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Utilization of walnut shell as a feedstock for preparing high surface area activated carbon by microwave induced activation: effect of activation agents

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Abstract: The present work attempts to convert walnut shell into a high surface area activated carbon by microwave heating and chemical activation. Different activation agents such as KOH, NaOH, K_2CO_3 and Na_2CO_3 were utilized to identify a suitable activation agent. The result indicated that KOH was the most suitable activation agent among those agents, with the highest porosity and surface area of activated carbon. The effect of KOH/C mass ratio on the pore structure characteristics of the activated carbon was investigated. An optimal KOH/C mass ratio of 4 was identified, beyond which the surface area as well as the pore volume were found to decrease. The surface area and the pore volume were estimated to be 3276 m²/g and 1.952 ml/g, respectively, meanwhile the microporous proportion was 77.51% under the optimized KOH/C mass ratio. The activated carbon was further characterized using Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The findings strongly support that the activation agent has a great effect on the preparation of activated carbon from walnut shell.

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Keywords: activation agent; high surface area activated carbon; microwave heating; walnut shell.

1 Introduction

Walnut shell is grown in large quantities and is a renewable source of biomass. According to statistics, more than 1 million tons of walnut shells are being produced in China annually [1]. Activated carbon can be produced from different types of materials like lignite, peat, coke, wood, sawdust and coconut shell and so on [2–5]. The properties of the activated carbon depend on the type of precursor as well as the activation methods [6]. In common opinion, advantages of activated carbon are low cost, hydrophobicity and flexibility in terms of porous texture and surface properties [7]. Activated carbon is an important material with well-developed pore structure and good mechanical characteristics, which is mainly used in environmental applications, such as water treatment, environment protection, gas filters, catalyst and catalyst support, etc. [8–12].

In principle, the methods for preparing an activated carbon can be divided into physical and chemical activation [13]. Physical activation is realized in two stages of carbonization and activation; the activation of char is performed at a high temperature (usually above 1073 K) with steam, carbon dioxide or their mixture [14–17]. Chemical activation is usually realized in one stage. The precursor is impregnated with chemical agents like KOH [18, 19], K_2CO_3 [20, 21], $ZnCl_2$ [22], H_3PO_4 [23, 24] and activated at comparatively lower temperatures than the physical activation. Chemical activation has several advantages compared to physical activation, such as higher yields and well-controlled porosity.

The conventional heating method is surface heating, which does not ensure a uniform temperature, as the heat transfers through conduction and convection. Microwave heating is volumetric and the heat is generated from inside the material through dipole rotations and ionic conduction.

Microwave heating has been proved beyond doubt to possess qualities such as fast heating, is energy efficient, easy to control, has small thermal inertia and selective heating. Application of microwave heating is increasingly being adopted for processes such as biomass conversion, activated carbon preparation and so on [25–28].

The main purpose of this work was to find the suitable activation agent for preparing walnut shell based activated carbon from KOH, NaOH, K_2CO_3 and Na_2CO_3 by microwave heating. The resultant product was characterized using the nitrogen adsorption isotherm, Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) analysis.

2 Materials and methods

2.1 Materials

Walnut shell was obtained from Yangbi in Yunnan province of China. The starting material was manually chosen, grounded to 5–7 mm, cleaned with distilled water, dried at 110°C for 48 h and finally stored in sealed containers for next experiments. The proximate analysis of the walnut shell is shown in Table 1. The activation agent such as KOH, K_2CO_3 , NaOH, Na_2CO_3 with analytical grade (Tianjin Chemicals Reagent, Tianjin, China).

2.2 Carbonization of walnut shell

The carbonization of walnut shell was carried out by loading 1000 g into a high temperature conventional resistance furnace, and heating to the carbonization temperature of 600°C at a heating rate of 10°C/min and held for 2 h under N_2 atmosphere (100 cm^3/min). Then, the carbonized materials were cooled to room temperature with N_2 atmosphere (100 cm^3/min). Last, the resultant chars were crushed to particles with sizes of 600–1000 μm .

2.3 Preparation of activated carbon

The activation experiments were carried out in a self-made microwave tubular furnace with a multi-mode continuous controllable power. The microwave frequency was 2.45 GHz, while the output power could be set to a maximum of 1000 W.

