



Preparation of high surface area activated carbon from coconut shells using microwave heating

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ABSTRACT

The present study attempts to utilize coconut shell to prepare activated carbon using agents such as steam, CO₂ and a mixture of steam-CO₂ with microwave heating. Experimental results show that the BET surface area of activated carbons irrespective of the activation agent resulted in surface area in excess of 2000 m²/g. The activation time using microwave heating is very much shorter, while the yield of the activated carbon compares well with the conventional heating methods. The activated carbon prepared using CO₂ activation has the largest BET surface area, however the activation time is approximately 2.5 times higher than the activation using steam or mixture of steam-CO₂. The chemical structure of activated carbons examined using Fourier transformed infra-red spectra (FTIR) did not show any variation in the surface functional groups of the activated carbon prepared using different activation agents.

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1. Introduction

Activated carbons with highly developed surface area are widely used in a variety of industries for applications which include separation/purification of liquids and gases, removal of toxic substances, as catalysts and catalyst support (Moon and Shim, 2006; Fuente et al., 2001). With the development of technology, the applications of activated carbons keep expanding, with newer applications such as super-capacitors, electrodes, gas storage, and so on (Yuan and Zhang, 2006; Oda and Nakagawa, 2003; Biloe et al., 2002). Activated carbons have been traditionally produced by the partial gasification of the char either with steam or CO₂ or a combination of both. The gasification reaction results in removal of carbon atoms and in the process simultaneously produce a wide range of pores (predominantly micropores), resulting in porous activated carbon. Precursors to activated carbons are either of botanical origin (e.g. wood, coconut shells and nut shells) or of degraded and coalified plant matter (e.g. peat, lignite and all ranks of coal). Agricultural by-products are considered as very important feedstock as they are renewable and low-cost materials (Ioannidou and Zabaniotou, 2007; González et al., 2006).

In general, there are two main steps for the preparation of activated carbon: (1) the carbonization of carbonaceous precursor be-

low 800 °C, in the absence of oxygen, and (2) the activation of carbonized product (char), either using physical or chemical activation methods. Generally physical activation is a two-step process which involves carbonization of a carbonaceous material followed by activation of the resulting char at elevated temperature in presence of suitable oxidizing gases such as carbon dioxide, steam, air or their mixtures. In the chemical activation process the precursors are impregnated with dehydrating chemicals such as H₃PO₄, ZnCl₂, K₂CO₃, NaOH or KOH and carbonized at desired conditions in a single step. Chemical activation offers several advantages which include single step activation, low activation temperatures, low activation time, higher yields and better porous structure. However the process involves a complex recovery and recycle of the activating agent, which generates liquid discharge that demands effluent treatment. Physical activation process is widely adopted industrially for commercial production owing to the simplicity of process and the ability to produce activated carbons with well developed micro porosity and desirable physical characteristics such as the good physical strength.

The conventional heating methods results in surface heating from the hearth wall, which do not ensure a uniform temperature for different shapes and sizes of samples. This generates a temperature gradient from the hot surface of the sample particle to its interior and impedes the effective removal of gaseous products to its surroundings, thereby resulting in long activation time and higher energy consumption. Recently, microwave heating is being used in various technological and scientific fields for variety of

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applications. The main difference between microwave heating and conventional heating systems is in the way the heat is generated. Energy transfer is not by conduction or conventional heating, but is readily transformed into heat inside the particles by dipole rotation and ionic conduction. When high frequency voltages are applied to a material, the response of the molecules with a permanent dipole to the applied potential field is to change their orientation in the direction opposite to that of the applied field. The synchronized agitation of molecules then generates heat (Ania et al., 2005; Nabais et al., 2004; Jones et al., 2002). Therefore, the tremendous temperature gradient from the interior of the char particle to its cool surface allows the microwave induced reaction to proceed more quickly and effectively, resulting in energy saving and shorter reaction time.

