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Original Article

Microstructural characterization of white charcoal for rapid reduction of chemical oxygen demand and automatically adjust pH to neutral in wastewater treatment

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ABSTRACT

The purpose of this study was to produce, characterize and apply white and black charcoal for wastewater treatment application. Characterization of charcoals, the results confirmed that white charcoal had higher thermal stability and carbon content than black charcoal. The other elemental analysis results showed that both black and white charcoal show the highest intensity of potassium content. The surface of black charcoal was rougher than the surface of white charcoal. The morphology from scanning electron microscope coupled with Energy Dispersive X-ray Spectrophotometer (SEM-EDS) images demonstrated white charcoal shows a highly porous material, especially coconut shell white charcoal with small holes connected to large holes. X-ray diffraction patterns (XRD) showed peak patterns of graphite. The specific surface area, total pore volume, and pore size of all charcoal were evaluated by the BET method. This result was a higher specific surface area of white charcoal than black charcoal. The coconut shell white charcoal had the highest specific surface area (458.80 m 2/g). All charcoals had an average pore size in the range from 1 to 2 nm, all charcoals had an average pore size in the range from 1 to 2 nm which was microporous materials. Both black and white charcoal were used as an adsorbent for the wastewater treatment from a chemistry laboratory building. The results indicated that white charcoal could neutralize the pH of wastewater after treatment in both acidity and alkalinity wastewater. After treatment, wastewater treated with white charcoal shows a higher level of %COD reduction than wastewater treated with black charcoal.

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1. Introduction

There are many kinds of waste wood in Thailand because it is one of the agricultural countries. The waste woods occurred from many activities. Zero waste is a necessary method of important role in environmental management [1]. It is better than this if using these waste woods for environmental pollution treatment such as air and wastewater pollution, because air quality has directly affected the respiratory system of humans [2] and wastewater pollution is an urgent need for treatment. Adding the value of waste is a global priority, not only adding value to waste, but can also be used to solve environmental problems [3]. These waste woods are now often converted to charcoal.

Currently, there are three kinds of charcoals include the ordinary "black charcoal" (BC), "activated charcoal" (AC), and "white charcoal" (WC). These charcoals are from preparation using different technical methods of burning [4,5]. The quality of the charcoal differs significantly depending on how the fire is extinguished. Many researchers reported the adsorption of a variety of gases such as ethylene [6], hydrogen [7], and some work studied the adsorption of acetic acid vapor [8]. There are many applications of charcoal. For example, BC has used for food preparation. However, it generates a large amount of smoke when used for grilling [9]. Nowadays, the preparation process of smoke-free fuel is an important role in food preparation from coal for good health. BC is used as an absorber in washing or cleaning cosmetics [10]. AC is used for radio frequency (RF) shielding [11], electromagnetic wave absorber [12,13], odor absorber [14], and WC is used in water treatment [15]. This technology is applied mostly in Japan [16]. Each type of charcoal differs at the temperature used for firing. BC, AC, and WC is burned at temperatures <400 °C, 400–800 °C, and >1000 °C, respectively [17–19]. These charcoals have been applied in many different fields [20,21]. Although white charcoal has many benefits, characterization method, the microstructural study of Thailand white charcoal, and its applications for wastewater treatment have rarely been reported.

Therefore, the objectives of this study are to prepare, characterize microstructure of black and white charcoal, and apply for wastewater treatment. Black charcoal is produced from bamboo, BBC) and white charcoals are prepared from four kinds of waste wood. Further could be used in pharmaceutical and cosmetic applications, for example as a toxic adsorbent in disinfectants and as a scrub in the cleansing gel.

2. Materials and method

2.1. Materials

Waste of Bamboo, miscellaneous, coconut shell, and coconut spathe was collected from basketwork, construction building site, market after the food production process, and coconut farm, respectively from Ubon Ratchathani province, The North East of Thailand.

2.2. Preparation of charcoal

Bamboo black charcoal was prepared by burning at temperature 300–400 °C in a furnace for 3 h. On the other hand, white charcoal from each waste woods (bamboo, miscellaneous, coconut shell, and coconut spathe) was burned at a temperature ~ 1000 °C in a specifically designed oven. After pyrolysis, all charcoals were ground and characterized. In this study, there are five types of charcoal classified by the type of waste wood. The abbreviation name of them in specific code is the following; black bamboo charcoal (BBC), white bamboo charcoal (BWC), white miscellaneous charcoal (MWC), white coconut shell charcoal (SWC), and white coconut spathe charcoal (CWC).

