

Rice Husk Activated Carbon with NaOH Activation: Physical and Chemical Properties

(Karbon Teraktif Sekam Padi Diaktivasi dengan NaOH: Sifat Fizikal dan Kimia)

MOHAMAD JANI SAAD*, CHIA CHIN HUA, SUFFIAN MISRAN, SARANI ZAKARIA, MOHD SAIFUL SAJAB & MOHAMMAD HARIZ ABDUL RAHMAN

ABSTRACT

Activated carbon was produced from rice husk by activating with NaOH. Three types of samples were made at 850, 750, and 650 °C activation temperature. The properties of the samples were determined. The activated carbons have surface area of 429.82 m²/g from 850 °C activation, 121.39 m²/g (750 °C) and 93.89 m²/g (650 °C). The results were higher than rice husk carbon without activation (0.23 m²/g). The activated carbons have mesopore size (2-50 nm). Proximate and ultimate analyses of the samples were also determined. The activation process increased the carbon content of the samples. Physical characteristics of the activated carbons were shown from the XRD analysis. FTIR demonstrated the different functional of the rice husk carbon and activated. The SEM images showed the pores on the surface of the activated carbon due to the NaOH activation.

Keywords: Activated carbon; chemical properties; NaOH activation; physical properties; rice husk

ABSTRAK

Karbon teraktif (AC) daripada sekam padi telah dihasilkan dengan mengaktifkan NaOH. Tiga jenis sampel karbon teraktif dihasilkan pada suhu 850, 750 dan 650 °C. Keputusan luas permukaan sampel sekam karbon teraktif ialah 429.82 m²/g (850 °C), 121.39 m²/g (750 °C) dan 93.89 m²/g (650 °C). Nilai ini lebih tinggi jika dibandingkan dengan sekam karbon kawalan iaitu 0.23 m²/g. Saiz liang bagi karbon teraktif sekam padi ialah 2-50 nm iaitu dalam saiz mesoliang. Hasil analisis proksimat dan muktamad turut ditentukan. Proses pengaktifan telah meningkatkan kandungan karbon sampel. Ciri fizikal karbon teraktif ditunjukkan daripada analisis XRD. FTIR menunjukkan kefungsi berbeza karbon sekam padi dan teraktif. Pemerhatian daripada ujian SEM mendapati adanya liang-liang pada permukaan karbon teraktif hasil daripada pengaktifan NaOH.

Kata kunci: Karbon teraktif; pengaktifan NaOH; sekam padi; sifat fizikal; sifat kimia

INTRODUCTION

In Malaysia, more than 3 million tonnages of rice husk were generated from 685,545 hectares of paddy land in the year of 2017 (JPM 2017). Most of them were burned which creating air pollution, hence not healthy for the environment. Therefore, it will be meaningful if we can utilize the rice husk for useful products such as activated carbon (AC) which may give positive effect to the environment.

Charcoal is a carbon material from the process of carbonization and is a precursor for making AC. Due to cost constraint and dwindling of coal sources, charcoal can be produced from agricultural sources such as paddy,

coconut and banana (Danish & Ahmad 2018; Hamza et al. 2016; Kaman et al. 2017; Soltani et al. 2015).

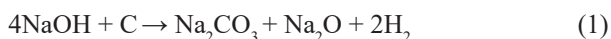
Activated carbon is a non-graphitic-graphitizable carbon with much disordered microstructure. It has a unit of basic structural which is nearer to the graphite structure. It has high adsorption capacity from its high porosity and surface area. It usually made from the activation process of carbon materials such as coal and cellulosic sources (Park & Oh 2002). AC mostly used in water treatment, pharmacy and medical (Abdulsalam et al. 2018; Alkhatib et al. 2015; Sharma & Bhattacharya 2017).