Although microwave heating has been used to produce and regenerate activated carbon, the relevant literature is very limited. The studies pertaining to preparation of activated carbon using physical activation has been limited to Guo and Lua (2000) and Williams and Parkes (2008), while the regeneration studies were limited to Ania et al. (2005), Ania et al. (2004) and Coss and Cha (2000). Nabais et al., (2004) have reported the surface chemistry modification of activated carbon fibers by means of microwave heating. None of the previous attempts have reported comparison of activated carbon prepared with steam, CO₂ and a mixture of steam-CO₂ by microwave heating for coconut shells. The present study attempts to investigate the effect of activation time on the yield and porous structure of activated carbons produced from coconut shells employing different activation agents such as steam, CO₂ and combination of steam-CO₂.

2. Experiment

2.1. Materials

Coconut shells utilized in the present study were obtained from Xishuangbanna in Yunnan province of China. The starting materials were manually chosen, cleaned with deionized water, dried at 110 °C for 48 h and ground using a roller mill and sieved to a size range of 3.35–4.75 mm and stored in sealed containers for experimentation. The proximate analysis of the coconut shell is shown in Table 1.

2.2. Preparation of activated carbons

2.2.1. Carbonization of coconut shells

Carbonization was performed in a horizontal tube furnace by electric heating and coconut shells were placed inside a stainless steel reactor. The coconut shells were heated up to a carbonization temperature of 1000 °C at a heating rate of 10 °C/min with conventional heating and were held for 2 h at the carbonization temperature under N₂ gas flow (100 cm³/min). After carbonization, the samples were cooled to room temperature under N₂ flow (100 cm³/min).

2.2.2. Activation procedure

The activation experiments were carried out in a self-made microwave tubular furnace, which has a single-mode continuous controllable power and is shown in Fig. 1. The microwave fre-



Fig. 1. Microwave reactor of multi-mode with continuous controllable power.

quency was 2.45 GHz, while the output power could be set to a maximum of 3000 W. The temperature of sample in the microwave equipment was monitored using a type of K (chromel–alumel) thermocouple, placed at closest proximity to the sample.

Approximately 25 g of the pre carbonized material was placed into reactor and set to the desired temperature along with the N₂ flow rate at 200 cm³/min. Upon reaching the desired temperature the gasifying agent was allowed into the reactor at a desired flow rate. Approximately 5 to 7 min of time was required to raise the temperature of sample to the desired temperature of 900 °C. Three experimental series were conducted employing the flowing conditions,

- A) Steam activation (1.35 g/min): to assess the effect of activation time at 900 °C;
- B) CO₂ activation (600 cm³/min): to assess the effect of activation time at 900 °C;
- C) CO₂-steam mixture activation (600 cm³/min + 1.35 g/min): to assess the effect of activation time at 900 °C.

Selected sets of experiments were repeated to ensure the reproducibility of experimental data.

2.3. Structure characterization

The pore structure of the sample is characterized by nitrogen adsorption at 77 K with an accelerated surface area and porosimetry system (Autosorb-1-C, Quantachrome). Prior to gas adsorption measurements, the carbon was degassed at 300 °C in a vacuum condition for a period of at least 2 h. Nitrogen adsorption isotherm was measured over a relative pressure (P/P₀) range from approximately 10^{−7} to 1. The BET surface area was calculated from the isotherms by using the Brunauer–Emmett–Teller (BET) equation (Gregg and Sing, 1982). The cross-sectional area for nitrogen molecule was assumed to be 0.162 nm. The Dubinin–Radushkevich (DR) method was used to calculate the micropore volume (Dubinin, 1975). The micropore size distribution was ascertained by Non-local Density Functional Theory (NLDFT) (Lastoskie et al., 1994) by minimizing the grand potential as a function of the fluid density profile. The total volume (Gregg and Sing, 1982) was estimated by converting the amount of N₂ gas adsorbed at a relative pressure of 0.95 to equivalent liquid volume of the adsorbate (N₂). The mesopore volume was estimated by the subtracting the micropore volume from the total volume.

Table 1
Proximate analysis results of coconut shell.