2.3. Characterization of charcoal

The proximate analysis and ultimate analysis of all charcoals were studied. The elemental analysis were studied by Energy dispersive X-ray fluorescent (EDX) using EDX-LE, Shimadzu, Japan. The crystal structure was examined by X-ray diffractometer (XRD) using X'Pert MPD, Phillips using Cu Kα radiation with $\lambda = 0.15406$ nm, beam voltage 30 kV, beam current 15 mA in scan rate 2° min^{-1}, and the range 2θ around 20–80°. For each material, thermal stability and its fraction of volatile components were determined by Thermogravimetric analysis (TGA; Thermo plus TG8120, Rigaku, Japan) at a constant rate of 10 °C/min. Morphology of four white charcoals was observed using Scanning Electron Microscope (SEM; JSM 5410, JEOL, Japan) at 3.0 kV. The high resolution of morphologies and carbon content were studied using Field Emission Scanning Electron Microscope coupled with Energy Dispersive X-ray Spectrophotometer (FE-SEM-EDS; JSM-7610F Plus, JEOL, Japan). All charcoals were placed on each stub, dried in a vacuum chamber at room temperature, coated with a thin platinum layer before observing with FE-SEM-EDS. The specific surface area, total pore volume and total pore size were determined by surface area analyzer with the Brunauer Emmett-Teller method (BET; Quanta chrome Quadrawin-Data Acquisition and Reduction for Quadrasorb SI, USA). The degassing time was 7 h at 300 °C, then the nitrogen adsorption was carried out at −196 °C, which took 431.9 min to complete.

2.4. Application for wastewater treatment

Study the adsorption efficiency of white and black charcoal for wastewater treatment applications was studied and adjusted from Standard Methods for the Examination of Water and Wastewater (SMEWW) [22]. Wastewater treatment parameters were analyzed according to the following;

2.4.1. pH measurement

Both BBC and BWC were ground and sifted with sieve 50 mesh before testing. 4.00 g of each charcoal was added to 100 mL of each wastewater sample flask. The mixture was immediately stirred at room temperature (10 min) and allowed to adsorb (30 min) [21]. After finish the adsorption time, the sample was filtered immediately to remove charcoal and then measured

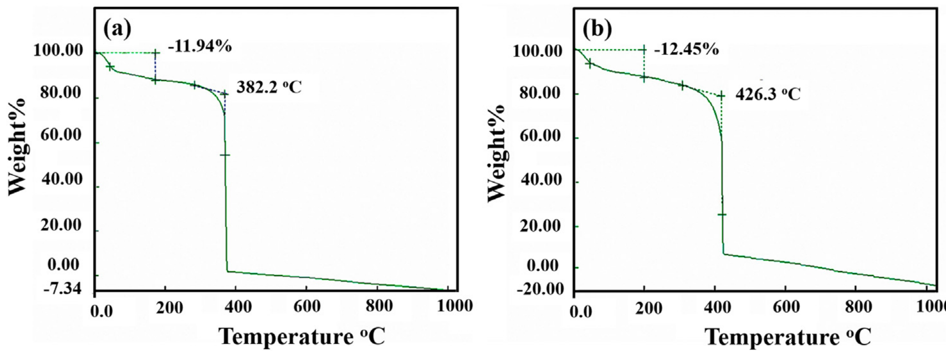

Fig. 1 – TGA curve of (a) BBC and (b) BWC.

the pH using a pH meter (Cyberscan pH 500, Eutech Instruments, Singapore).

2.4.2. *Chemical oxygen demand (COD)analysis*

The test tube and Erlenmeyer flask were dried at 105 °C for 1 h and cooled to room temperature. The blank solution was distilled water (7.5 mL). The water sample, the dilution ratio of the water sample: distilled water was 1:2. In the procedure, 2.5 mL water sample: 5 mL distilled water in a dried test tube. After that, potassium dichromate (1.5 mL) and H_2SO_4 3.5 mL were added to both the blank and water sample. The water sample (7.5 mL) from the last step was transferred into a dried Erlenmeyer flask. Ferro-indicator was added 2–3 drops into the water sample, which changed to the blue-green solution. This sample was slowly titrated drop by drop with 0.025 N of ferrous ammonium sulfate (FAS). The solution become to red (the endpoint color). Then the volume of FAS was recorded and calculated from the standard method [23].