The production of AC can be either by chemical or physical activation process. Physical activation process

can be done using steam or carbon dioxide gas, whereas chemical activation using chemicals such as sodium hydroxide (NaOH) (Schröder et al. 2007), potassium hydroxide (KOH) (Chen et al. 2008; Enaime et al. 2017; Oh & Park 2002; Yakout et al. 2015), zinc chloride (ZnCl₂), iron oxide (Hidayu & Muda 2016; Zainol et al. 2017) or phosphoric acid (Mopoung et al. 2015; Shamsuddin et al. 2016) prior to heating at certain temperature in an inert gas (Kalderis et al. 2008). Activated carbon obtained through chemical activation produced a larger surface area and developed better mesoporosity (Khadiran et al. 2015; Nasri et al. 2015; Viboon et al. 2008).

Alkali hydroxides, such as NaOH and KOH, are usually used as agent for the activating of AC because of the good development of porosity produced by them (Foo & Hameed 2011; Guo et al. 2003; Perrin et al. 2004). Research on rice husk AC prepared using two-steps method had been reported earlier (Chang et al. 2014). The method includes biomass carbonization followed by activation. The method makes activating agent react more with the biomass carbon resulted in higher surface area and pore volume of the AC (Basta et al. 2009).

The chemical reactions of NaOH and carbon during the activation process can be written as follow (Chunlan et al. 2005):



Due to the Malaysia's rice husks ACs are less reported, therefore, in this research, rice husk AC was produced using two-steps method, i.e. carbonization followed by the activation processes. In this study, the physical and chemical properties of rice husk activated carbons prepared using two-steps method at three different activation temperature (650, 750, and 850 °C) which activated for 2 h with NaOH activation agent, were determine and analyzed by elemental analyses, Fourier-transform infrared (FTIR), Brunauer–Emmett– Teller (BET), X-ray diffraction (XRD) and scanning electron microscopy (SEM).

MATERIALS AND METHODS

MATERIALS

Rice husk was obtained from a rice mill in Sekinchan, Selangor, Malaysia. The rice husk was washed from contaminants using tap water, dried under the sun and then with oven. Activating agent, sodium hydroxide (NaOH), in granule form was purchased from Merck (M) Sdn. Bhd.

CHEMICAL ACTIVATION

The rice husk was carbonized in a closed chamber at 400 °C for 4 h for making rice husk carbon. Then, the rice husk carbon was separated from fine materials using sieve with mesh size of 60, then impregnated with NaOH by soaking in the 13% solution for 24 h with rice husk carbon to NaOH ratio of 1:4 (w:v). The mixture was filtered and dried in oven at 105 °C overnight. The impregnated rice husk carbon was activated in a tube furnace under nitrogen gas flow at certain temperatures (650, 750, and 850 °C) for 2 h. After that, the sample was cooled to ambient temperature and washed with hydrochloric acid solution (1.0 M) and deionized water to get pH6 to 7. The non-activated samples were labelled as RH (rice husk) and RHC (rice husk carbon). The rice husk carbon activated at 650, 750, and 850 °C were designated as RHAC650, RHAC750, and RHAC850 (or RHACNa), respectively.

PHYSICAL AND CHEMICAL ANALYSES

The samples were analyzed for carbon, hydrogen, sulphur, and nitrogen contents using elemental analyzer (Elementar Macro Cube). Proximate analysis was conducted to determine ash content, volatile matter, and fixed carbon. The samples were analyzed with Fourier-transform infrared (FTIR) spectroscopy (Perkin Elmer 2000) to examine the functional groups in region of 500 to 4000 cm⁻¹. The nitrogen adsorption-desorption analysis (Micromeritics ASAP 2010) was used to study the surface area and porosity. The X-ray diffraction (XRD) analysis was conducted with Bruker AXS D8 Advance in order to study the crystallinity of the samples. The morphology observations of the samples were done using scanning electron microscopy (SEM), FEI Quanta 400.