Firstly, a certain weight of activation agent and char were mixed completely according to a fixed mass ratio. Secondly, these were placed in a ceramic crucible, and were heated for 15 min at the microwave power of 700 W under nitrogen atmosphere (300 cm^3/min).

Table 1: Proximate analysis result of walnut shell.

Sample	Moisture (wt.%)	Volatile matter (wt.%)	Fixed carbon (wt.%)	Ash (wt.%)
Walnut shells	10.91	70.71	17.65	0.73

Thirdly, the activated samples were rapidly transferred to the beaker loaded with distilled water, followed by washing with 1:1 HCl (by volume) to eliminate the residual alkali. Then, the samples were rinsed with hot distilled water until the pH was about 7. Finally, the cleaned samples were dried at 110°C for 12 h. The activated samples with high temperature were rapidly transferred to the distilled water for the gasification reaction char to produce more holes.

2.4 Characterization of activated carbon

The iodine adsorption number is an important parameter and widely used to characterize microporous material; it was calculated using the standard testing methods of the People's Republic of China (GB/T12496.8-1999) [29] for activated carbon. The yield was defined as the weight ratio of activated carbon to char utilized for activation. The porosity of the resultant sample was characterized with N_2 adsorption at 77 K using an automatic adsorption apparatus (Autosorb-1-C, Quantachrome). The surface area was calculated by the Brunauer–Emmett–Teller (BET) method. The total pore volume was determined at a relative pressure (P/P_0) of 0.98. The t-plot method was applied to calculate the micropore volume and external surface area. In addition, the pore size distribution was determined by non-local density functional theory (NLDFT) method. FTIR (Nicolet 8700, Thermo Nicolet Co., USA) was applied to identify the chemical function groups of activated carbon. The microstructure was analyzed by SEM (Philips XL30ESEM-TMP).

3 Results and discussion

3.1 Effect of activation agent

3.1.1 The iodine number and yield of activated carbon

Activated carbons were prepared at a microwave power of 700 W and heating time of 15 min, with an activation agent/char mass ratio of 2 using KOH, NaOH, K_2CO_3 and Na_2CO_3 . The effect of activation agent on the iodine number and yield are shown in Figures 1 and 2, respectively.

Figure 1 shows that the activation agent has a significant influence on the iodine number of activated carbon. The iodine number of activated carbon prepared by a different activation agent was of the order of $KOH > NaOH > K_2CO_3 > Na_2CO_3$. The iodine number of activated carbon prepared by KOH activation was the highest. The iodine number was equal substantially with NaOH and K_2CO_3 activation, but the iodine number was the lowest obtained from Na_2CO_3 activation. Figure 2 shows that the yield of activated carbon doesn't increase with the increase in iodine number for different activation agents. An increase in the porosity of the carbon made the yield of carbon reduction in turn.

During KOH and K_2CO_3 activation, the reactions are complicated, but generally the reactions may be as

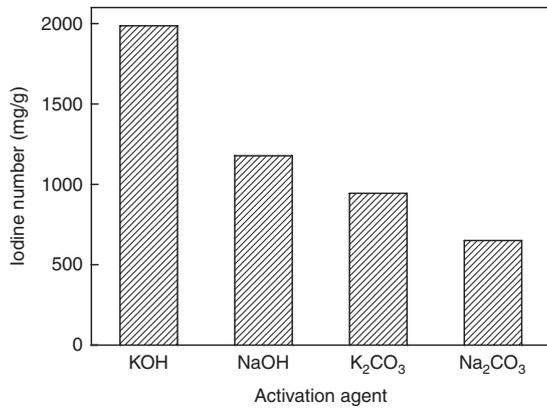


Figure 1: Effect of activation agent on the iodine number of activated carbon.

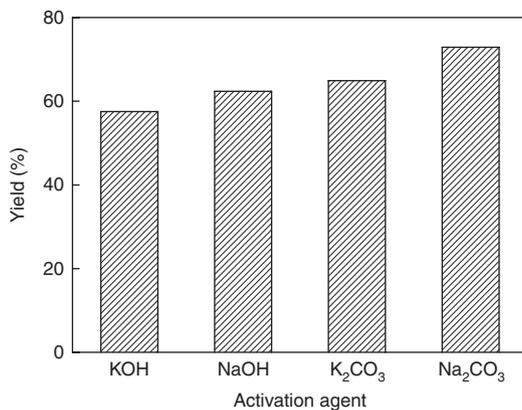
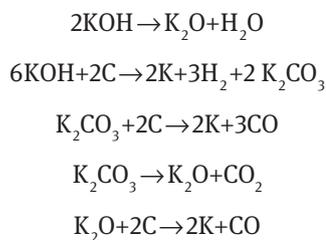