3. Results and discussion

3.1. *Thermal stability of black and white charcoal*

The thermal stability of charcoals was observed by Thermal gravimetric analysis (TGA). Fig.1(a) shows TGA data profiles of black charcoal. There are three steps in weight loss. The first step occurs over the temperature range 80–100 °C involves water or moisture evaporation. The second was found over the temperature range 140–170 °C related to hemicellulose and pectin depolymerization [24]. The third step shows over the temperature range of 280–380 °C involves lignin degradation [25]. Fig. 1(b) shows three steps, removal of water or moisture and some volatile organic compounds (−12.45%) at temperature range ~80–200 °C and burn out at 426.3 °C. These results were caused by the white charcoal that burned at a higher temperature than black charcoal. This result disguised that higher thermal treatment temperature provides higher thermal stability [26].

3.2. *The proximate analysis and ultimate analysis*

There are two methods for analyzing the chemical structure of biomass including proximate analysis and ultimate analysis. First, proximate analysis of charcoals, parameters consist of ash, volatile matter, and fixed carbon. Second, the ultimate analysis was needy on quantitative analysis of various elements present in the charcoal sample, such as carbon, oxygen, and nitrogen. Table 1 showed the proximate analysis and ultimate analysis of all charcoals. The results clearly showed that the BBC had the highest percentage of ash (4.84%) and highest volatiles content (5.97%). However, the fixed carbon (89.19%) was lower than four white charcoals. Four white charcoals, BWC, MWC, SWC, and CWC had a high percentage of fixed carbon as 95.14%, 95.88%, 96.65%, and 95.61%, with a low percentage of ash as 2.26%, 1.55%, 0.91%, and 2.37%, respectively. Moreover, it shows low volatile matter content as 2.60%, 2.57%, 2.44%, and 2.02%, respectively. The low volatiles content of white charcoal indicates low molecular weight compounds and burns very cleanly [27]. This is generally related to previous studies of commercially available oak wood white charcoal (fixed carbon > 95% by mass) [28], and previous research reported that the fixed carbon of white charcoal was 95.1% [29]. In the ultimate analysis,

Table 1 – Proximate analysis and ultimate analysis of all charcoals.

Method	Amount (%)	BBC	BWC	MWC	SWC	CWC
Proximate analysis	% Ash	4.84	2.26	1.55	0.91	2.37
	% Volatile	5.97	2.60	2.57	2.44	2.02
	% Fixed carbon	89.19	95.14	95.88	96.65	95.61
	% Total	100	100	100	100	100
Ultimate analysis	% C	85.42	90.67	92.01	93.13	85.74
	% O	11.80	8.45	6.94	5.91	12.29
	% Other elements	2.78	0.88	1.05	0.96	1.97
	% Total	100	100	100	100	100

