 om-02 [15], respectively. Additionally, holocellulose is a necessary preparatory stage to determine the alpha-cellulose content.

Proximate and ultimate analysis

The proximate analysis was carried out to determine the proximate amount of moisture content, volatile matter content, and ash content using the British Standard BS EN 14774-2:2009, British Standard EN 15148:2009, and British Standard EN 14775:2009, respectively.

The ultimate analysis was performed using CHNS Analyzer, LECO Instrument Model 628 at Global Testing and Consultancy for Rubber (G-TACR), Malaysian Rubber Board (MRB). The analysis was carried out to determine carbon (C), hydrogen (H), nitrogen (N), and sulphur (S) content in the bamboo samples.

Fourier transform infrared spectroscopy (FTIR)

The sample was ground and sieved with a 150-µM mesh. Exactly 2 mg of each sample was mixed with 100 mg of potassium bromide (KBr) powder, and the mixture was further ground and pressed into a 13-mm diameter disc. The IR spectra were recorded on a Spectrum 100 FTIR spectrometer (Perkin Elmer, CA, USA), equipped with a mid-infrared deuterated triglycine sulphate (DTGS) detector. The spectra were obtained in the frequency range of 4000–450 cm^{-1} with a resolution of 4 cm^{-1} and with a total accumulation of 16 scans.

Percentage of yield

The yield of bamboo AC is defined as a ratio of weight of the resultant bamboo AC to that of the bamboo raw. The yield of bamboo AC is measured using Equation 1 as follows [16]:

$$\text{Yield of bamboo AC (\%)} = (W_1 / W_0) \times 100 \qquad (1)$$

where, W_0 is the mass of bamboo raw and W_1 is the mass of carbonized bamboo after washing with hot water and oven-drying.

Surface area and porous structure characterization

The surface area and porous structure of bamboo AC were characterized using the nitrogen adsorption-desorption technique at 77 K of accelerated surface area and porosimeter analyser (Micromeritic, ASAP 2020). The porous structure of bamboo AC is referring to pore size, pore size distribution, pore volume, and surface area. The surface area of bamboo AC was analysed using the BET equation, while porous structure was calculated using the desorption Barret–Joyner–Halenda (BJH) equation. Both equations are as in the instrument software package.

Surface morphology characterization

The bamboo AC samples used for FESEM analysis were prepared by placing the dried samples onto the specimen stub. The sample surface was sputter coated with a thin film of platinum before any observation was made. The morphology structure of AC was observed under the Model: Nova NanoSEM 230, THERMO SCIENTIFIC formerly known as FEI at a magnification of 5000× at a scanning voltage of 10 kV.

Results and Discussion

Characteristics of raw material: Chemical composition

In general, biomass contains three main cellulosic components (hemicelluloses, cellulose, and lignin) and closely associated in a complex structure. Previous study reported that the range of cellulose and hemicelluloses content for various types of bamboo is from 37% to 47% and 15% to 30%, respectively [17]. The characteristics of beting and aur bamboos were evaluated and compared with other literatures. The chemical contents of bamboos for this study were (44%–46 % cellulose, 25%–33% hemicellulose, and 32%–33% lignin. This result is close to the chemical composition of other bamboo species as depicted in Table 1. During the carbonization of biomass, the hemicelluloses and cellulose decompose mostly into volatile products, while the lignin content is more difficult to decompose and contributes to the formation of a solid residue called char. Meanwhile, the fixed carbon and ash content decreased with the increase in the volatile content [16]. The chemical composition of bamboo is almost similar to that of the woody biomass materials [18, 19]. High phenolic content in lignin compound of biomass makes it important in the AC preparation because it leads to higher carbon yields than those obtained from the other two main macromolecular compounds of biomass: cellulose and hemicellulose [20]. Thus, Beting and Aur bamboos have great potential as a precursor to produce high quality AC because the result is within the range of chemical content. The formation of micropores and macropores of AC are strongly influenced by cellulose and lignin content of raw material. Table 1 also presents that the density and thickness of beting and aur bamboos was around 706–785 kg/m^3 and 8.16–10.51 mm, respectively.

Table 1. Chemical and physical analyses of different bamboo species

Analysis	**Unit**	***Gigantochloa levis* (beting) (This study)**	***Bambusa vulgaris* (aur) (This study)**	***Gigantochloa nigrociliata* (Tabah)[17]**	***Schizostachyum brachycladum (*Tamblang*)*)[17]**
Chemical					
Alpha cellulose	%	46.39	44.97	44.94	42.52
Hemicellulose	%	25.73	33.17	16.99	16.73
Lignin	%	32.25	33.33	22.91	21.23
Physical					
Thickness	mm	10.51	8.16	-	-
Density	kg/m^3	785	706	-	-