Sample	Moisture (wt.%)	Volatile matter (wt.%)	Fixed carbon (wt.%)	Ash (wt.%)
Coconut shells	10.53	70.06	18.75	0.66

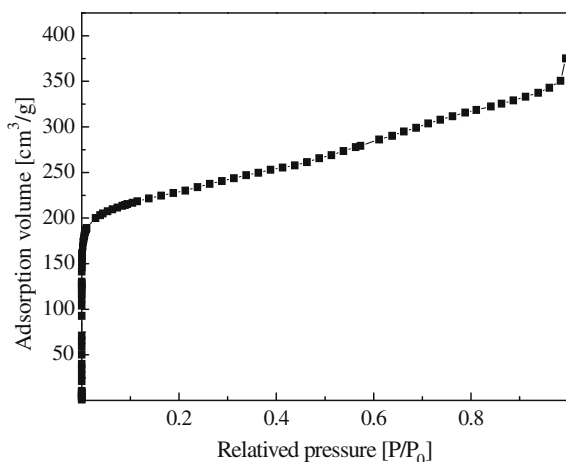


Fig. 2. N₂ isotherm of coconut shell chars prepared at temperature 1000 °C.

3. Results and discussion

3.1. Characteristics of porosity in coconut shell chars

The carbonization process prior to activation enriches the carbon content with removal of volatile matter which creates the initial porosity in the char. The pore structure of the char was estimated using the nitrogen adsorption isotherm and is shown in Fig. 2. The total BET surface area and the pore volume distribution is listed in Table 2. The nitrogen adsorption isotherm (Fig. 2) of coconut shell chars correspond an intermediate between type I and II of the referred IUPAC classification (Rouquerol et al., 1999). A BET surface area of 702 m²/g is significant, with micro pore volume accounting to 66%. This type of isotherm is usually exhibited by microporous solids containing a well developed mesopore structure. This indicates that chars produced at 1000 °C offer higher potential to produce activated carbon of greater adsorption capacity. The porosity is generated due to the conversion of hemicellulose, cellulose and lignin in coconut shells by the process of dehydrating, linkage breaking reactions, the structural ordering process of the residual carbon and finally polymerization reaction (Byrne and Nagle, 1997). At a carbonization temperature of 1000 °C, a significantly higher rate of polymerization reaction results in faster size reduction of the sample contributing to the formation of micropores in the samples (Cagnon et al., 2003; Inagaki et al., 2004; Tan and Ani, 2003). The high porous char produced at 1000 °C forms a good basis for further enhancing the porous nature of the char, by promoting the reaction between the char and the reacting agents such as steam and CO₂ (Li et al., 2008).

3.2. Characteristics of activated carbons

3.2.1. Effects of activation agent and activation time on yield of activated carbons

The yield of activated carbon is an important factor in the process of activated carbon preparation as it has direct bearing on the process economics. The yield is defined as the % ratio of weight of activated carbon produced to the weight of carbonized char uti-

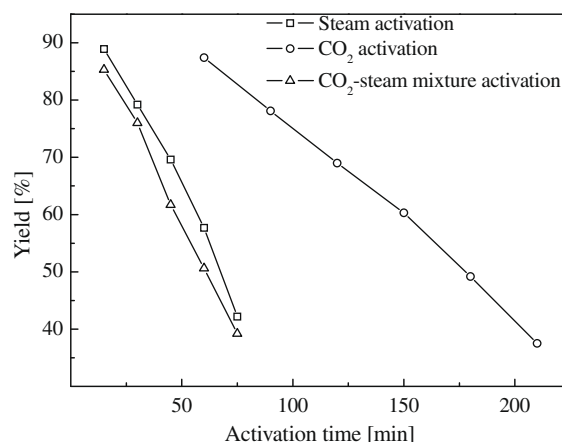


Fig. 3. Effects of activation agent and hold time on yield of activated carbons.