RESULTS AND DISCUSSION

The results of proximate and ultimate analyses of the RH, RHC and RHACNa are tabulated in Table 1. There are increasing in percentage of fixed carbon and reducing of ash content and volatile matter after the rice husk was activated. The RSACNa sample showed high percentage of carbon and low hydrogen, Sulphur, and nitrogen contents. The use of NaOH had enhanced the chemical activation of the material. The percentage of carbon content increased due to the carbonization and activation processes (Mohd Iqbalin et al. 2013; Rhaman et al. 2015).

The FTIR spectra of the RH, RHC and RHACNa samples are shown in Figure 1. The RH and RHC spectra were more intense as compared to the RHACNa at 3100-3600 cm⁻¹. The RHACNa was less intense could be due to less prevalent OH stretching as the results of the carbonization and activation processes. As such, the aromatic structure was developed (Oh et al. 2003). The same pattern also displayed from the absorbance at 1043 cm⁻¹ from OH bending (San Miguel et al. 2003). The

absorbance at 3425 cm^{-1} can be assigned to absorbed water or O–H stretching of OH groups (Guo & Rockstraw 2007). Weak band observed at $2800\text{--}2900\text{ cm}^{-1}$ of the RH and RHC spectra, indicating stretching vibrations of the C–H bonds of the methylene groups ($-\text{CH}_2-$) (Cazetta et

al. 2011). However, the bands were not seen in RHACNa sample. It could be due to the deformation of the C–H bonds to create an aromatic C=C bond at the higher activation temperature (Zhu et al. 2012). The absorbance between 700 and 1200 cm^{-1} related to aromatic which is out of plane C–H bending with different type of substitutions.

TABLE 1. Proximate and ultimate analyses results

Type of analysis	Samples		
	RH	RHC	RHACNa
Proximate			
Ash content (%)	30.04	19.11	13.09
Volatile matter (%)	63.32	14.06	7.47
Fixed carbon (%)	6.64	66.83	78.63
Ultimate			
Carbon (%)	39.63	55.08	69.02
Hydrogen (%)	4.39	7.83	4.70
Sulphur (%)	0.021	0.045	0.21
Nitrogen (%)	0.46	0.51	0.34

The spectra showed no SiO_2 peaks at 1101 , 944 , and 789 cm^{-1} (An et al. 2011). The absorbance at 1380 cm^{-1} is attributed from the deformation of aliphatic of CH_2 or CH_3 groups or bending of the O–H of phenolic–OH.

The band was weaker in AC sample, which is due to the aromatization and dehydration from the condensation and decomposition of volatile matter (Wu et al. 2012).