Proximate and ultimate analysis

The proximate analysis of raw bamboo is very important to determine the quality of AC produced and the distribution of its contents. Four important parameters that need to be measured comprises moisture content, volatile matter content, ash content, and fixed carbon content. The moisture content present in the sample can also be considered as water vapor when it is heated to high temperatures. Hence, the volatile matter content and moisture content tend to leave the sample when heated. In view of the proximate analysis data, beting and aur bamboos show high moisture content (32%–41%) and high percentage of volatile matter (83%–90%), while the ash content and fixed carbon content of the bamboo sample was 1.4%–3.84% and 8.58%–12.88%, respectively. This gives an overview about the properties and components of bamboo [20]. The high volatile matter and low ash content of biomass resources enable them suitable to be used as starting materials to produce ACs [21]. High volatile matter content usually reduces the solid yield in the carbonization stage, while low inorganic content is vital due to their abilities to produce low ash and high fixed carbon content [22]. Apart from ash content, fixed carbon content is very important to determine the quality of AC produced from raw bamboo. The content of fixed carbon will be increased after converting it to AC. Due to its relatively high volatile matter content and low ash content, bamboo has the potential as a suitable precursor for conversion to AC. The low ash content indicates that inorganic impurities would have little impact on pore growth during the activation process. Since minerals including silica, alumina, iron, magnesium, and calcium make up majority of ash's composition, ash is an unwanted contaminant that presents in AC [5].

The ultimate analysis shows that raw bamboo and AC has 40%–43% and 69.85% of carbon, while 1.40%–3.84% and 7.22% of ash, respectively. It was predicted that compared to the raw material, AC has higher amount of carbon and ash. Past study investigated the carbonization of Pawlonia wood with phosphoric acid and discovered another pattern that is also comparable [21]. Besides the experimental conditions of the carbonization and activation steps, the porous structure of AC is also influenced by the original nature and structure of the starting material. The results of proximate and ultimate analyses from different biomass of wood and non-wood are shown in Table 2.

Table 2. Proximate and ultimate analyses of biomass and its activated carbon

Analysis	Bamboo (This study)	Bamboo [17]	Paulownia Wood [21]	Bamboo Activated carbon [5]	Pawlonia Activated carbon [21]
Proximate (%)					
Moisture Content	31.60-40.92	7.67-9.27	3.50	5.27	2.30
Volatile matter	83.29-90.02	83.48-84.56	76.54	10.64	17.80
Ash content	1.40-3.84	2.92-4.26	1.05	7.22	2.63
Fixed carbon	8.58-12.88	3.222-4.61	18.91	76.86	77.27
Ultimate (%)					
Carbon	40.39-42.85	41.26-42.47	45.83	69.85	70.83
Hydrogen	2.14-3.14	6.09-6.10	0.29	-	3.41
Oxygen	54.77-56.13	-	47.48	-	25.76
Nitrogen	0.24-0.25	0.95-1.03	0.40	0.23	-
Sulphur	0.08-0.09	-	-	1.71	-

Characteristics of activated carbons produced: Yield of activated carbon

In general, the bamboo-based ACs exhibit various physiochemical properties at various H_3PO_4 concentrations, including the product yield. Yield of bamboo AC is defined as the weight of carbonized bamboo after washing using hot water to remove H_3PO_4 residue or contaminants and oven dried. With the increase of H_3PO_4 concentration, the yield of beting and aur ACs were increased from 58.0% to 62.0% and 58.1% to 60.1%, respectively (Figure 2). According to Ismail et al. [23], H_3PO_4 impregnation acts as a reactant to create crosslinks and as an acid catalyst to enhance the bond breaking of the carbon precursors during the activation. Phosphate and polyphosphate species are created because of the crosslinking of the H_3PO_4 and organic components in the biomass sample, and they may persist on the AC structure even after washing. Hence, more H_3PO_4 usage led to a larger AC yield and increment of leftover solid biomass by reducing tar formation and the emission of volatile compounds during the carbonization reaction [23]. The yield of both bamboo ACs obtained was higher than that of the ACs reported from other biomasses, such as bamboo (36.8%–40.2%), coconut shell (50.84%), and durian shell (45.5%) [23-25]. This shows that bamboo is one of the potential carbon precursors to produce AC.

