

Preparation of Pinewood- and Wheat Straw-based Activated Carbon *via* a Microwave-assisted Potassium Hydroxide Treatment and an Analysis of the Effects of the Microwave Activation Conditions

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This study explored the effect of activated carbon preparation conditions on their adsorption performance. Pinewood and wheat straw were used as source materials to prepare activated carbon *via* a fast activation process using KOH and microwave heating. The iodine numbers and carbon yields were determined to evaluate the adsorption properties of the activated carbon. The effects of various KOH/char mass ratios, particle sizes, humidity levels, and microwave heating times on the physical characteristics of the activated carbon were investigated. The iodine number and yield and SEM images were used to characterize the activated carbon. Small particle sizes, the presence of humidity in the purge gas, and high KOH/char ratios resulted in higher iodine numbers. The best activated carbons were obtained using a KOH/char ratio of 3.0, a microwave power of 600 W, a radiation time of 30 min, and a particle size of 0.1 to 0.42 mm in a humid environment; these carbons showed iodine numbers of 2208 mg/g (pinewood activated carbon) and 1420 mg/g (wheat straw activated carbon), with carbon yields of 73% and 52%, respectively. Longer microwave heating times increased the iodine number. The iodine numbers and yields of the pinewood activated carbons were much higher than those of their wheat straw counterparts.

Keywords: Activated carbon; Microwave heating; Chemical activation; Iodine number

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INTRODUCTION

Pinewood has been firmly established as a first-class joinery wood for furniture, windows, doors, shutters, paneling, siding, moldings, and other architectural millwork and joinery items. Processing of pinewood generates lignocellulosic biomass in the form of pinewood sawdust and chips, which can amount to 20% of the total input mass (Foo and Hammed 2012a). Wheat straw residue is a by-product of existing agricultural practices, and the availability of wheat straw is high in Canada, Asia, and Africa. In these regions, wheat straw and pinewood are not only abundantly available but are also less expensive than non-renewable precursors, *e.g.*, natural coal (Ali *et al.* 2013).

These agricultural residues are disposed of *via* on-site combustion deposition in fields and landfills, resulting in the emission of air pollutants such as SO₂ and NO_x. To avoid this situation and provide a rapid technique for improving the processing of agricultural wastes (such as woodchips and wheat straw), agricultural residues are

currently being considered as promising precursors on account of their low cost, large surface area, extensive microporous structures due to their high concentrations of volatile matter and lignocellulosic material, and their renewable nature (Ahmed and Theydan 2013).

Microwave heating reduces the time required for char activation and is therefore more energy-efficient than conventional heating methods. Because microwave heating results in simultaneous internal and volumetric heating, it can be combined with the chemical activation of char to provide the aforementioned advantages while using less energy (Chen *et al.* 2011). In addition to being faster than conventional heating methods, microwave heating involves no direct contact between the heating source and the heated material; the materials are heated from the inside out, waste is minimal, and an increased rate of chemical reactivity can be achieved (Dehdashti *et al.* 2010).

Recently, research has focused on the preparation, surface characteristics, and adsorptive properties of the large surface area and well-developed micropore structures of activated carbon prepared using microwave heating. The preparation of activated carbon from phenolic resin (Williams and Parkes 2008) and cotton stalk (Deng *et al.* 2010) using acid activation with microwave heating has been reported. Foo and Hameed prepared a series of activated carbons from wood sawdust (Foo and Hamed 2012a), mangosteen peels (Foo and Hameed 2012b), biodiesel industrial solid residues (Foo and Hameed 2012c), sunflower seed oil (Foo and Hameed 2011a), and orange peels (Foo and Hameed 2012d) *via* microwave-assisted K_2CO_3 activation. Based on their studies, intensifying the activation, *e.g.*, by increasing impregnation ratios, microwave power, or radiation time, improves the BET surface area and mesopore ratio. Activated carbons derived from coconut husks using various activation agents (H_2SO_4 , H_3PO_4 , HNO_3 , K_2CO_3 , NaOH, and KOH) and microwave radiation displayed a high capacity for adsorption of methylene blue (Foo and Hameed 2012e).