lized for activation. It should be noted that the yield in the process of conversion of coconut shell into carbonized char is 22%. Fig. 3 illustrates the relationship between the activation time and the yield of activated carbon corresponding to different activation agent at an activation temperature of 900 °C. It can be seen that the yield of activated carbon decreases progressively with the activation time which could be attributed to the reaction between carbon and activation agent. The rate of reduction of yield with respect to activation time is the lowest for the CO₂ activation, while it is an order of magnitude higher using steam activation. The rate of reaction of the steam with carbon is well established to be higher than the CO₂ by several other earlier reported works (González et al., 2006; Román et al., 2008) which accounts for the faster reduction in yield using steam activation as compared to CO₂ activation. However reduction in yield for combination of mixture of steam and CO₂ was found to be higher than steam alone, which could be attributed the higher net flow of the activation agent and occurrence of simultaneous reaction of steam and CO₂ with carbon.

3.2.2. N₂ Isotherms of activated carbon

It is well known that the adsorption capacity of an adsorbent largely depends on the amount of micropores and surface area. The activation process develops porosity in the carbon by creating a more orderly porous structure. There are usually four stages in the pore development during the activation process: (i) opening of previously inaccessible pores; (ii) creation of new pores by selective activation; (iii) widening of the existing pores and (iv) merger of the existing pores due to pore wall breakage (Rodríguez-Reinoso, 1991).

The most popular and widely followed method utilized to estimate the porous nature of adsorbents is based on the nitrogen adsorption isotherm. Fig. 4 shows the nitrogen adsorption isotherms exhibited by activated carbons prepared at 900 °C for different activation time with steam, CO₂ and CO₂-steam mixture by microwave heating. It can be ascertained from Fig. 4 that the isotherms of all activated carbons pertain to intermediate between type I and II of the referred IUPAC classification (Rouquerol et al., 1999). This type of isotherm is usually exhibited by microporous solids that include a well developed mesopore structure. An increase in the activation time produces a positive effect on the amount of nitrogen adsorbed indicating development of additional pores in the activated carbon. From the micropore and mesopore volumes provided in Table 3, it can be inferred that the mesopores account approximately about 20% of the total pore volume irrespective of the activating agent for carbons with well developed porous structure.

Table 2
Characteristics of porosity in coconut shell chars prepared at temperature 1000 °C.

Sample	BET surface area (m ² /g)	V _{tot} (cm ³ /g)	V _{micro} (cm ³ /g)	V _{meso} (cm ³ /g)
Coconut shell chars	702	0.5319	0.3482	0.1837