Figure 2: Effect of activation agent on the yield of activated carbon.

follows; in addition, there are similar reactions during NaOH and Na₂CO₃ activation [18, 30, 31]:



Concurrently, alkaline hydroxide activation involves the redox reduction and carbon oxidation to generate porosity. Gases such as CO₂ and CO are generated by these chemical reactions, resulting in the formation of ultramicropores in mesopore walls, which can also contribute to increasing surface area and pore volume. The main difference in behavior between KOH, K₂CO₃ and NaOH, Na₂CO₃ can be explained by the different intercalation ability of K and Na with the structural order and organization of the pristine material [18].

3.1.2 Pore structure of activated carbon

The N₂ adsorption isotherms of activated carbon using different activation agents are shown in Figure 3. Figure 3 shows that the isotherms of activated carbon with KOH, NaOH and K₂CO₃ activation can be categorized to be a type I isotherm according to the International Union of Pure and Applied Chemistry (IUPAC) classification [32]. When the relative pressure (P/P_0) was below 0.1, N₂ adsorption increased quickly for the activated carbon. More than 70% of nitrogen was adsorbed within the region of very low relative pressures $P/P_0 < 0.1$, indicating the microporous character [33]. This type of isotherm was usually exhibited by microporous solids that include a well-developed mesopore structure. The isotherm of activated carbon obtained from Na₂CO₃ activation belongs to the type II isotherm. N₂ adsorption increases has a similar trend at low relative pressure, and the adsorption capacity reached 53.2% of saturation adsorption capacity at the relative pressure of 0.1, indicating more mesoporous than other activation agents. From the micropore and mesopore volumes listed in Table 2, it is inferred that the mesopores account of the activated carbon obtained by Na₂CO₃ activation was 58% more than other activated carbons in total pore volume.

Table 2 shows the results of the adsorption isotherms in terms of surface area, micropore volume and total pore volume. The highest surface area was 1835 m²/g, corresponding to the KOH activation, with much higher pore volume compared to the other activation agents. In addition, the proportion of micropore volume to the total pore volume was much higher than with the other activation agents. The Na₂CO₃ activation resulted in a very low surface area of 659.6 m²/g. Based on this fact, KOH performed much better than other activation agents in terms of its ability to produce high surface area carbon. The average diameter of

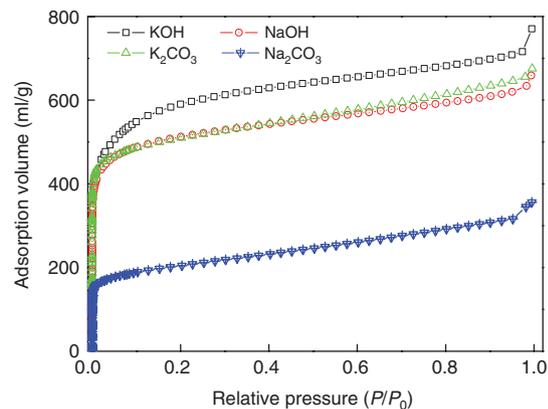


Figure 3: N₂ adsorption isotherm of activated carbon with different activation agent.

Table 2: Pore structure characteristics of activated carbon with different activation agent.

Activation agent	S_{BET} (m ² /g)	V_{tot} (ml/g)	D (Å)	V_{mic} (ml/g)	V_{mes} (ml/g)	S_{mic} (m ² /g)	S_{External} (m ² /g)	$V_{\text{mic}}/V_{\text{tot}}$ (%)
KOH	1835	1.1100	24.2	0.8122	0.2978	1560	274.7	73.17
NaOH	1558	0.9831	24.77	0.6980	0.2851	1345	242.8	71.00
K ₂ CO ₃	1579	1.0170	25.76	0.6846	0.3324	1304	274.7	67.32
Na ₂ CO ₃	659.6	0.5353	32.46	0.2227	0.3126	235.7	423.9	41.60

activated carbon doesn't increase with the increase in $V_{\text{mic}}/V_{\text{tot}}$ of activated carbon with different agents.