Foo and Hameed reported the activation of pineapple peels (Foo and Hameed 2012f) and rice husks (Foo and Hameed 2011b) *via* K_2CO_3 and KOH activation and that of jackfruit peels (Foo and Hameed 2012g) and durian shells (Foo and Hameed 2012h) *via* NaOH activation using microwave irradiation. The results indicated that the activated carbon mixed with KOH achieved higher BET surface areas ($1006\text{ m}^2/\text{g}$) than K_2CO_3 ($680\text{ m}^2/\text{g}$) when using pineapple peels and a microwave heating time of 6 min. In addition, the chemical activation of bamboo (Liu *et al.* 2010) and pinewood powder using H_3PO_4 and $ZnCl_2$ *via* microwave heating was reported to be a highly suitable process for the preparation of activated carbon with high BET surface areas and highly porous structures.

Based on all of these studies, the preparation of activated carbon from agricultural waste *via* chemical and microwave-induced activation has been widely studied. Among the various chemical agents, it has been demonstrated that the soaking of KOH into the pores of activated carbon favors the development of micropores, resulting in promising application potential in environmental fields such as air pollution control (Tseng and Tseng 2005). Depending on the precursor of the activated carbon and the various activating agents during microwave-induced activation, the preparation conditions and adsorption characteristics still need to be optimized to a certain extent. For example, the effect of the KOH/char ratio should be investigated because KOH is expensive. Moreover, increasingly stringent environmental regulations require the use of high-quality activated carbons in industry, which also results in the optimization of activation processing.

Owing to the abundance and low cost of pinewood and wheat straw, these materials are suitable raw materials for the preparation of activated carbon. However, to our knowledge, there are no reported studies of the preparation of activated carbon from pinewood and wheat straw using KOH and microwave-induced activation, particularly regarding the effects of the preparation conditions on the adsorption properties of the final product. Traditional activation is a high-energy process due to slow conventional heating and the high temperatures that are required, which cause this process to be both energetically and commercially expensive (Chen and Hashisho 2012). Current interest in the low cost and the time savings of microwave preparation of activated carbon has encouraged research into alternative activation methods for preparing high-quality activated carbon derived from pinewood and wheat straw (Dehdashti *et al.* 2010).

The aim of this study was two-fold: (1) to prepare pinewood chip- and wheat straw-based activated carbon using potassium hydroxide (KOH) as the activating agent and microwave irradiation as the heat carrier and (2) to analyze the impact of the char particle size, humidity of the activation carrier gas, KOH/char ratio, and activation time on the iodine number and the yield of activated carbon. The amount of iodine adsorbed (in milligrams) per gram of carbon at a residual iodine concentration of 0.02 N was obtained from the corresponding adsorption isotherm and was reported as the iodine number. In this study, an iodine number test was conducted at least twice on each sample. The ratio of the weight of activated carbon (after washing and drying) to the weight of the dry raw char is herein defined as the activated carbon yield.

EXPERIMENTAL MATERIALS AND METHODS

Materials

Pinewood chips and wheat straw collected from Millar Western Forest Products Ltd. in Alberta, Canada, were the precursors used in this study. The samples were washed thoroughly with deionized water to remove any adhering dirt particles from the surfaces. The samples were first air-dried for 5 days and then oven-dried overnight at 80 °C. The dried woodchips and wheat straw were cut to lengths shorter than 3 cm prior to being stored in sealed containers for the experiments. A chemical activating agent, KOH, with a purity greater than 85.0% (Wako Pure Chemical Industries, Ltd., USA) was used as the impregnation chemical in the experiments.

Methods

Preparation of the activated carbon

The prepared pinewood chips and wheat straw were placed into a lab-scale muffle furnace consisting of a cylindrical quartz reactor 80 mm in diameter and heated at a rate of 10 °C/min from room temperature to 550 °C under a humid N₂ flow (0.5 L/min). In this oxygen-limited environment, the pinewood chips and wheat straw were thermally pyrolyzed to porous carbonaceous materials (char) and hydrocarbon compounds. After carbonization, the char was cooled to room temperature under an N₂ flow. The char was then ground and sieved to a uniform particle size range of 0.1 to 5 mm. The char product was impregnated with KOH at KOH/char ratios of 0.5, 1.5, and 3.0 based on the dry weight of KOH and the weight of the pinewood char or wheat straw char. The resulting samples were designated PAC0.5, PAC1.5, and PAC3.0 and WAC0.5, WAC1.5, and

WAC3.0. The mixtures were stirred for 2 h, dried overnight at 110 ± 5 °C, and then stored in a desiccator.