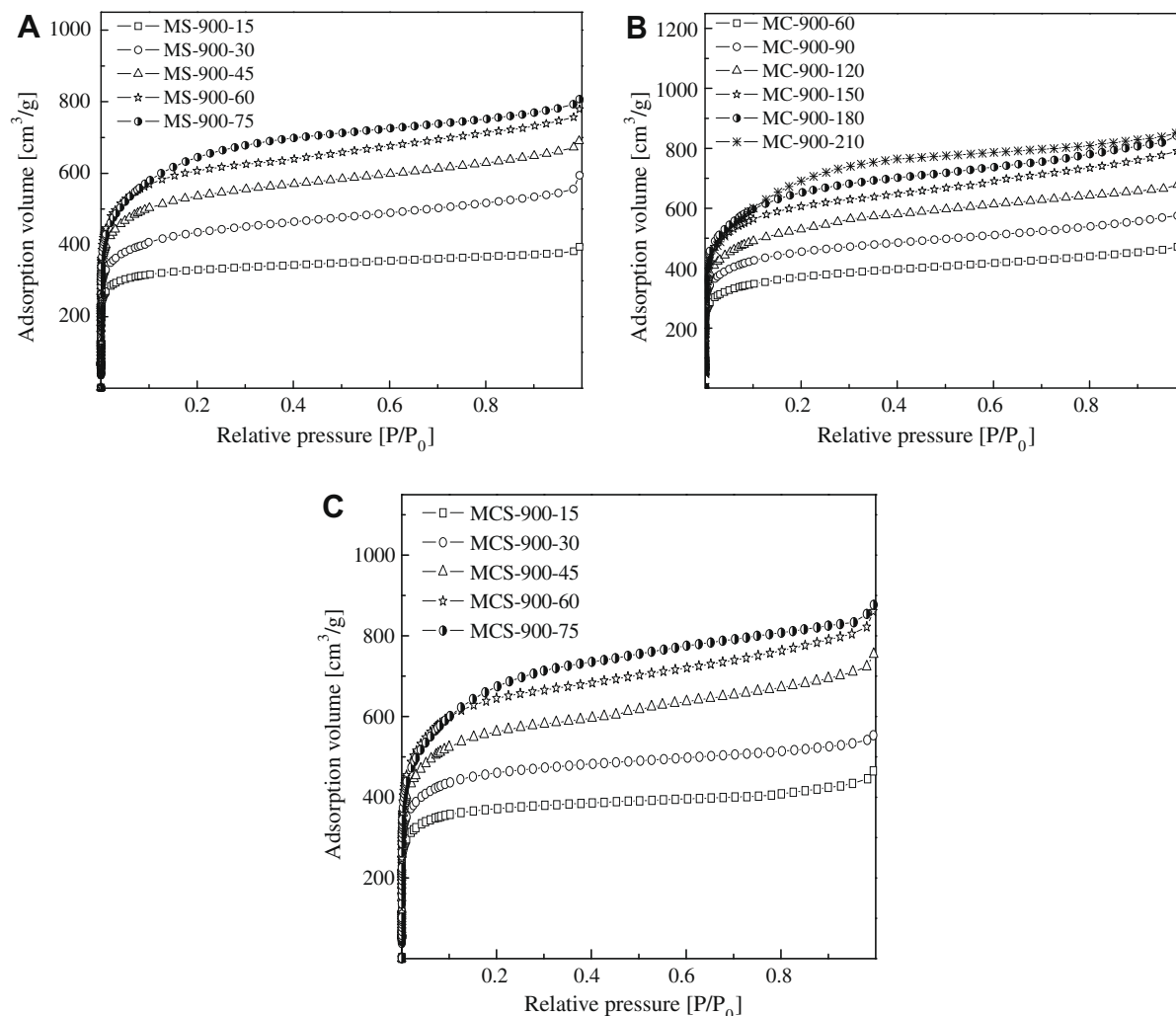


Fig. 4. N_2 isotherms of activated carbon using different activation agent under different hold time at activation temperature of 900 °C (activation agents: (A) steam activation; (B) CO_2 activation; (C): CO_2 -steam mixture activation).

A comparison of the nitrogen isotherms of activated carbon with different activation agent reveal that activation using CO_2 -

steam mixture produces a higher nitrogen adsorption capacity compared to activation using CO_2 or steam, at identical activation times. It should however be noted that the total flow rate of the mixture of CO_2 -steam is much higher than the individual flow of either CO_2 or steam. The increased porosity using CO_2 -steam mixture could possibly be due to higher reactivity of steam which generates and widens micropores at a faster rate, which aids the diffusion of CO_2 to the interior parts of the carbon contributing to additional development of micropores. Further the higher net flow of activation agent could possibly enhance the reaction rate of the steam and CO_2 with carbon. However the nitrogen adsorption capacity of the activated carbon prepared using CO_2 activation has the highest surface area but at a much higher activation time, owing to the lower rate of reaction of CO_2 with carbon. It can be concluded that the CO_2 based activation demands a higher activation time to the order of 2.5 times as compared to steam or combination of steam- CO_2 to produce activated carbon with comparable porous nature.

Table 3 lists the BET surface area, total pore volume, micropore volume and mesopore volume of activated carbon corresponding to various activation agents at different activation times. It can be observed from the data that BET surface area and pore volume increases with increase in the activation time irrespective of the activation agent. The magnitude of increase in porosity reduces at higher activation time, possibly indicating proximity to the opti-

Table 3
Characteristics of porosity of activated carbons prepared with different activation agent.