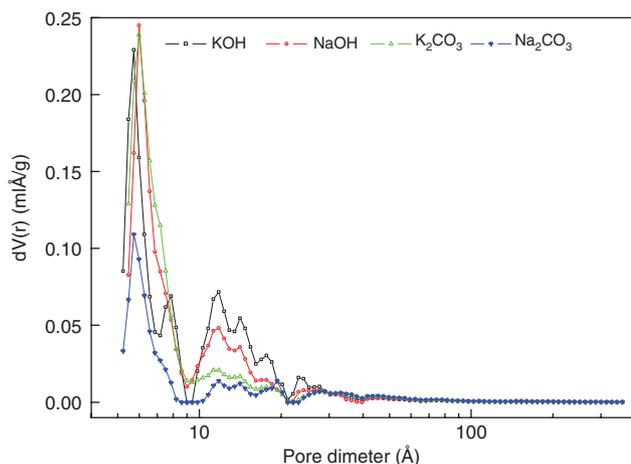
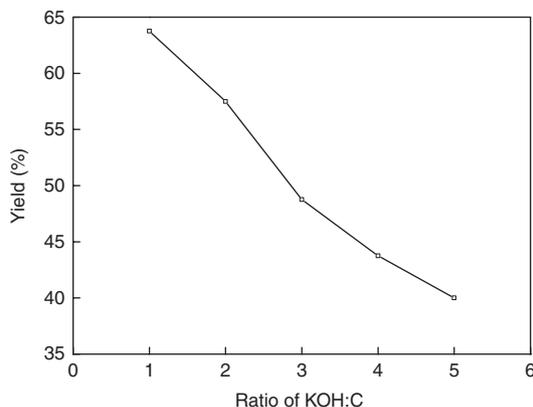
3.1.3 Pore size distribution of activated carbon

According to IUPAC classification [32], pores are divided into micropores (<20 Å), mesopores (20–500 Å) and macropores (>500 Å). Pore size distributions of activated carbon from different activation agents are presented in Figure 4. A distinct variation in the pore size distributions could be observed. The contribution of KOH is distinctly higher throughout, but for the ultramicropore range, both K₂CO₃ and NaOH generate a significantly higher proportion of ultramicropores.

3.2 Effect of KOH/C ratio

3.2.1 The yield of activated carbon

Figure 5 shows the effect of KOH/C mass ratio on the yield of activated carbon. We found that the KOH/C mass ratio has a great effect on the yield of activated carbon, and the yield of activated carbon decreased sharply with increase

**Figure 4:** Pore size distribution of activated carbon with different activation agent.**Figure 5:** Effect of KOH/C mass ratio on the yield of activated carbon.

in KOH/C mass ratio from 1 to 5. With increasing the KOH/C mass ratio, the activation reaction would be strengthened and many more pores would be formed. When the KOH/C mass ratio reached 4, the carbon on the active site has almost completely reacted with KOH and has many pores. The pore would be widened and burnt off when the KOH/C mass ratio over 4.

3.2.2 Pore structure of activated carbon

The nitrogen adsorption isotherms of activated carbon derived from walnut shell char with different KOH/C mass ratios are shown in Figure 6. The N₂ adsorption isotherms of activated carbon belong to the type I. It can be observed that the adsorption volume increases sharply at low relative pressure, which indicates the process of filling the micropores. When the relative pressure reached 0.1, the adsorption amount was about 70% of saturation adsorption, indicating the microporous character. As the relative pressure continued to increase, adsorption increased slowly and the isotherm can be seen as an upwardly convex shape which indicated that adsorption was transitioned from monolayer to multi-molecular layer. The adsorption capacity continued to increase and showed a tailing at a relative pressure close to 1.

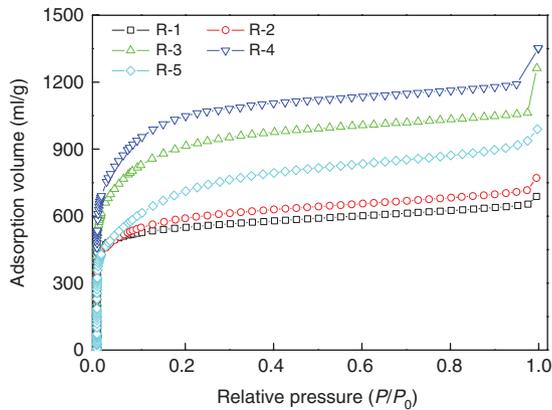


Figure 6: N_2 adsorption isotherm of activated carbon with different KOH/C ratio.