For the microwave activation, 30 g of KOH-impregnated char was added to a ceramic crucible in a cylindrical quartz reactor, placed in a 2450-MHz microwave (Goldstar Co., Ltd., China) with a nominal power output of 600 W, and irradiated for 30 min under a 0.5-L/min flow of humidified N₂; the gas was introduced by bubbling through water. After the microwave activation, the KOH-impregnated char was cooled to room temperature using a flow of humid N₂ at a rate of 0.5 L/min. The samples were then mixed with 0.1 N hydrochloric acid (HCl) and stirred for 1 h. Then, they were washed with deionized water until they reached a pH of 7. The activated char samples were finally dried at 110 ± 5 °C for 24 h. The experimental setup is shown in Fig. 1. Each char sample was activated twice to confirm the results. The ratio of the weight of the activated carbon (after washing and drying) to the weight of the dry raw char is defined as the activation yield.

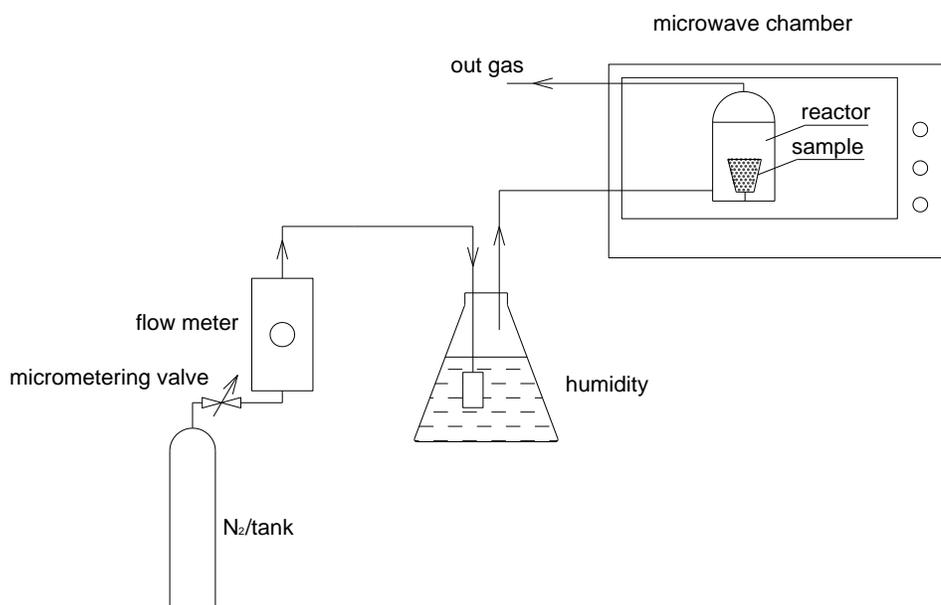


Fig. 1. Experimental setup for microwave activation

Activated carbon characterization

The iodine number is an index that is used to express the adsorption capacity, surface area, and porosity of the activated carbon. In this experiment, the iodine number was determined using ASTM D4607 (2006) and expressed in milligrams of iodine adsorbed by the activated carbon (mg/g) (Chen and Hashisho 2012). For each sample, this analysis was performed twice to ensure the consistency of the results.

The shapes and morphology of the pinewood and wheat straw char were evaluated using scanning electron microscopy (SEM; Hitachi S-2500 Tokyo, Japan). The samples were coated with gold to ensure that the particles had suitable conductivity. For better image quality, the secondary electron images were selected. The acceleration voltage was set to 8 kV, and the magnification was 200.