Sample	SBET (m^2/g)	Vtot (cm^3/g)	Vmicro (cm^3/g)	V meso (cm^3/g)
MS-900-15	1011	0.585	0.5179	0.0671
MS-900-30	1363	0.8454	0.6727	0.1727
MS-900-45	1677	1.0250	0.8339	0.1911
MS-900-60	1888	1.1570	0.9483	0.2087
MS-900-75	2079	1.2120	0.9735	0.2385
MC-900-60	1162	0.7159	0.5703	0.1456
MC-900-90	1425	0.8820	0.7022	0.1798
MC-900-120	1703	1.0320	0.8153	0.2167
MC-900-150	1905	1.204	0.9365	0.2675
MC-900-180	2080	1.2700	0.9974	0.2726
MC-900-210	2288	1.2990	1.0120	0.287
MCS-900-15	1139	0.6911	0.5869	0.1042
MCS-900-30	1424	0.8276	0.7229	0.1047
MCS-900-45	1761	1.1020	0.8773	0.2247
MCS-900-60	2020	1.2480	1.0080	0.2400
MCS-900-75	2194	1.2930	1.0100	0.2830

Note: M-microwave heating; S-steam activation; C- CO_2 activation; CS- CO_2 + steam activation; 900-activation temperature(°C); 15~210-activation time (min).

imum activation time. The improvement in porous structure coupled with decrease in yield of the activated carbon, with increase in the activation time indicates the increase in extent of the reaction between the activation agent and carbon. The increased extent of the reaction aids enhancement and generation of new pores in the carbon resulting in higher surface area and pore volume. A BET surface area of activated carbon in excess of 2000 m²/g could be produced with all the three different combinations of activation agents, however with different activation times.

A comparison of the pore characteristics of the activated carbons due to the present study with other literature reported values for the coconut shell based precursor, under optimum activation conditions are listed in Table 4. It can be observed from the data provided in table that the BET surface area due to the present study is much higher than those of reported using physical activation. The yield of activated carbon well compares with the conventional heating methods reported in literature. A yield of 40%, with high surface area of 2200 m²/g could be attributed to the higher carbonization temperature (1000 °C) than the conventional carbonization temperatures (550–700 °C) (Singh et al., 2008; Sarkar and Bose, 1997) and to the microwave heating. At high carbonization temperatures, a more ordered structure is likely to be developed in the char that leads to a slower rate of gasification in the interior of the particle (Li et al., 2008; Rodríguez-Reinoso, 1991).

The activation time due to present work is much shorter owing to the thermal efficiency of microwave heating system. The uniform, faster heating rate due to microwave heating increases the reaction rate between the carbon and the activation agent, which leads to more active sites taking part in reaction. It can be concluded that the higher carbonization temperature of 1000 °C coupled with microwave heating plays a key role for the enhanced porous nature of the carbon and for higher yields. The micropore volume of the activated carbon corresponding to high surface area samples contributes more than 80%. Activated carbons prepared from char for other precursors at high carbonization temperature have been reported to have higher micropore volume. These precursors include almond shell (Rodríguez-Reinoso, 1991), prune pit (Gergova et al., 1993), olive stone (Gonzalez et al., 1970) and palm shell (Daud et al., 2000).

3.2.3. Pore size distributions of activated carbons

Fig. 5A shows the pore size distributions of activated carbons prepared with steam, CO₂ and CO₂-steam mixture corresponding to different degrees of activation. The degree of activation indicated in % corresponds to the yield of activated carbon. Label “Steam 88.9%” indicate the pore size distribution of the activated carbon sample with 88.9% yield or in other words the activated carbon produced at low activation time resulting in high yield. At

Table 4

Comparison of the characteristics of porosity in activated carbons of present work with other literature data under optimum conditions.