Surface characteristics of porous activated carbon

One of the methods to determine the surface functionalities of a material and performing qualitative analysis is FTIR spectroscopy. Therefore, by employing the FTIR spectral analysis at the mid-infrared range (4000–450 cm^{-1}), the functional groups that are present in raw bamboo and manufactured ACs can be determined. Figure 3(a) and (b) shows the results of spectrum for raw bamboo and ACs, respectively. Figure 3(a) shows that the bamboo precursor created more bands than the manufactured ACs. Beting and aur raw bamboos show a broad absorption band at 3340 to 3348 cm^{-1} due to the O–H stretching and H-bonded bond structure of the main functional groups of phenols, alcohols, and waters. The C–H stretching and O–H stretching bond structure, which contains a functional group of alkanes (cellulose and lignin) and carboxylic acids, was attributed to a small peak at 2881 and 2889 cm^{-1}. The minor peak in the C–H stretching bond structure can also comprise methyl (CH_3), methylene (CH_2), and aliphatic saturated functional groups (CH) [20]. The C=C stretching bond structure from the functional group of alkenes (lignin) is best described by the small peak at 1605 cm^{-1}, whereas the peak at 1463 cm^{-1} defined the C–H bending bond from the functional group of alkanes (cellulose, hemicellulose, and lignin). The peak marked at 1050 cm^{-1} indicates the C–O stretching bond structure from the functional group of alcohol (cellulose, hemicellulose, and lignin), carboxylic acids, and esters. The C–O stretching bond structure of the functional group of glycosides linkage is likewise recognized by the peaks band around 1100 to 1000 cm^{-1} [26].

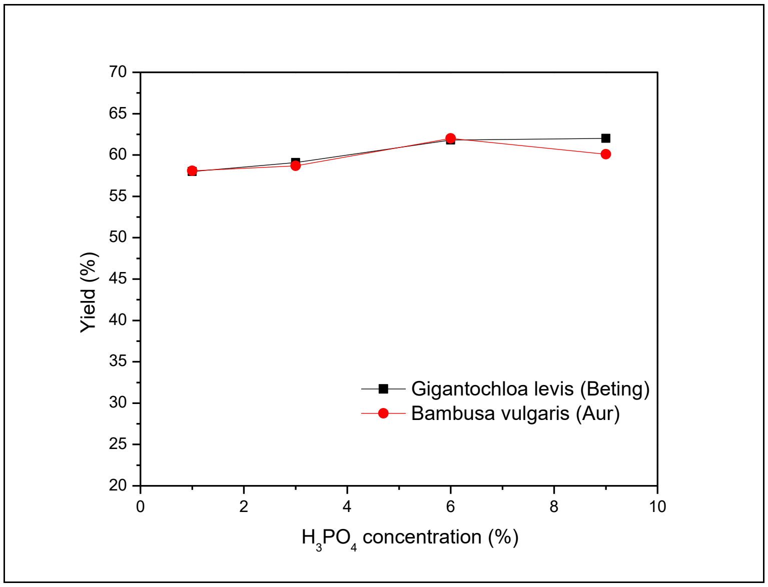

Figure 2. Effect of H_3PO_4 concentration on the yield of bamboo AC

The AC has less absorption peaks than the raw bamboo depending on the H_3PO_4 concentration used. It suggests that H_3PO_4 concentrations had less impact on the surface functionalities, while majority of the organic matter from raw bamboo was removed during the activation process [23]. Figure 3(b) reflects the results where the band at 3700 cm^{-1} is responsible for hydroxyl bonding group O–H with H-bonded vibration. The absorption band at 3340 to 3348 cm^{-1} disappeared due to the formation of polyphosphate and inorganic species in the AC [27]. Meanwhile, the stretching of C=O bands were attributed to the aromatic structures around 1540 cm^{-1}. The peak at 1210 cm^{-1} might be attributed to P=OOH, O–C stretching vibrations in the P–O–C linkage, and hydrogen-bonded P=O stretching mode. The peak at 1000 cm^{-1} can be caused by the stretching vibrations in P–O–P polyphosphate chains, and P^+–C^- in acid phosphate esters. Aromatic structures are responsible for the weaker bonds around 560 cm^{-1}. These results are in a good agreement with the study by Yorgun et al. [21]. Basically, during the H_3PO_4 chemical activation process, four main reactions will occur which are dehydration, depolymerization, the formation of aromatic ring, and the elimination of phosphate group [28]. H_3PO_4 acts as a dehydrating catalyst during acid hydrolysis to promote decomposition and depolymerization of the biopolymers (cellulose, hemicellulose, lignin). The increasing concentration of H_3PO_4 will enhance its ability as the dehydrating catalyst that removes excess water and converts the methyl group in the bamboo to AC with rich-pore structure [12, 29].