RESULTS AND DISCUSSION

Effects of Particle Size

The effect of the raw char particle size on the quality of the activated carbon obtained with a KOH/char ratio of 3.0, microwave heating time of 30 min, and microwave power level of 600 W was assessed. As is well known, the precursor in the preparation of activated carbon is often crushed to a small particle size during the chemical activation step. Therefore, after activation, sieving was performed to obtain three particle size distributions of the activated carbon: size I (2.00-4.76 mm); sizes II (0.42-2.00 mm) and III (0.10-0.42 mm). Figure 2 depicts the iodine number of the activated carbon as a function of the particle size. For a better comparison, the iodine number of the raw char is also presented. The pinewood char and wheat straw char had specific iodine numbers of 375 mg/g and 182 mg/g, respectively. The iodine number of high-grade activated carbon is typically greater than 1000 mg/g (Chen and Hashisho 2012). In this study, the iodine number of the raw char was lower than that of a typical activated carbon. After the microwave activation with KOH, the iodine number of the activated carbon composed of large particles (size I) was approximately 45% lower than that of the carbon composed of small particles (sizes II and III). The iodine number of the activated carbon steadily increased as the activated carbon particle size decreased. Therefore, the grinding of activated carbon to obtain a smaller particle size may result in a greater adsorption capacity. The iodine numbers of the pinewood increased by approximately 29% with a reduction in particle size from size I to size II, whereas in the wheat straw activated carbons, the iodine numbers increased by approximately 3% with a reduction from size II to size III. Essentially, larger particles result in lesser penetration by KOH and a reduction in activation reactions (Chen and Hashisho 2012). Therefore, smaller activated carbon particles can achieve better activation and higher iodine numbers.

The iodine number of the activated wheat straw char was consistently lower than that of the activated pinewood char, which could be explained by the differences in the structures of the two chars. Pinewood char has a more ray-like xylem structure (Fig. 3a) than wheat straw char (Fig. 3b), and it has been reported that pinewood char has a 12% to 20% volatile content, whereas wheat straw char has a 7.6% to 8.6% volatile content (Lehmann and Joseph 2009).

The higher volatile content of pinewood allows for the formation of interconnected pores, which facilitates the diffusion of KOH into the pinewood char particles, resulting in a well-developed micropore structure (Chen and Hashisho 2012). In addition, the ray-like xylem structure of pinewood char is due to the structure of xylem ray cells in pinewood. Therefore, after carbonization, the final char maintains the original structural features of pinewood.

This type of structure favors the development of micropores inside the char particles, which makes the material more accessible to KOH impregnation *via* the xylem ray holes. Such a mechanism can explain why the adsorption capacity of activated pinewood char is greater than that of activated wheat straw char (Tseng and Tseng 2005).