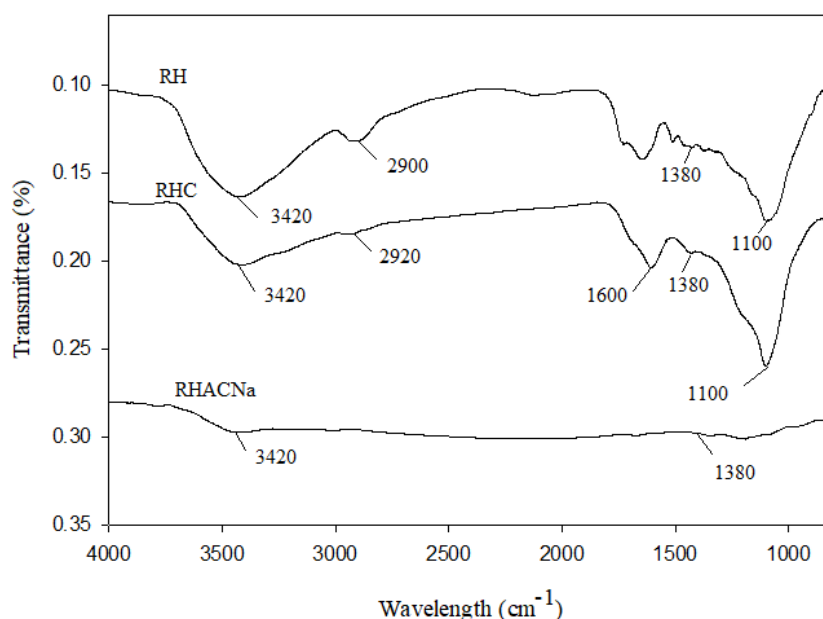


FIGURE 1. FTIR spectra of RH, RHC and RHACNa

The porosity characteristics i.e. total pore volume (V_{total}), pore sizes and surface area (S_{BET}) of RHC and RHACNa from the nitrogen adsorption-desorption test using BET model are demonstrated in Table 2. The surface area of RHC is 0.23 m^2/g which much lower than the RHACNa as results of the activation process. The surface area of rice husk carbon activated at 850 °C was highest (429.82 m^2/g) as compared to 750 °C (121.39 m^2/g) and 650 °C (93.89 m^2/g). The process temperature has favorable impact to the pores development. It could be because of the influence of volumetric widening of the pores and heat-induced internal (Foo & Hameed 2012). The volatile component

released at the time of heat activation process resulted in the improvement of BET surface area. The reducing of NaOH into metal Na probably performed during the process of activation (Eq. 1, 2, and 3). Na was diffused into the surfaces of carbon at the boiling point temperature and pores created in the structure of carbon which increasing the surface area (Rostamian et al. 2015). The RHACNa's total pore volume (V_{total}) increased with activation temperature in which the value varies from 0.12 to 0.29 cm^3/g and mostly contributed by pore area. Increasing in surface area resulted to the increase of mesopore volume (Viboon et al. 2008).

TABLE 2. Porosity properties of the RHC and RHAC

Property	RHC	RHAC650	RHAC750	RHAC850
S_{BET} (m^2/g)	0.23	93.89	121.39	429.82
V_{total} (cm^3/g)	0.0026	0.12	0.13	0.29
Average pore size (nm)	4.42	5.03	4.19	2.69

Average pore size of RHC and activated varies from 2.69 to 5.03 nm and fall in the mesopore size (2-50 nm). According to the IUPAC (1972), there are three pore sizes namely micropore (less than 2 nm), mesopore (2-50 nm) and macropore (more than 50 nm) (Lu et al. 2015). The pore size of the activated carbon is important due to its effect on the properties whenever used in the adsorption study. The adsorption ability of the activated carbon towards the adsorbates mostly influenced by the activated carbon's pore size. For example, the mesopore size suitable used for adsorption on water pollutants (Sobhy et al. 2015).

XRD diagram of the RHC and RHACNa are shown in Figure 2. It exhibits broad peaks in the range of 20 to 30°. They indicate the existence of amorphous silica (Puziy et al. 2002). The RHACNa showed two broad peaks at 20-30 and 40-50° which related to the existence of more amorphous carbon (Shamsuddin et al. 2016). During the activation, pores were created and crystal structure was disappeared (Ma & Ouyang 2013). The activated carbon made of graphite-like micro-crystallites was bounded by cross linking network including some graphite-like layer (Srenscek-Nazzal et al. 2013).