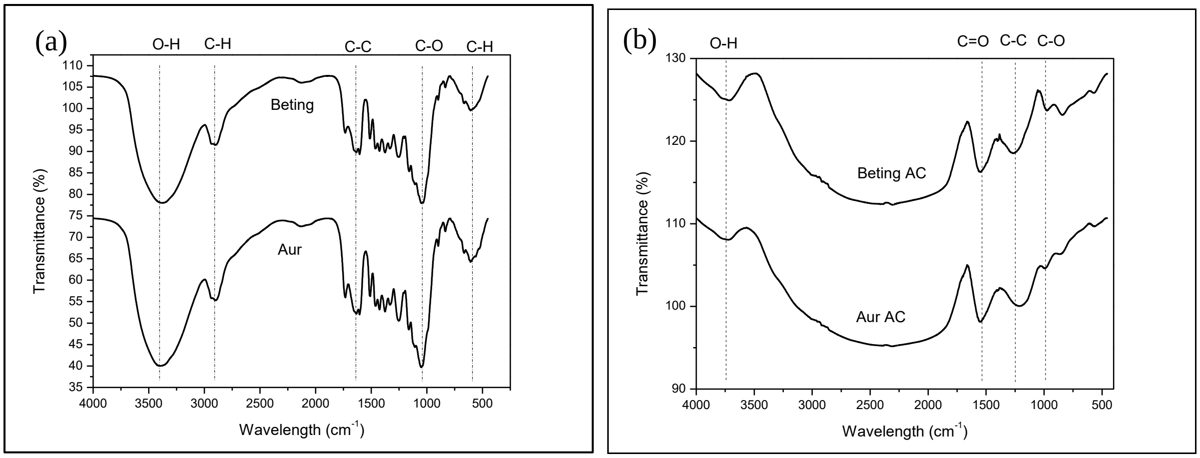

Figure 3. FTIR spectra of bamboo (a) raw precursor (b) porous activated carbon

Surface area and porous structure

Surface area and porous structure (pore size and pore volume) of bamboo AC is important to note to determine its end application. The effect of H_3PO_4 on the surface area and porous structure of bamboo AC was investigated using N_2 adsorption-desorption spectroscopy at 77 K. The effects of carbon precursor (bamboo species) on the surface area and porous structure of bamboo AC were also studied. Figure 4(a) and (b) summarizes the surface area and porous structure of ACs. The figure shows that the porous structure (pore size and pore volume) of bamboo AC increased with increasing H_3PO_4 concentration. These results indicate that high concentration H_3PO_4 impregnation benefitted the formation of pores [4, 30].

Furthermore, the surface area of bamboo AC increased with the increasing of H_3PO_4 concentration. The effect of an increased H_3PO_4 concentration on the BET surface area and pore volume was stronger at 9% v/v H_3PO_4. At this concentration, the maximum surface area of 1319 and 1285 m^2/g, total pore volume of 0.68 and 0.73 cm^3/g, and pore size of 3.6 nm were obtained in beting and aur ACs, respectively, which are comparable with that of the commercial AC (500–1500 m^2/g) [31]. The lowest H_3PO_4 concentration (1% v/v) shows the lower surface area and total pore volume of 368–381 m^2/g and 0.17 cm^3/g, respectively. The surface area and total pore volume can also be compared with the raw material of bamboo. There is no formation or presence of pores and also any increment of pore volume in the raw bamboo. The surface area and pore volume were only 0.437 m^2/g and 0.000826 cm^3/g, respectively. The effect of H_3PO_4 concentration on the surface area and total pore volume of the AC is shown in Figure 4(a) and (b). The process of thermal degradation and volatilization accelerates further when carbonization is applied to an impregnated bamboo, resulting pores forming and surface area increasing [21]. It is believed that the porosity of AC was mainly caused by the H_3PO_4 remaining in the impregnated material. The H_3PO_4 introduced into the interior of the bamboo particle during impregnation can inhibit the contraction of the structure during carbonization, thus producing a porous structure when it is extracted by washing after carbonization. The H_3PO_4 can accelerate the hydrolysis of lignocellulosic material, thus it promotes the formation bond cleavage in the biopolymer structure of bamboo. This can weaken the bonding within the structure and favouring the swelling of the biomaterial, which promotes the formation of pores and the increment of pore volume. As the H_3PO_4 dosage increases, it is reasonable to expect more potential sites could be penetrated and occupied by H_3PO_4, which enhances the pore-opening and widening processes. Besides that, during the carbonization at 500 °C using the tubular furnace, H_3PO_4 exhibits the formation of volatile cellulose products which causes the bamboo biopolymer structure begins to dilate and cross-link between the biopolymer chains through the ester linkages formation in the H_3PO_4 and OH groups. The similar findings were also reported by Feng et al. [4], Tumirah et al. [12], and Jagtoyen & Derbyshire [29].

The production of ACs with high surface areas from a low cost and renewable material is indeed of importance from the viewpoint of economic and environmental aspects. High surface area of bamboo AC is believed to provide large space to trap chemical contaminants if it is used as an absorbance. Table 4 summarizes the surface area and porous structure of bamboo ACs prepared using the H_3PO_4 chemical activation method.