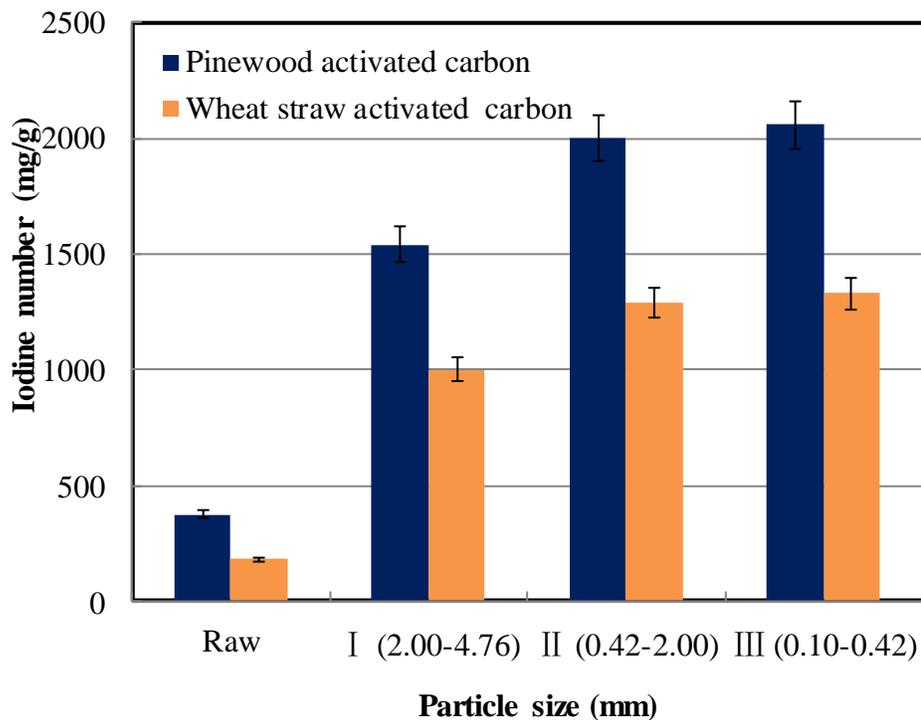


Fig. 2. Iodine numbers of activated carbons as a function of particle size (KOH/char ratio of 3.0, microwave heating time of 30 min, microwave power level of 600 W, and a dry nitrogen environment). Each value is the average of two iodine number measurements \pm one standard deviation.

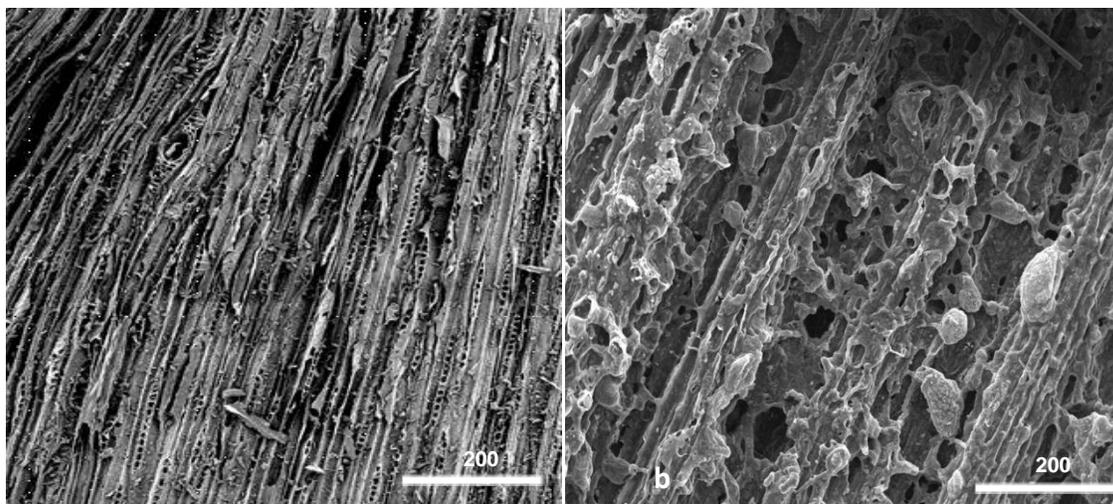


Fig. 3. SEM images of raw pinewood and wheat straw char. (a) pinewood char and (b) wheat straw char

Effect of Humidity

The effect of humidity on the activation environment during microwave activation was also studied for particles of size III (0.42 to 0.1 mm) that were activated at a KOH/char ratio of 3.0 for 30 min at a microwave heating power of 600 W. Dry nitrogen was passed through an impinger containing water before being introduced into the activation chamber. Figure 4 depicts the effect of the humid N₂ on the iodine number. As expected, the iodine numbers of the activated carbons derived from pinewood and wheat straw increased, by 34% and 28%, respectively, when the humid vapor setup was used, which allowed for a type of physical activation involving H₂O. Through this process, porous structures can develop, and the reaction proceeds as follows:



Furthermore, the impregnation of the char with KOH created additional pores, which facilitated the access of water molecules to the carbon skeleton for additional activation. In addition, potassium acts as a catalyst that increases the rate of reaction between water molecules and carbon atoms (Wu *et al.* 2005). Therefore, the use of humidified nitrogen resulted in higher iodine numbers than the use of dry nitrogen. The ray-like xylem structure of the pinewood char favored the diffusion of water vapor into the cores of the pinewood char particles. Therefore, the use of humidified nitrogen had a greater effect on the pinewood char than it did on the wheat straw char.