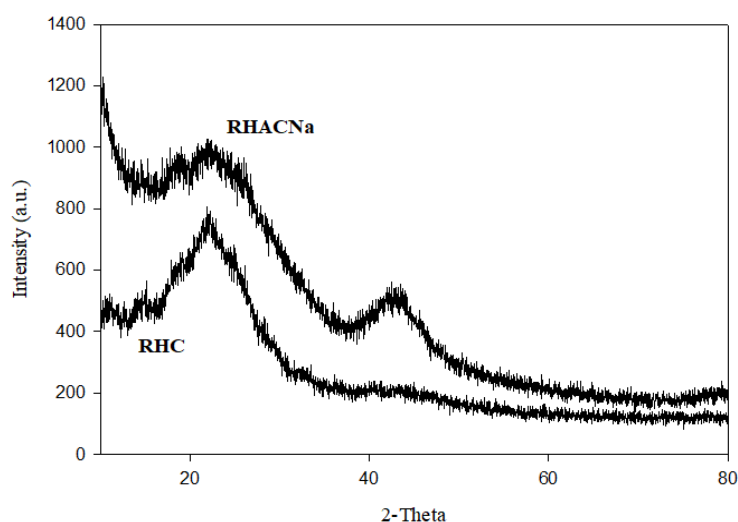


FIGURE 2. Results of XRD for RHC and RHACNa

Figure 3(a) shows SEM micrograph of the RHC with no pores. Developed pores can be found on the surfaces of RHACNa which could be due to the NaOH activation as shown in Figure 3(b). The NaOH activation occurred

in structure porous of carbon which created the pores (Pandey et al. 2015). The large pores produced high surface area as reported. The findings are similar with studies by Oh et al. (2003) and Oh and Park (2002).

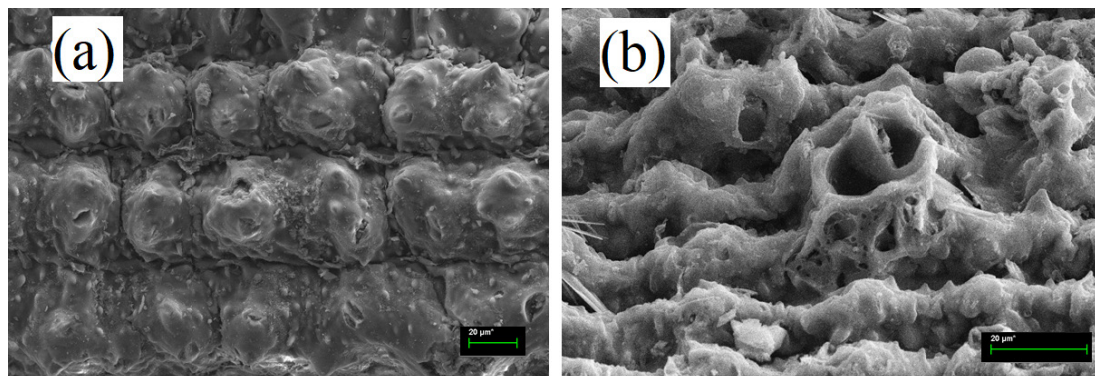


FIGURE 3. SEM of RHC (a) and RHACNa (b) at magnification of 500-1000×

CONCLUSION

The NaOH activation affected the properties of rice husk activated carbon. It reduced the ash content and volatile matter and increased the fixed carbon and carbon content. Some functional groups of the activated carbon were differed than the RH and RHC. Surface area and pore volume increased due the activation process and gradually increased as the activation temperature increased. The rice husk activated carbon attained highest surface area (429.82 m²/g) at 850 °C temperature. The pore sizes of the activated carbons were in mesopore sizes. Graphite-like micro-crystallites was observed on RHACNa from the XRD analysis. The surface of the RHACNa was porous as shown from the SEM image.

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- Mohamad Jani Saad*, Chia Chin Hua & Sarani Zakaria
Bioresources and Biorefinery Laboratory
Materials Science Program
Faculty of Science and Technology
Universiti Kebangsaan Malaysia
43600 UKM Bangi, Selangor Darul Ehsan
Malaysia
- Mohd Shaiful Sajab
Research Centre for Sustainable Process Technology
Faculty of Engineering and Built Environment
Universiti Kebangsaan Malaysia
43600 UKM Bangi, Selangor Darul Ehsan
Malaysia
- Sufian Misran
Forest Research Institute of Malaysia (FRIM)
52100 Kepong, Kuala Lumpur, Federal Territory
Malaysia
- Mohamad Jani Saad* & Mohammad Hariz Abdul Rahman
Malaysian Agriculture Research and Development Institute
(MARDI)
43400 Serdang, Selangor Darul Ehsan
Malaysia
- *Corresponding author; email: mohamadjanisaad72@gmail.com
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