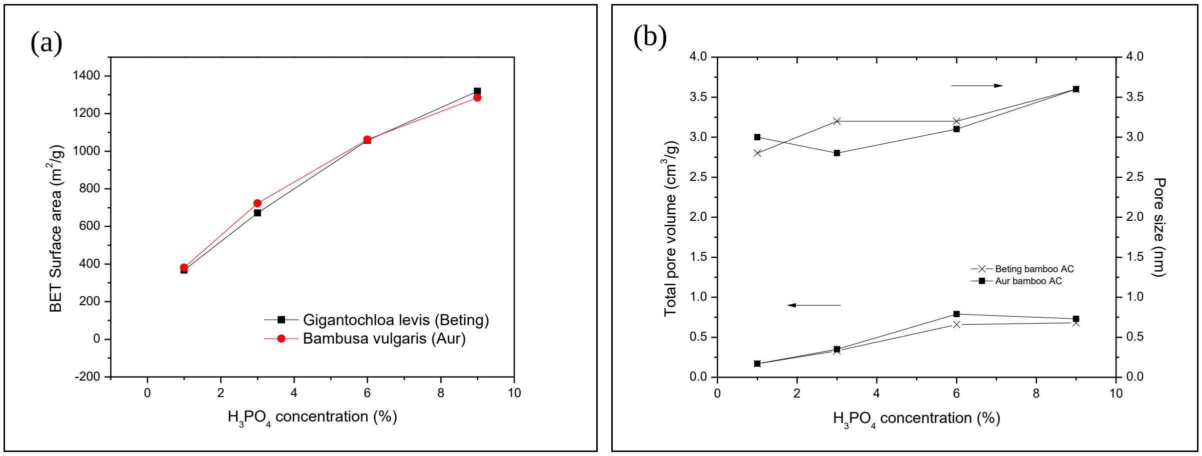

Figure 4. Effect of H_3PO_4 concentration on (a) the BET surface areas, (b) the total pore volume and pore size of activated carbons

Surface morphology of bamboo raw material and activated carbon

The FESEM analysis at the magnification scale of 5000× were carried out to confirm the textural properties and differences in the surface morphology of bamboo raw material and ACs (Figure 5). According to Figure 5(a) and (c), the micrographs of bamboo show no evidence of pore presence on the surface. The surface textures were rough, uneven, and undulating, due to the condensable volatile compounds that have gathered on the carbon surface. During activation and carbonization, most volatile matter was released thus showing surface changes of pores opening in bamboo ACs as shown in Figure 5(b) and (d). The AC's surface also generated well-developed pores because of the H_3PO_4 concentration [2]. The differences between these two carbonaceous materials' exterior morphologies can be related to the activation of H_3PO_4, which primarily involves the production of volatile chemicals and the interaction between the activating agent and the carbon precursor. The reaction between the carbon and the activating agent causes pore volume to increase as well as the formation of new pores during the chemical activation process. Similar trend was also found by Ismail and Yorgun et al [21,23]. Therefore, H_3PO_4 is a successful activation agent for producing ACs with large surface area.

Potential application of bamboo activated carbon

The performance of AC is largely depending on its surface area, porous structure, and chemical surface characteristics [32-34]. AC was used as an absorbance material because it has a massive surface area and well-developed pore structure (Table 3). These properties enable AC to have an excellent adsorption ability. Therefore, AC is widely used as a component in the water filtration system to remove contaminants. However, the adsorption effectiveness of AC is depending on the pores and pore size distribution relative to the size of adsorbent (organic matter) [35]. For example, the natural organic matter adsorption occurs in micropores and mesopores of AC [36]. However, AC with macropores is needed to filter bacteria [37]. Wang et al. [38] reported that the most suitable pore size range of AC for microbial attachment is between 2 and 5 µm. Therefore, the surface area and porous structure of bamboo AC obtained in this study indicates that it has the potential to be used as an absorbance material.

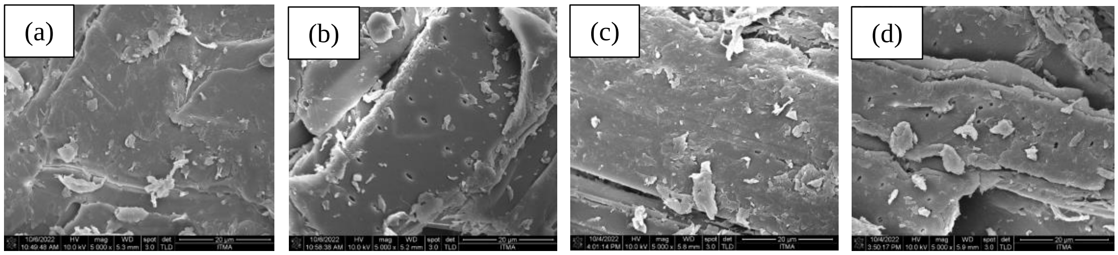

Figure 5. FESEM micrograph of (a) beting bamboo raw (b) beting bamboo activated carbon at 9% v/v H_3PO_4 impregnation (c) aur bamboo raw (d) aur bamboo activated carbon at 9% v/v H_3PO_4 impregnation