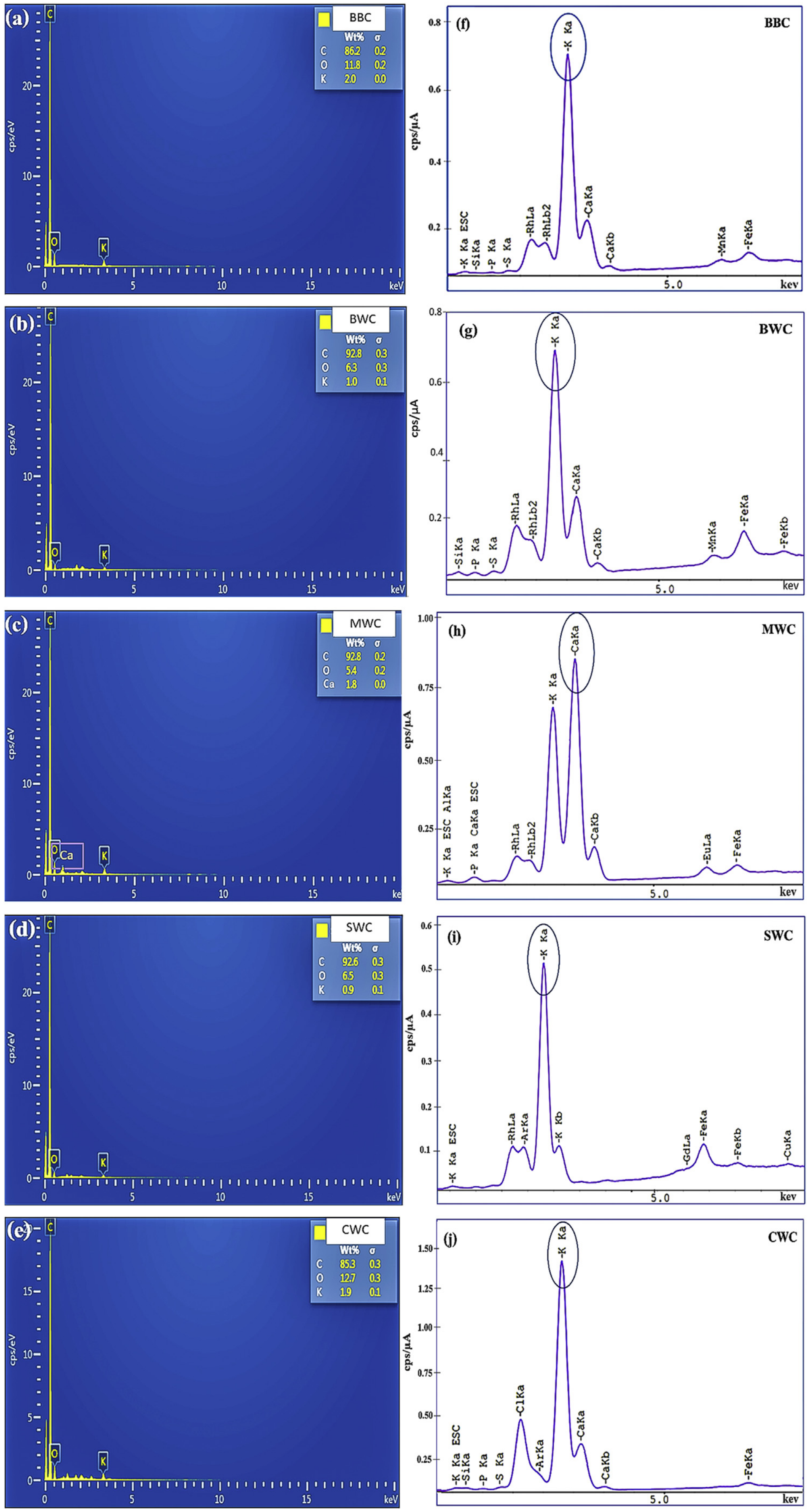

Fig. 2 – EDS spectrum: (a) BBC, (b) BWC, (c) MSC, (d) SWC, and (e) CWC, and ED-XRF spectrum: (f) BBC, (g) BWC, (h) MSC, (i) SWC, and (j) CWC.

the results show that white charcoal has higher carbon content than black charcoal, except for the CWC that has low carbon content. This result may be because the chemical composition in coconut spathe is lower than bamboo, miscellaneous, and coconut shell ones.

3.3. *Elemental analysis*

The EDS spectra displayed that the samples consisted of C, O, and K (or Ca for some sample), the compositions of all charcoals. The mass fractions of C, O, and K in BBC were 86.2%, 11.8%, and 2.0%, respectively (Fig. 2(a)), BWC were 92.8%, 6.3%, and 1.0%, respectively (Fig. 2(b)), SWC were 92.6%, 6.5%, and 0.9%, respectively (Fig. 2(d)), CWC were 85.3%, 12.7%, and 1.9%, respectively (Fig. 2(e)). The mass fractions of C, O, and Ca in MWC were 92.8%, 5.4%, and 1.8%, respectively (Fig. 2(c)).

3.4. *Another abundant element*

Another abundant elemental analysis of black and four white charcoals were studied by Energy dispersive X-ray fluorescent (ED-XRF). This technique could not detect light elements such as carbon and oxygen because of the detector limitation. Although carbon is the main element in charcoal, the ED-XRF results also detected the other elements. The spectra of elements in black charcoal and white charcoal are shown in Fig. 2(f)–(j). The spectrum showed that the BBC and BWC contained a high content of K and followed by Ca. The metal in charcoal has the following order; K > Ca > Fe > Mg. This result has corresponded to a previous report [30]. The results confirmed that white charcoal contained K as the main component, except in MWC (Ca in high content). The spectrum showed that the BWC (Fig. 2(f)), SWC (Fig. 2(h)), and CWC (Fig. 2(j)) had highest potassium intensity in the alpha of K-shell (K-Kα) peak. As opposed to MWC (Fig. 2(g)) had the highest intensity of calcium in the alpha of K-shell (Ca-Kα) peak. These results were consistent with the results of another abundant cation from ED-XRF. The corresponding ED-XRF and SEM-EDS spectrum provided the highest of the potassium of alpha in the K-shell (K Kα) spectrum in all charcoal except MWC that had calcium of alpha in the K-shell (Ca Kα) in the highest content similar to some previous report [31].