References	Activation and heating method	Activation time (min)	S _{BET} (m ² /g)	V _{tot} (cm ³ /g)	V _{micro} (cm ³ /g)	Yield (%)
Present work	Physical activation (steam), Microwave heating	75	2079	1.212	0.9735	42.2
	Physical activation (CO ₂), Microwave heating	210	2288	1.299	1.012	37.5
	Physical activation (CO ₂ + steam), Microwave heating	75	2194	1.293	1.010	39.2
Li et al. (2008)	Physical activation (steam), Conventional heating	120	1926	1.260	0.931	39.1
Singh et al. (2008)	Physical (inter atmosphere), Conventional heating	60	378	0.26	0.12	
Sarkar and Bose (1997)	Physical activation (steam), Conventional heating	120	1018		0.4008	
Su et al. (2003)	Physical (without activation agent), Conventional heating		663	0.23		23.2
Achaw and Afrane (2008)	Physical activation (35%steam, 65%N ₂), Conventional heating	120	524	0.226	0.210	
Cagnon et al. (2009)	Physical activation (N ₂ + H ₂ O), Conventional heating	120		0.39	0.35	76.3
Cagnon et al. (2003)	Physical activation (N ₂ + H ₂ O), Conventional heating	210			0.52	44.5
Su et al. (2007)	Physical activation (CO ₂), Conventional heating	2880	1964	0.9812		
Din et al. (2009)	Chemical and physical activation (with KOH followed by CO ₂ activation), Conventional heating	120	1026	0.5768		
Azevedo et al. (2007)	Chemical activation (ZnCl ₂), Conventional heating		1266	0.731	0.676	
	Chemical and physical activation (with ZnCl ₂ followed by physical activation), Conventional heating		2114	1.307	1.142	
Hu and Srinivasan, (1999)	Chemical (KOH), conventional heating	120	2451	1.210		23.6

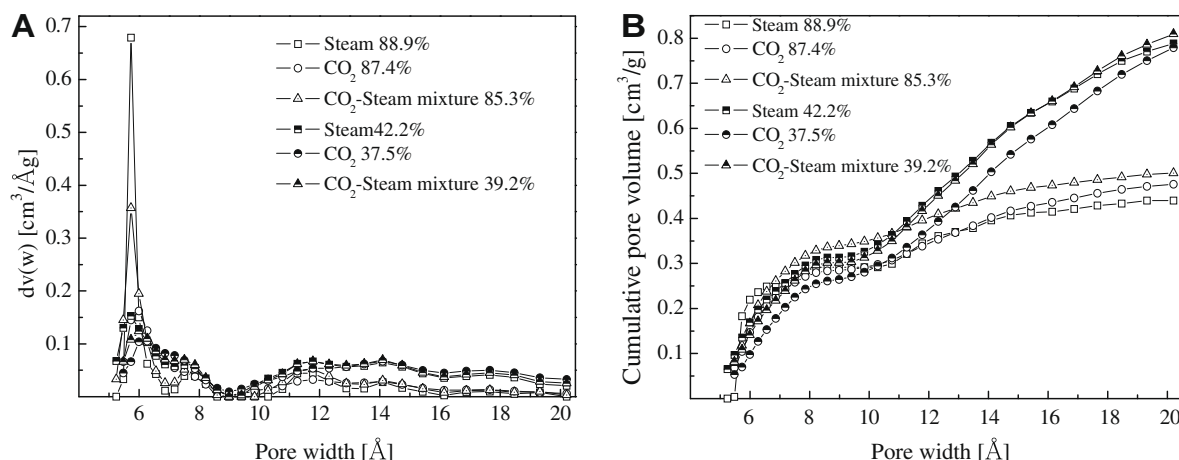


Fig. 5. Pore size distribution of activated carbons prepared under 900 °C with steam, CO₂ and CO₂-steam mixture activation by microwave heating method ((A) Pore size distribution; (B) Cumulative pore volume distribution).