Table 3. Application of activated carbon prepared using phosphoric acid chemical activation method of varies carbon precursor as an absorbance material

Carbon Precursor	BET Surface Area (m^2/g)	Pore Size	Adsorbents	Findings	References
Mango kernel	490.43	38.9 µm	Cr (VI)	The maximum adsorption capacity of Cr (IV) was 7.8 mg/g at pH 2, T: 35 ºC	[39]
Tea waste	824	<50 nm	Oxytetracycline	The maximum adsorption capacity of oxytetracycline was 175 mg/g at T: 20 ºC	[40]
Coconut shell	2648	1-7 nm	benzene	Excellence benzene adsorption performance	[41]
Coconut front	483	29.72 Å	Carbofuran insecticide	Adsorption capacity was found to be 198.4 mg/g at T: 30 ºC	[42]
Banana tree pseudo-stem fiber	1975	-	Methylene blue dye	Adsorption capacity of 728 mg/g at T: 30 ºC	[43]
Typha orientalis leaves	1238	-	Pb(II)	The maximum adsorption capacity was found to be 7.95 mg/g	[44]
Bamboo	2237	Micropores	Ciprofloxacin	Adsorption amounts reach 613 mg/g	[45]
Coconut shell	89.09	-	Methylene blue	Adsorption amount 14.35-16.92 mg/g	[46]

Conclusion

In this work, the Sarawak wild bamboo, *Gigantochloa levis* (beting) and *Bambusa vulgaris* (aur) was used as a carbon precursor to produce ACs through the H_3PO_4 chemical activation method. The effect of H_3PO_4 concentration on the surface area, porous structure, and yield of AC was studied. The concentration of H_3PO_4 greatly influences the surface area, porous structure, and yield of AC. The maximum surface area of 1319 and 1285 m^2/g were obtained at 9% v/v H_3PO_4 impregnation of beting and aur bamboos, respectively. Furthermore, the maximum AC yield of 62% was obtained at 9% H_3PO_4 impregnation of beting bamboo and 6% H_3PO_4 impregnation of aur bamboo. The results show that bamboo is proven to be an effective precursor and has the potential to be a promising precursor to produce ACs. The large surface area AC that was produced can be used as an extremely promising adsorbent for various environmental applications. Low cost ACs could be produced by using cheap raw materials and an appropriate production technique. Cost is the key factor in determining the potential of an adsorbent in the industry. To produce low-cost ACs, it is necessary to look at novel source materials. To our knowledge, no research has been done on using bamboo as a raw material to prepare ACs using the low concentration of H_3PO_4 chemical activation. This is to supplement the information on the properties of AC which is particularly lacking.

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References

1. Negara, D. N. K. P., Nindhia, T. G. T., Surata, I. W., Sucipta, M. and Hidajat, F. (2019). Activated carbon characteristics of tabah bamboo that physically activated under different activation time. *IOP Conference Series: Materials Science and Engineering,* 539(1): 012011.
2. Mohd Iqbaldin, M. N., Khudzir, I., Mohd Azlan M. I., Zaidi, A.G., Surani, B. and Zubri, Z. (2013). Properties of coconut shell activated carbon. *Journal of Tropical Forest Science,* 25(4): 497-503.
3. Bagheri, N. and Abedi, J. (2009). Preparation of high surface area activated carbon from corn by chemical activation using potassium hydroxide. *Chemical Engineering Research and Design*, 87(8): 1059-1064.
4. Feng, J., Zhu, H., Xu, Y., Jiang, J. and Pan, H. (2021). Preparation and characterization of high-performance activated carbon from papermaking black-liquor at low temperature. *Journal of Analytical and Applied Pyrolysis*, 159: 105292.
5. Mahanim, S. M. A., Wan Asma, I., Rafidah, J., Puad, E. and Shaharuddin, H. (2011). Production of activated carbon from industrial bamboo wastes. *Journal of Tropical Forest Science,* 23 (4): 417-424.
6. Huang, Y. P., Hou, C. H., His, H. C. and Wu, J. W. (2015). Optimization of highly microporous activated carbon preparation from Moso bamboo using central composite design approach. *Journal of Taiwan Institute of Chemical Engineers,* 50: 266-275.
7. Huang, G. Wang, Y., Zhang, T., Wu, X. and Cai, J. (2019). High-performance hierarchical N-doped porous carbons from hydrothermally carbonized bamboo shoot shells for symmetric supercapacitors. *Journal of Taiwan Institute of Chemical Engineers,* 96: 672-680.
8. Negara, D. N. K. P., Nindhia, T. G. T., Surata, I. W., Hidajat, F. and Sucipta, M. (2020). Textural characteristics of activated carbons derived from Tabah bamboo manufactured by using H_3PO_4 chemical activation. *Materials Today: Proceedings,* 22: 148-155.
9. Tay, T., Ucar, S. and Karagöz, S. (2009). Preparation and characterization of activated carbon from waste biomass. *Journal of Hazardous Materials,* 165(1): 481-485.
10. Li, W., Yang, K., Peng, J., Zhang, L., Guo, S. and Xia, H. (2008). Effects of carbonization temperatures on characteristics of porosity in coconut shell chars and activated carbons derived from carbonized coconut shell chars. *Industrial crops and products*, 28(2):190-198.
11. Supriya, S., Palanisamy, P. N. and Shanthi, P. (2014). Preparation and characterization of activated carbon from casuarina for the removal of dyes from textile wastewater. *International Journal of ChemTech Research,* 6 (7): 3635-3641.
12. Tumirah, K., Mohd Zobir, H., Zulkarnain, Z. and Rafaedah, R. (2015). Textural and chemical properties of activated prepared from tropical peat soil by chemical activation method. *BioResources,* 10 (1): 986-1007.