Table 2 – Others abundant elemental composition of black and white charcoals from ED-XRF.

Element	BBC	BWC	MWC	SWC	CWC
K	78.57	62.66	32.67	76.51	69.64
Ca	13.21	18.20	59.78	13.79	5.57
Fe	2.35	4.55	1.19	5.48	0.61
Si	1.91	9.10	–	–	2.73
Mn	1.53	1.29	1.58	0.66	–
P	0.79	2.35	1.63	–	0.49
Cu	0.61	0.72	0.31	1.30	0.14
S	0.41	0.62	–	–	0.10
Zn	0.38	0.51	0.12	–	0.15
Rb	0.25	–	0.14	–	0.05
Sr	–	–	0.22	–	0.03
Tb	–	–	–	–	0.17
Al	–	–	2.35	–	–
Cl	–	–	–	–	20.24

The other elemental compositions in all charcoals from ED-XRF were summarized in Table 2. The results showed that all types of charcoal contained potassium content higher than the other. The results of this research are related to previous research [32]. Although miscellaneous white charcoal has the most calcium content, it has a large amount of potassium in the next order. The result showed that BBC, BWC, MWC, SWC, and CWC had potassium as 78.57%, 62.66%, 32.67%, 76.51%, and 69.64, respectively. From these experimental results, it can be seen that MWC had much less potassium than other products because it has higher calcium than potassium. However, the SEM-EDS technique is used for only surface analysis, not used for bulk analysis. This could explain the high standard deviation for elements present in trace amounts [33].

3.5. *Crystal structure of charcoals*

The crystal structure of black and four white charcoals were studied by X-ray diffraction (XRD). Fig. 3 shows the XRD patterns of all samples. The XRD peaks of white charcoals, the broader peaks at 25°, and 43° are consistent with the presence of amorphous carbon and/or the presence of nanoparticles or lower crystallinity structure than black charcoal. Peaks at both 2-theta were assigned as graphite (JCPDS number 08–0415) [34]. The XRD peaks of all charcoal indicated that peak at around 25° which can be assigned as the (0 0 0 2) plane of graphite, highest intensity in bamboo black charcoal. The smaller peaks at 43° assigned as the {1 0 1 0}/{1 0 1 1} planes of graphite were similar to the result in wood char [35]. However, the black charcoal not only shows that both peaks at 25° and 43°, but also shows the small peak around 27°. This peak is characteristic of the (1 0 4) plane of calcite [36], which maybe some part matter in wood still stable at a temperature lower than 600 °C, confirming the presence of degradation in the pyrolysis process at more than 1000 °C for production of white charcoal. Therefore, this peak was disappeared in all white charcoal diffraction patterns.

3.6. *Morphologies of white charcoal*

Fig. 4(a) shows the morphology of BBC in magnification 100× showing the porosity lower than BWC in the same

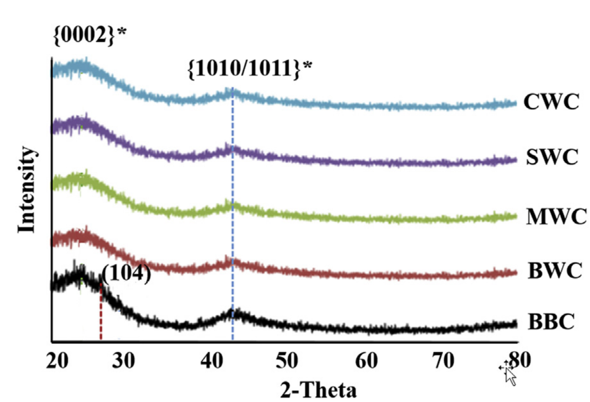

Fig. 3 – XRD patterns of all charcoals in this work.