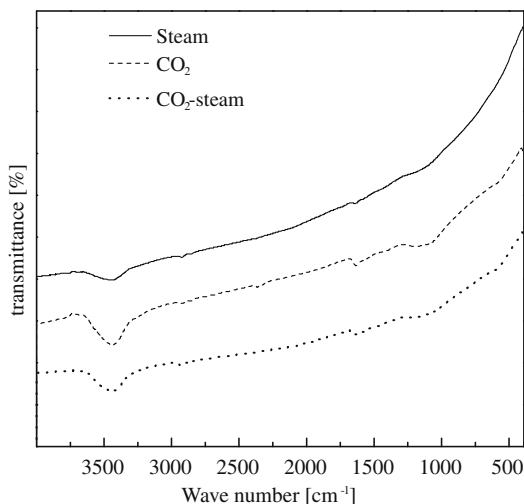


Fig. 6. FTIR spectra of the activated carbons prepared with different activation agent by microwave heating.

low degrees of activation (Fig. 5A), the pore development is mainly in the ultra micropore (5–7 Å) range with the steam having higher degree of pore development. The quantum of pore development using steam-CO₂ is one half as compared to steam alone. Similarly, the pore development using CO₂ is one half as compared to the steam-CO₂ mixture. This is well in agreement with the low rate of activation of carbon-CO₂ reaction in comparison with the carbon-steam reaction. The quantum of pores higher than ultra micropores is insignificant at lower degree of activation, evidenced from Fig. 5B which do not show significant rise in the cumulative pore volume beyond the ultra micropore range, in addition, non existence of pores beyond a pore size of 14 Å is insignificant. The comparison of the pore development with degree of activation well indicates the formation of micropores initially, which progressively widens with increase in the degrees of activation. At higher degrees of activation, the higher total pore volume is due to the continued generation of the ultra micropores and widening the existing pores progressively in tune with the extent of the carbon conversion. The difference in the quantum of ultra micropores is marginal at higher degree of activation among the different activation agents considering the variation in the degree of activation in the data presented in Fig. 5A. These observations are in concurrence with the earlier reports due to Arenas and Chejine (Arenas and Chejine, 2004).

3.3. FTIR analysis

The Fourier transformed infra-red spectra of the activated carbons prepared using microwave heating with different activation agent is shown in Fig. 6. The FTIR spectroscopy provides information on the chemical structure of the materials. It can be observed from the figure that irrespective of the activation agent utilized the overall shapes of the spectra are very similar. The band at around 3430 cm⁻¹ can be assigned to the O–H stretching vibration mode of hydroxyl functional groups, while the band at around 2917 cm⁻¹ can be assigned to the C–H symmetric and asymmetric vibration mode of methyl and methylene groups. The band at around 1631 cm⁻¹ can be assigned to C = C stretching vibration mode of olefinic C = C bonds (Nakanishi, 1962).

4. Conclusion

Coconut shell is the most popular and industrially widely adopted precursor for preparation of activated carbon. The present

study utilizes coconut shell to prepare activated carbon using different activating agents such as steam, CO₂ and combination of steam-CO₂ with microwave heating, in order to compare the porous structure and yield of activated carbon. A two-stage activation process with carbonization at 1000 °C under N₂ atmosphere with conventional heating followed by activation at 900 °C with microwave heating has been utilized for preparation of activated carbon. The BET surface area of the activated carbons irrespective of the activation agent resulted in surface area in excess of 2000 m²/g. The activation time using microwave heating is very much shorter, while the yield of the activated carbon well compares with the conventional heating methods. The activated carbons are primarily micropores with the mesopores contributing to 20% of the pore volume, for activated carbons with well developed pores. The process of pore formation is faster with steam as the activating agent compared to CO₂ owing to the higher rate of reaction of steam with carbon. The ultra micropores formed initially activation which continues to widen to higher pore diameter in tune with the extent of the reaction. The activated carbon prepared using CO₂ activation has the largest BET surface area; however the activation time is approximately 2.5 times higher than the activation using steam or mixture of steam-CO₂. The chemical structure of activated carbons examined using Fourier transformed infra-red spectra did not show any variation in the surface functional groups of the activated carbon prepared using different activation agents.

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