13. Wise, L. E., Murphy, M. and D' Addieco, A.A. (1946). Chlorite holocellulose: Its fractiontion and bearing on summative wood analysis and on studies on the hemicelluloses. *Paper Trade Journal* 122 (2): 35-43.
14. TAPPI. 203 om-93. Alpha, Beta and Gamma Cellulose in Pulp. (2009). Technical Association of the pulp and industry, Atlanta.
15. TAPPI. T222 om-02Acid-Insoluble Lignin in Wood and Pulp. (2002). Technical Association of the pulp and industry, Atlanta.
16. Liu, Q-S., Zheng, T., Wang, P. and Guo, L. (2010). Preparation and characterization of activated carbon from bamboo by microwave-induced phosphoric acid activation. *Industrial Crops and Products,* 31(2): 233-238.
17. Negara, D.P., Nindhia, T.T., Surata, I.W. and Sucipta, M. (2017). Chemical, strength and microstructure characterization of Balinese bamboos as activated carbon source for adsorbed natural gas application. *IOP Conference Series: Materials Science and Engineering.* 201(1): 012033.
18. Kasim, J. Ahmad, A. J., Mohamod A. L. and Khoo, K. C. (1994). A note on the proximate chemical composition and fibre morphology of bambusa vulgaris. *Journal of Tropical Forest Science,* 6 (3): 356-358.
19. Azeez, M. A. and Orege, J. I. (2018). Chapter 3: Bamboo, its chemical modification, and products. In H. P. S. Abdul Khalil (Ed.), *Bamboo*. IntechOpen. http://dx.doi.org/10.5772/intechopen.76359: 25-48.
20. AssefaAragaw, T. (2016). Proximate analysis of cane bagasse and synthesizing activated carbon: Emphasis on material balance. *Journal of Environmental Treatment Techniques,* 4(4): 102-110.
21. Yorgun, S. and Yıldız, D. (2015). Preparation and characterization of activated carbons from Paulownia wood by chemical activation with H_3PO_4. *Journal of the Taiwan Institute of Chemical Engineers*, 53: 122-131.
22. Shamsuddin, M. S., Yusoff, N. R. N. and Sulaiman M. A. (2016). Synthesis and characterization of activated carbon produced from kenaf core fiber using H_3PO_4 activation. *Procedia Chemistry,* 19: 558-565.
23. Ismail, I.S., Rashidi, N.A. and Yusup, S. (2022). Production and characterization of bamboo-based activated carbon through single-step H_3PO_4 activation for CO_2 capture. *Environmental Science and Pollution Research*, 29(9): 12434-12440.
24. Chandra, T. C., Mirna, M. M., Sunarso, J., Sudaryanto, Y. and Ismadji, S. (2009). Activated carbon from durian shell: Preparation and characterization. *Journal of the Taiwan Institute of Chemical Engineers,* 40(4): 457-462.
25. Liang, Q., Liu, Y., Chen, M., Ma, L., Yang, B., Li, L. and Liu, Q. (2020). Optimized preparation of activated carbon from coconut shell and municipal sludge. *Materials Chemistry and Physics,* 241: 122327.
26. Bakri, M. K. and Jayamani, E. (2016). Comparative study of functional groups in natural fibers: Fourier transform infrared analysis (FTIR). *Futuristic Trends in Engineering, Science, Humanities, and Technology,* 1: 167-174.
27. Hesas, R. H., Wan Daud, W. M. A., Sahu, J. N. and Arami-Niya, A. (2013). The effects of a microwave heating method on the production of activated carbon from agricultural waste: A review. *Journal of Analytical and Applied Pyrolysis,* 100: 1-11.
28. Suárez-García, F., Martínez-Alonso, A. and Tascón, J. M. D. (2004). Activated carbon fibers from Nomex by chemical activation with phosphoric acid. *Carbon,* 42(8-9): 1419-1426.
29. Jagtoyen, M. and Derbyshire, F. (1993). Some considerations of the origins of porosity in carbons from chemically activated wood. *Carbon,* 31(7): 1185-1192.
30. Wang, Y. X., Ngo, H. H. and Guo, W. S. (2015). Preparation of a specific bamboo based activated carbon and its application for ciprofloxacin removal. *Science of The Total Environment,* 533: 32-39.
31. Nachenius, R. W., Ronsse, F., Venderbosch, R. H., and Prins, W. (2013). Chapter two - biomass pyrolysis. In D. Y. Murzin (Ed.), *Advances in Chemical Engineering*, (Vol. 42, pp. 75–139). Academic Press Inc.
32. Karanfil, T., Kitis, M., Kilduff, J. E. and Wigton, A.