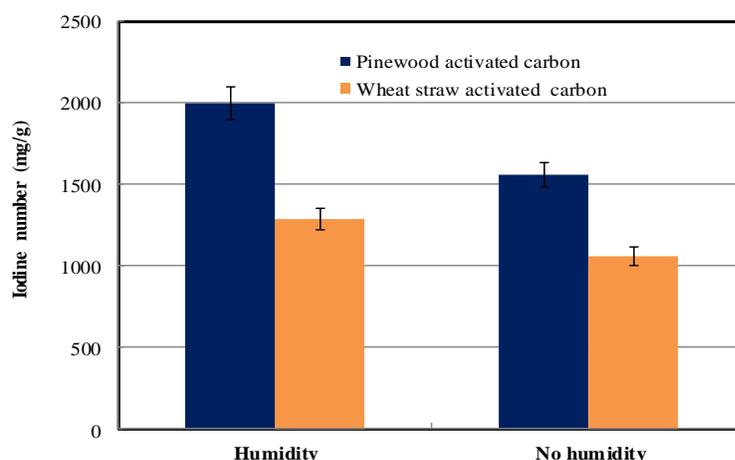


Fig. 4. The effect of humidity on the microwave activation of pinewood and wheat straw chars (microwave power of 600 W, particle size III, KOH/char ratio of 3, microwave heating time of 30 min). Each value is the average of two iodine number measurements \pm one standard deviation

Effect of the KOH/Char Ratio

The effect of the KOH/char ratio on the properties of the activated carbons was studied for particles of size III (0.42 to 0.1 mm) that were exposed to a microwave power of 600 W for 30 min under humidified nitrogen. A high microwave power of 600 W was used because KOH absorbs microwaves more weakly than do chars. Therefore, with a higher KOH/char ratio, more power was needed to heat and activate the chars. Figure 5 illustrates the average iodine number as a function of the KOH/char ratio (microwave power of 600 W, particle size III, humidified nitrogen, and microwave heating time of 30

min). Each value is the average of two iodine number measurements \pm one standard deviation. Even a low KOH/char ratio of 0.5 was effective in activating the chars.

The iodine numbers of the pinewood and wheat straw chars activated with a low KOH/char ratio of 0.5 were sharply increased, by 3 and 6 times, respectively, compared with those of the raw chars. Tseng and Tseng also reported similar effects due to KOH: when the ratio of KOH to corncob char increased from 0 to 0.5, the surface area was greatly increased from 309 to 841 m²/g (Tseng and Tseng 2005). Moreover, Foo and Hameed (2012f) reported that the soaking of KOH into pineapple peels char resulted in a much greater surface area than that of K₂CO₃ under microwave heating. It can be clearly observed that the iodine number also increased when using a higher KOH/char ratio. For example, when the KOH/char ratio was increased from 0.5 to 3, the iodine numbers of pinewood activated carbon increased from 1126 to 2170 mg/g, and the values of wheat straw activated carbon improved from 1078 to 1399 mg/g. These values are similar to those reported by others. For example, the surface area of corncob activated at a KOH/char ratio of 3 under conventional heating was found to be 1976 m²/g (Tseng and Tseng 2005). This difference may be due to the effects of microwave heating. Note the higher iodine adsorption values of pinewood and wheat straw activated carbons (iodine number of 1126 mg/g for pine activated carbon and 1078 mg/g for wheat straw activated carbon) with KOH/char ratio of 0.5 in comparison with that of activated carbons (BET surface area of 1006 m²/g) prepared from pineapple peel using microwave irradiation and an impregnation KOH/char ratio of 1.5, as reported by Foo and Hameed (2012f). Therefore, pinewood and wheat straw wastes show promise as raw materials for preparation of activated carbon, particularly in potential applications in pollution control systems and environmental remediation.

Interestingly, the increase was not directly proportional. For example, the iodine number of pinewood activated carbon increased approximately 69% as the KOH/char ratio was increased from 0.5 to 1.5 but increased only 14% when the KOH/char ratio was further increased from 1.5 to 3.0.