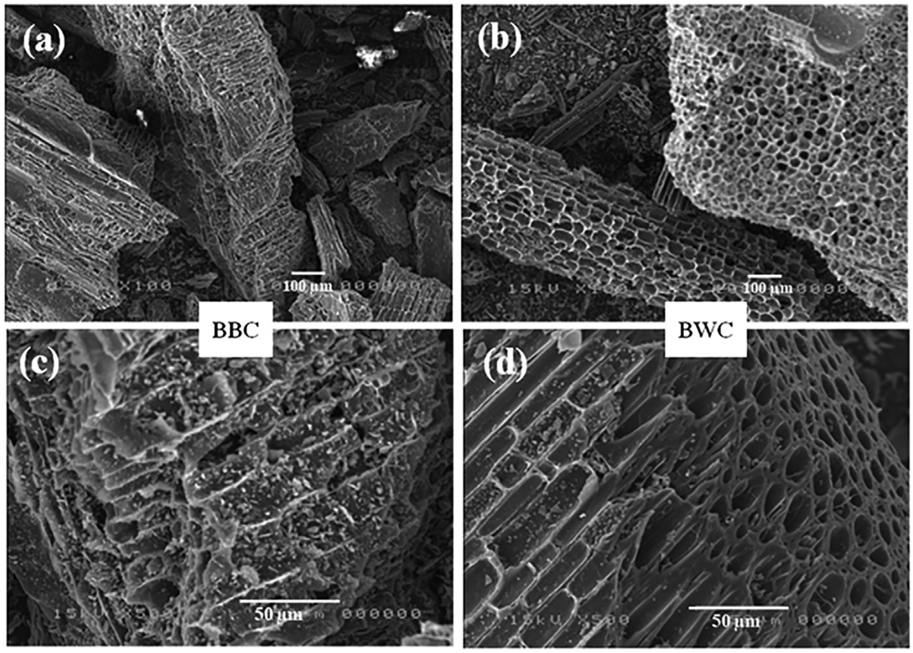

Fig. 4 – SEM of charcoals: (a) BBC in magnification of 100×, (b) BWC in magnification of 100×, (c) BBC in magnification of 500×, and (d) BWC in magnification of 500x.

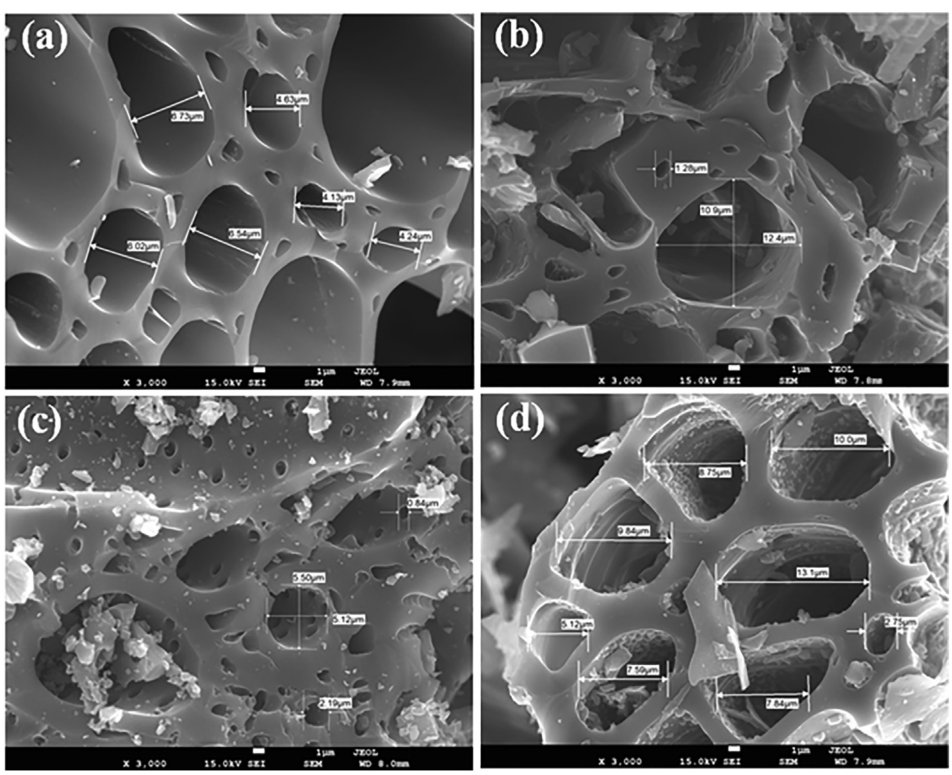

Fig. 5 – SEM of white charcoal in magnification 3000x: (a) BWC, (b) MWC, (c) SWC and (d) CWC.

Table 3 – Specific surface area, total pore volume, and total pore size of bamboo black charcoal and all of white charcoals.