(1999). Role of granular activated carbon surface chemistry on the adsorption of organic compounds. 2. Natural organic matter. *Environmental Science Technology,* 33(18): 3225-3233.

33. Moore, B. C., Cannon, F. S., Westrick, J. A., Metz, D. H., Shrive, C. A., DeMarco, J. and Hartman, D. J. (2001). Changes in GAC pore structure during full-scale water treatment at Cincinnati: A comparison between virgin and thermally reactivated GAC. *Carbon,* 39(6): 789-807.
34. Lu, Z., Sun, W., Li, C., Cao, W. Jing, Z., Li, S., Ao, X., Chen, C. and Liu, S. (2020). Effect of granular activated carbon pore-size distribution on biological activated carbon filter performance. *Water Research,* 177: 115768.
35. Nowotny, N., Epp, B., von Sonntag, C. and Fahlenkamp, H. (2007). Quantification and modeling of the elimination behavior of ecologically problematic wastewater micropollutants by adsorption on powdered and granulated activated carbon. *Environmental Science & Technology,* 41(6): 2050-2055.
36. Hsieh, C-T. and Teng, H. (2000). Influence of mesopore volume and adsorbate size on adsorption capacities of activated carbons in aqueous solutions. *Carbon,* 38(6): 863-869.
37. Walker, G. M. and Weatherley, L. R. (1998). Bacterial regeneration in biological activated carbon systems. *Process Safety and Environmental Protection,* 76(2): 177-182.
38. Wang, J. Z., Summers, R. S. and Miltner, R. J. (1995). Biofiltration performance: Part 1, relationship to biomass. *Journal American Water Works Association, 87*: 55-63.
39. Rai, M.K., Shahi, G., Meena, V., Meena, R., Chakraborty, S., Singh, R.S. and Rai, B.N. (2016). Removal of hexavalent chromium Cr(VI) using activated carbon prepared from mango kernel activated with H_3PO_4. *Resource-efficient technologies*, 2: S63-S70.
40. Kan, Y., Yue, Q., Li, D., Wu, Y. and Gao, B. (2017). Preparation and characterization of activated carbons from waste tea by H_3PO_4 activation in different atmospheres for oxytetracycline removal. *Journal of the Taiwan Institute of Chemical Engineers*, 71: 494-500.
41. Zhang, Z., Lei, Y., Li, D., Zhao, J., Wang, Y., Zhou, G., Yan, C. and He, Q. (2020). Sudden heating of H_3PO_4-loaded coconut shell in CO_2 flow to produce super activated carbon and its application for benzene adsorption. *Renewable Energy*, 153: 1091-1099.
42. Njoku, V.O., Islam, M.A., Asif, M. and Hameed, B.H. (2014). Preparation of mesoporous activated carbon from coconut frond for the adsorption of carbofuran insecticide. *Journal of Analytical and Applied Pyrolysis*, 110: 172-180.
43. Silva, M. C., Spessato, L., Silva, T. L., Lopes, G. K., Zanella, H. G., Yokoyama, J. T., Cazetta, A. L. and Almeida, V. C. (2021). H_3PO_4–activated carbon fibers of high surface area from banana tree pseudo-stem fibers: adsorption studies of methylene blue dye in batch and fixed bed systems. *Journal of Molecular Liquids*, 324: 114771.
44. Anisuzzaman, S. M., Joseph, C.G., Daud, W. M. A. B. W., Krishnaiah, D. and Yee, H. S. (2015). Preparation and characterization of activated carbon from *Typha orientalis* leaves. *International Journal of Industrial Chemistry*, 6: 9-21.
45. Wang, Y. X., Ngo, H. H. and Guo, W. S. (2015). Preparation of a specific bamboo based activated carbon and its application for ciprofloxacin removal. *Science of the Total Environment*, 533: 32-39.
46. Lee, C. L., Chin, K. L., H'ng, P. S., Rashid, U., Maminski, M. and Khoo, P. S. (2021). Effect of pretreatment conditions on the chemical–structural characteristics of coconut and palm kernel shell: A potentially valuable precursor for eco-efficient activated carbon production. *Environmental Technology & Innovation*, 21: 101309.