The development of porosity is associated with the reaction of KOH and C under inert conditions, which leads to the formation of K₂O, K, K₂CO₃, CO, and CO₂. The potassium species formed during the activation step diffuse into the internal structure of the char matrix, which widens existing pores and creates new pores. The activation involving KOH proceeds according to the following reactions (Otowa *et al.* 1997):



Therefore, by increasing the KOH/char ratio, the activation process is strengthened. Accordingly, the pore width is successively broadened, and new micropores and mesopores are created in the original pore walls. Finally, further enhancements in the iodine numbers of both the pinewood and wheat straw activated

carbons are achieved (Foo and Hammed 2012a). A similar conclusion was reported by Ahmed and Theydan (2013), who investigated the effect of increasing the KOH/char ratio from 0.4 to 1 on siris seedpods after activation for 8 min at 620 W. The authors' results indicated that the increase in the KOH/char ratio led to an increase in the iodine number from 1332 to 1760 mg/g.

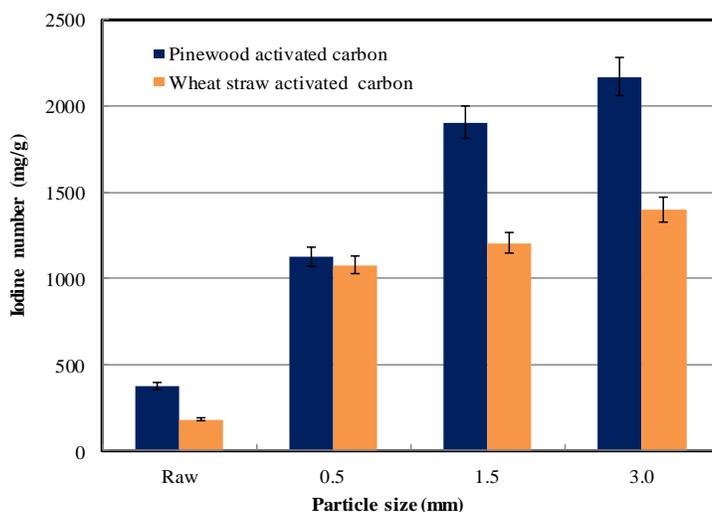


Fig. 5. The effect of the KOH/char ratio during microwave activation of pinewood and wheat straw chars (microwave power of 600 W, particle size III, humidified N₂, microwave heating time of 30 min). Each value is the average of two iodine number measurements \pm one standard deviation

Effect of Microwave Heating Time

The microwave heating time affected the development of pores in the chars and affected the carbon yield. The effect of the microwave heating time was studied in particles of size III (0.42 to 0.1 mm) that were activated at a KOH/char ratio of 3.0, in a humid nitrogen environment and at microwave power of 600 W. Figure 6 shows the iodine number (a) and yield (b) of pinewood and wheat straw activated carbons as a function of the microwave heating time. Even a microwave heating time as short as 10 min increased the iodine number of the pinewood activated char from 375 (raw char) to 1102 mg/g. This finding implies that prolonging exposure to microwave radiation results in an increase in temperature, which in turn increases the reaction rate, thus improving iodine adsorption (Foo and Hammed 2012a). However, the iodine number did not vary monotonically with the microwave heating time. For instance, for pinewood char, increasing the microwave heating time from 10 min to 20 min resulted in a 41% increase in the iodine number; however, increasing the microwave heating time from 30 min to 40 min increased the iodine number by 10%. This phenomenon was also reported by Foo and Hameed and can be explained by a dramatic increase in temperature, which led to the opening of micropores and mesopores and caused an increase in pore sizes as the radiation time increased (Foo and Hammed 2012a).

The activation of wheat straw char over 10, 20, 30, and 40 min of microwave heating was repeated to confirm the observed trend. The iodine number of wheat straw activated carbon displayed a similar trend with microwave heating time, the only difference being that the iodine number of the wheat straw activated carbon was approximately two-thirds that of the pine activated carbon, which was due to the differences in the structures of the chars.

The yield of prepared activated carbon decreased with increasing microwave heating time because higher activation temperature promoted C-H₂O, C-K₂CO₃, C-K₂O, and C-CO₂ reactions, which were facilitating the breaking of C-O-C and C-C bonds (Foo and Hammed 2012a). Increasing the heating time from 10 min to 40 min resulted in decreases in the carbon yields of pinewood activated carbon and wheat straw activated carbon of 89% to 68% and 68% to 44%, respectively. Figure 6b shows that the pinewood activated carbon exhibited a higher yield than did the wheat straw activated carbon, which may have been due to the different composition of the pinewood activated carbon in terms of extractives and cell wall components. In addition, the pinewood char contained a greater amount of ash (420.1 g/kg in pinewood char, 250.7 g/kg in wheat straw char), which may play a catalytic role during the activation process and alter the product distribution in terms of the yield of gas and carbon (Pushkaraj and Patwardhan 2010).