Table 3 summarizes the pore characteristics which showed an increased surface area and total pore volume with increase in the KOH/C mass ratio. An optimal mixture ratio was observed at the KOH/C mass ratio of 4, at which the surface area was $3276 \text{ m}^2/\text{g}$. A drop in surface area as well as pore volume was observed with KOH/C mass ratio beyond 4. The optimal mixture ratio in terms of pore volume and surface area can be attributed to the pore merger phenomena at a higher KOH/C mass ratio.

Table 4 compares the surface area of various activated carbons prepared using different activation agents as reported in literature. Although it was a small sample from the large number of activated carbons reported in

literature, it clearly proved the high surface area of the activated carbon prepared in this work as compared with the literature. A surface as high as $3276 \text{ m}^2/\text{g}$ was very rarely reported in literature which could form the basis for additional work on exploring the combination of walnut shell with the KOH activation agent.

3.2.3 Pore size distribution of activated carbon

Pore size distributions of activated carbon derived from walnut shell with different KOH/C mass ratios are presented in Figure 7. The pore size distributions of activated carbon were concentrated in the micropore field ($5\text{--}7 \text{ \AA}$, $12\text{--}18 \text{ \AA}$). At the range of $10\text{--}20 \text{ \AA}$, the pore size distributions of activated carbon increased with the KOH/C mass ratio increasing, and reached a maximum value at the KOH/C mass ratio of 4, and then decreased with increase in KOH/C mass ratio. The mesopore distributions around $20\text{--}40 \text{ \AA}$ was abundant, especially for a KOH/C mass ratio of 4. Pore volume becomes very small when pore size $>40 \text{ \AA}$. The KOH/C mass ratio of 1 seems to generate a significantly higher proportion of ultramicropores as compared with the other KOH/C mass ratio.

3.3 FTIR analysis

It can be observed from Figure 8 that, irrespective of KOH/C mass ratio utilized the overall shapes of the spectra

Table 3: Pore structure characteristics of activated carbon with different KOH/C mass ratio.

KOH/C mass ratio	S_{BET} (m^2/g)	V_{tot} (ml/g)	D (\AA)	V_{mic} (ml/g)	V_{mes} (ml/g)	S_{mic} (m^2/g)	S_{External} (m^2/g)	$V_{\text{mic}}/V_{\text{tot}}$ (%)
1	1669	1.013	24.3	0.734	0.279	1423	246	72.46
2	1835	1.110	24.2	0.812	0.298	1560	275	73.17
3	2880	1.648	22.9	1.347	0.301	2590	290	81.74
4	3276	1.952	24.0	1.513	0.439	2939	337	77.51
5	2357	1.453	24.7	0.901	0.552	1792	565	62.00

Table 4: Comparison surface area of activated carbon with different activation agent.

Activating agent	Precursor	Heating method	S_{BET} (m^2/g)	References
KOH	Walnut shell	Microwave heating	3276	Present study
	Coconut husk	Microwave heating	1356	[18]
	Isotropic petroleum pitch	Conventional heating	2992	[19]
K_2CO_3	Sisal waste	Conventional heating	1038	[20]
	Edible fungi residue	Microwave heating	684	[21]
$ZnCl_2$	Coffee husk	Conventional heating	1522	[22]
H_3PO_4	Acacia mangium wood	Conventional heating	1040	[23]
	Olive-waste cake	Conventional heating	1020	[24]

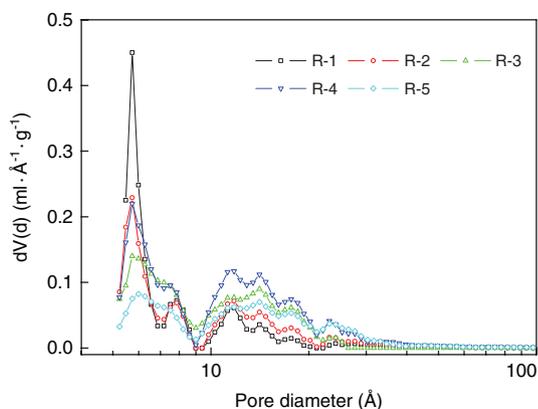


Figure 7: Pore size distribution of activated carbon with different KOH/C mass ratio.