Charcoal	Specific surface area (BET, m^2/g)	Total Pore volume (cm^3/g)	Total Pore size (nm)
BBC	27.81	0.0233	1.68
BWC	236.80	0.1351	1.14
MWC	270.10	0.1581	1.17
SWC	458.80	0.2623	1.10
CWC	24.52	0.0155	1.14

magnification in Fig. 4(b). Porous rigidity analysis of both charcoals, in Fig. 4(c), BBC morphology in magnification 500x consisted of more broken pieces. It was revealed that the porous structure of BBC was weaker than that of BWC in Fig. 4(d). It is clear that the porous structure of bamboo white charcoal is a rigid structure and stronger than that of bamboo black charcoal.

The morphology of all white charcoals in high resolution and magnification were shown in Fig. 5. Fig. 5(a) shows the porosity of bamboo white charcoal. The images show that the BWC had various sizes that smaller pores size than MWC in Fig. 5(b). Surprisingly, although the coconut shell before burning had hardly structure and smoothie surface, white charcoal from the coconut shell shows high porosity followed as Fig. 5(c). This result was related to the high surface area of SWC. The fact that it has a small pore in a large pore causes it to have a high surface area. However, the CWC in Fig. 5(d) shows a plate of particles that had low porosity which very different from the coconut spathe before calcining having more porosity and fiber.

3.7. *Specific surface area and porosity*

The volume of N_2 adsorption and relative pressure of all charcoals show pore-volume at STP of N_2 adsorption and relative pressure. The specific surface area, pore-volume, and pore sizes of the charcoal had changed as listed in Table 3. The result showed that the BWC had a higher specific surface area than that of BBC around 10-folds. This result implied that the BBC was unsuitable since it had a lower specific surface area and pore volume than white charcoal. Surprisingly, we found that the SWC shows a larger specific surface area than the others of 485.80 m^2/g, the smallest pore size of 1.10 nm, and the highest total pore volume of 0.2623 cm^3/g. The corresponding SEM image exhibited the hole in hole particles were shown in Fig. 6(c), proving that this morphology causes a high surface area. Therefore, several small holes connected to the large ones of SWC brings a higher surface area. The smaller pore size will encourage the adsorption process [37]. However, even though the CWC had a smaller pore size than the BBC, it shows a higher surface area because when considering the total pore volume, the BBC indicates a higher total pore volume than the CWC. Consequently, not only pore size can affect the specific surface area but the total pore volume also impact.

3.8. *Application for wastewater treatment*

3.8.1. *Effect on pH of water sample*

The water samples before and after treatment were measured pH with the pH meter three times. We found that the pH value of three times that an average pH was an acidic pH of 4.543 ± 0.002. After treatment with BBC (T-BBC), an average was 5.094 ± 0.002, slightly higher than before the treatment sample. Interestingly, after the treatment with white charcoal (T-BWC, T-MWC, T-SWC, and T-CWC), an average pH value for three times was 6.998 ± 0.002 which closely neutral. The results reveal that all of the white charcoal can change the acid water turn into neutral water. To improve the adsorption of charcoal an alkaline wastewater was studied. The result shows that white charcoal can neutralize the pH of wastewater from alkaline to be better than black charcoal. This

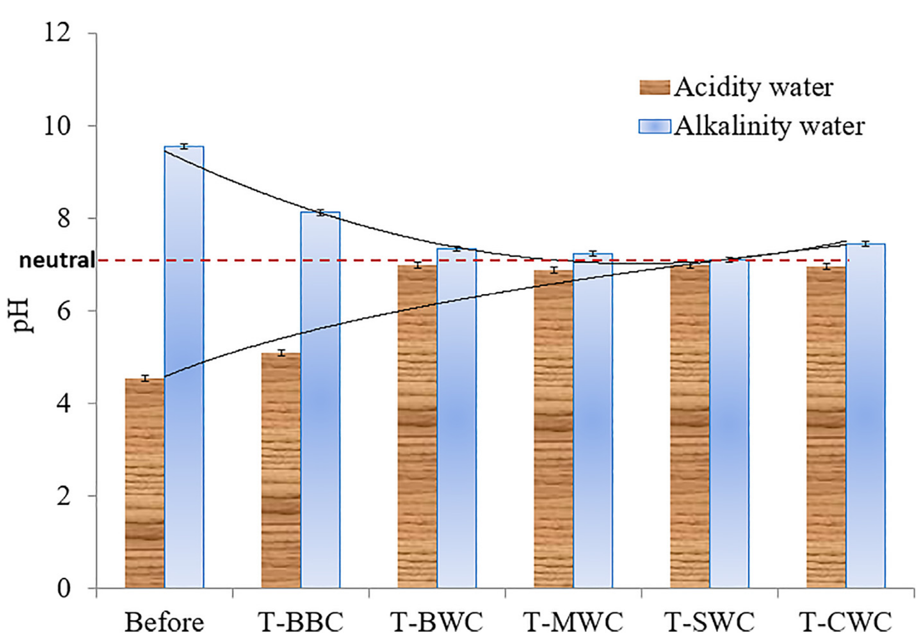

Fig. 6 – pH of the wastewater sample before and after treatment in both cases of acidity and alkalinity.