The yield of pinewood activated carbon (89%) using a KOH/char ratio of 3.0 and an irradiation time of 10 min was still comparable to the values reported for the K₂CO₃ activation of wood sawdust with microwave heating (77% using a K₂CO₃/char ratio of 2.0 with a 5-min irradiation time) (Foo and Hammed 2012a).

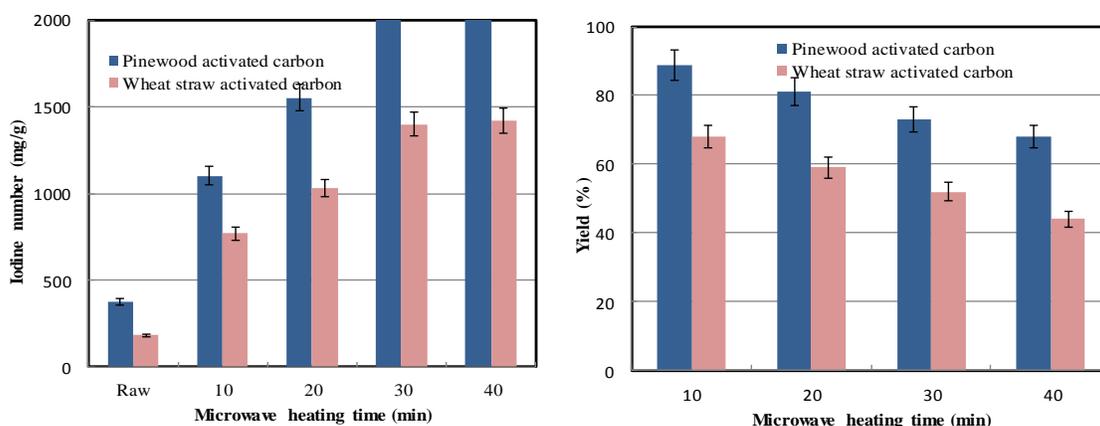


Fig. 6. (a) Iodine number and (b) yield of pinewood and wheat straw activated carbon with various microwave heating times (power of 600 W, particle size III, humidified nitrogen, and KOH/char ratio of 3.0). Each value is the average of two measurements \pm one standard deviation

These results are encouraging because they indicate that biomass can be converted to activated carbon following treatment of short duration. Therefore, microwave heating poses a significant advantage for the preparation of biomass-based activated carbon. This conversion can be improved by further optimizing the activation conditions. Future research could optimize the activation conditions to further improve the conversion process.

CONCLUSIONS

The results of this study show that pinewood chips and wheat straw are potential precursors for the preparation of high-quality activated carbon. Microwave heating increased the porosity of the activated carbon materials after a short heating period.

Experimental results show that microwave heating could reduce processing time remarkably and could yield a high-quality product, presumably because of the deeply penetrative and volumetric heating characteristics of the technique. Furthermore, this study demonstrated the effects of various factors on the preparation of high-quality activated carbon.

1. The best preparation conditions were as follows: particles of size III (0.42 to 0.1 mm), a KOH/char ratio of 3.0, and 30 min of 600-W microwave heating in the presence of humidity. The optimum pinewood and wheat straw activated carbons had iodine numbers of 2208 mg/g and 1420 mg/g, respectively. Moreover, the carbon yields were 73% and 52% for the pinewood and wheat straw activated carbons, respectively.
2. The iodine number of the activated wheat straw char was consistently lower than that of the activated pinewood char because pinewood char has a ray-like xylem structure.
3. Increasing the microwave heating time led to an increase in the iodine number. In addition, the iodine number of the activated carbons steadily increased as the activated carbon particle size decreased.
4. The presence of humidity in the activation environment resulted in a higher iodine number.
5. The iodine number did not vary monotonically with the microwave heating time, indicating that there is an optimum microwave heating time.

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