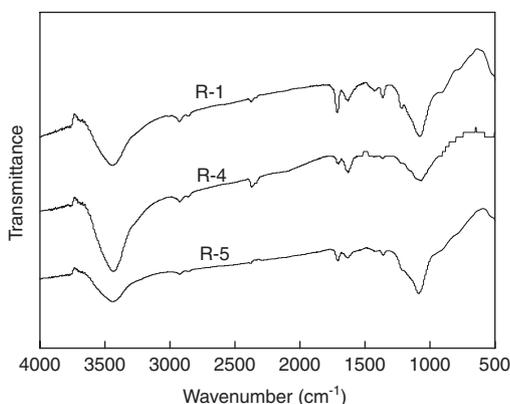


Figure 8: Fourier transform infrared spectroscopy (FTIR) spectra of activated carbon from walnut shell with different KOH/C ratio.

look similar, with the major peaks observed at 3440 cm^{-1} , 2925 cm^{-1} , 2860 cm^{-1} , 1710 cm^{-1} , 1630 cm^{-1} , 1430 cm^{-1} , 1360 cm^{-1} , 1215 cm^{-1} , 1080 cm^{-1} and $920\text{--}790\text{ cm}^{-1}$. An increase in width of the peak was observed with increase in KOH/C mass ratio. The wide peak located at 3340 cm^{-1} was assigned to the O-H stretching vibration. The band at around 2926 cm^{-1} can be assigned to the C-H symmetric and asymmetric vibration mode of methyl and methylene groups. The band at 1710 cm^{-1} indicated that there may exist a C=O functional group and the band at around 1080 cm^{-1} can be assigned to C=C symmetrical stretching of pyrone and the C=O of carboxylic groups. The intensity of the peak was relatively weak in the range 1200 cm^{-1} to 1600 cm^{-1} , which could be attributed to lower oxygen content. The adsorption peak at $920\text{--}790\text{ cm}^{-1}$ was weakened obviously, which means C-H outer surface rocking vibration absorption was weak and carbon network structure was large.

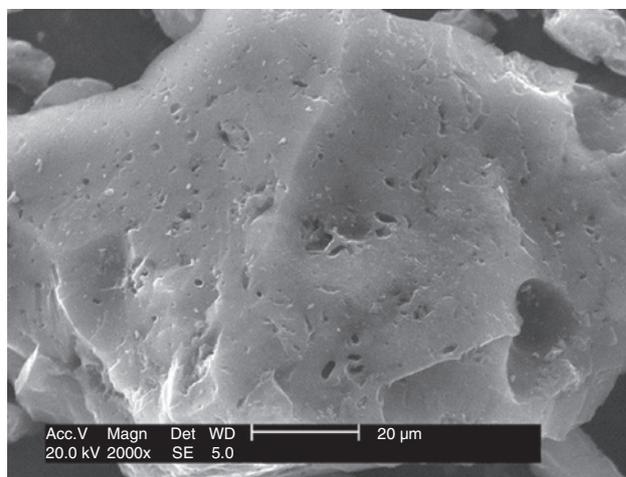


Figure 9: Scanning electron microscopy (SEM) image of activated carbon derived from walnut shell char.

3.4 SEM analysis

Figure 9 shows the SEM image of the high surface area activated carbon derived at a KOH/C mass ratio of 4 from walnut shell char. The surface of activated carbon was smooth and had abundant pores. The pores on the surface could be macropores, which leads to branching micropores in the interiors of the activated carbon. The macropores lead the way to the tributaries which were micropores.

4 Conclusion

Walnut shell, a wasted biomass, was utilized for preparing high surface area activated carbon using microwave heating and exhibited a well-developed pore structure. The results showed that the activation agent had great effects on the preparation of high surface area activated carbon, but KOH is the best activation agent among those activation agents. The effect of KOH/C mass ratio on the yield and pore structures of high surface area activated carbon was investigated and an optimum KOH/C mass ratio of 4 was obtained. The results showed that the highest surface area and total pore volume of activated carbon were $3276\text{ m}^2/\text{g}$ and 1.952 ml/g , respectively, under the KOH activation and KOH/C mass ratio of 4. The characterization of FTIR illustrated that surface of activated carbon had abundant functional groups. SEM showed that activated carbon has a developed pore structure.

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