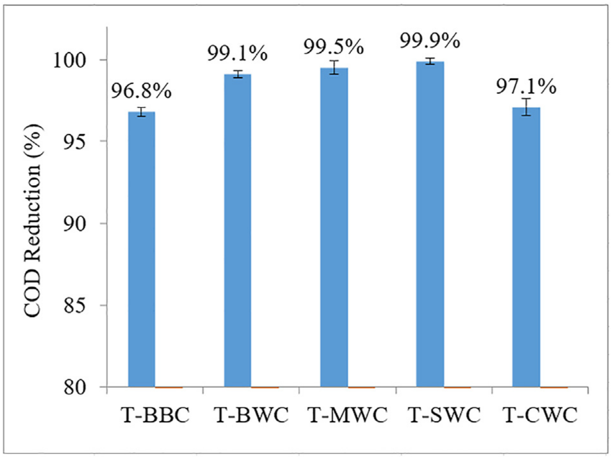

Fig. 7 – COD reduction of wastewater after treatment with BBC and BWC.

result may because the functional groups in the form of acetate ion (COO^-), proton (H^+), and hydroxide ion (OH^-) in the solution can adjust the pH by themselves. Therefore, the pH of wastewater was closely neutral in both cases of acidity and alkalinity. This point showed the automatics pH adjustment property of white charcoal to neutral wastewater. These results were shown in Fig. 6.

3.8.2. *Effect on chemical oxygen demand (COD)*

Fig. 7 shows a comparison of COD reduction of wastewater after treatment with BBC and BWC, respectively. The result verified that all of the white charcoal had higher efficiency than the BBC. This result may be from the specific surface area of all of the white charcoal (BWC, MWC, SWC, and CWC) that higher than BBC.

Table 4 presents some selected results of other investigations that have used adsorption. The calcined clay was used adsorbent and reported in terms of %COD reductions as 18% [38]. The zeolite-4A was used for 29% COD reduction [39], this value similarly to olive mill (Amberlite XAD16) of 25% COD reduction [40]. It was implied on activated carbon for high COD reduction, resulting in a 46% COD reduction [41], and reactivated carbon of 40% reduction [42]. However, above all, these values are lower than 50%. Many researchers reported higher %COD reduction. For example, soil doped with biochar gave higher % COD reduction (87%) [43]. Chitosan doped on coconut shell charcoal (Chitosan-CSC) gave a very high % COD reduction (99%) [44]. This work shows that using only pure white charcoal can reduce COD by 99.9% (SWC).

Table 4 – COD reduction compared with some others.

Sample	Initial COD (g/L)	Final COD (g/L)	COD reduction (%)	References
Clay (calcined)	40.0	62.8	18	[38]
Zeolite-4A	52.1	37.0	29	[39]
Amberlite XAD16	48.9	36.7	25	[40]
Activated carbon	60.0	32.4	46	[41]
Re-activated carbon	22.0	13.2	40	[42]
Soil-Biochar	NA	NA	87	[43]
Chitosan-CSC	3.4	0.034	99	[44]
SWC	26.8	0.23	99.9	This work

4. Conclusions

Black and four white charcoals are characterized using various techniques. The black charcoal had the highest ash and volatiles content but had fixed carbon content lower than four white charcoals. All charcoal contained a high content of potassium. Except miscellaneous white charcoal had calcium as the high content. White charcoal was amorphous carbon and a lower crystallinity structure than black charcoal. The black charcoal lump had a surface rougher than the white charcoal. The white charcoal is a rigid structure and stronger than black charcoal. All of the white charcoal had various small pore sizes and higher specific surface area than black charcoal. The highlight of this microstructural study as we found small holes connected with big holes in the coconut shell white charcoal. These results show that it is related to high specific surface area materials. Moreover, we found that both black and white charcoal can be used as an adsorbent for the wastewater treatment from a chemistry laboratory building. Especially white charcoal is good for neutralizing the pH of wastewater in both cases (acidity and alkalinity wastewater). Wastewater after treatment with white charcoal shows higher level of %COD reduction than after treatment with black charcoal, especially coconut shell white charcoal.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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