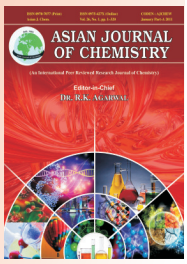
Asian Journal of Chemistry; Vol. 26, No. 16 (2014), 5295-5297

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2014.17449



Ramp Rate Influence on Synthesis of Sliced Porous Activated Carbon from Date Palm Tree by Physical Activation Method

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Received: 24 February 2014; *Accepted*: 30 April 2014; *Published online*: 28 July 2014; AJC-15681

Saudi Arabia is considered as a mass date producer all over the date producing countries around the world. Extensive amount of waste material is available in the form of date fronds because of the pruning process. Date fronds as a biomass is considered as a precursor for activated carbon production. The BET surface areas of the activated carbons prepared at a ramp rate of 5, 10, 20, 30 and 40 °C/min after 0.5 h activation time are 818, 1094, 1020, 565 and 784 m^2/g, respectively. The activated carbon prepared at a ramp rate of 10 °C/min attains larger surface area 1094 m^2/gm and can offer higher potential to produce activated carbon of greater adsorption capacity from bio mass such as date fronds. Supporting techniques like scanning electron microscopy verifies the pore generation too. Moreover by increasing the ramp rate from 10 and 20 °C/min the yield remains same *i.e.*, 18.75 % whereas at a ramp rate of 40 °C/min the yield decreases from 20.3 to 16 %.

Keywords: Activated carbon, Date palm tree fronds, Physical activation, Porous carbon.

INTRODUCTION

Kingdom of Saudi Arabia produced 15 % of the overall date's production of the world and this value is considered as a massive value[1]. Pruning of date trees are considered as an ideal process to maximize the production along with the quality[2]. Tons of the bio mass material by the pruning process in the form of fronds is available throughout the Saudi Arabia and there is a need of proper disposal of the bio mass. As the biomass contains high carbon contents of lignin, lignocelluloses[3,4] therefore the selection of date fronds for the production of activated carbon ultimately help the kingdom towards cleaner environment and low cost absorbent materials for many applications like adsorption of noxious emissions, heavy metals adsorption, removal of dyes, H_2S and odor, *etc.*[5-11].

Variety of lignocelluloseic biomass has already been reported for the synthesis of activated carbon with enhanced adsorption capacity, such as cotton stalks[12], wood[13], hazelnut shell[14-16], durian shell[17-20], walnut shell[21], plum stones[22], coconut shells[23,24], palm kernel shell[25-29] and rubber seed shell[30].

Two methods have been reported for synthesis of activated carbon by physical and chemical methods. In this study physical method a single step procedure is taken into consideration due to its ease compare to chemical activation as it involves complex steps like filtration, impregnation along with the time consumption.

During this study, a single step physical method was used carbonization of the biomass in an inert atmosphere *i.e.*, grade 5 nitrogen, followed by the regulated gasification at a fix temperature of 850 °C with a mixture of carbon dioxide and nitrogen with varying ramp rate from 5, 10, 20, 30, 40 °C/min to get the end product with required porosity.

EXPERIMENTAL

For the present synthesis study the ramp rates of 5, 10, 20, 30 and 40 °C/min are considered using grade 5 nitrogen (N_2) for carbonization followed by activation using the mixture of grade 5 nitrogen and carbon dioxide (N_2 and CO_2) at 850 °C. Pressure within the reactor is maintained at 0.25 bar and the excessive pressure is released with the help of simple electronics using solenoid valve and a pressure switch.The detailed procedure are as follows: Fronds initially washed with water followed by drying, the dried fronds are then cut in to slices of 2-3 mm with the help of precision wood cutter. 4 g of sliced precursor in high alumina sample tray (90 mm) is then placed in a carbolyte MTF 12/38/250 tube furnace for carbonization under grade 5 nitrogen followed by the activation using CO_2 and N_2 gases at temperature 850 °C. Flow rate of nitrogen during carbonization is kept at 150 mL/min wheras during activation flow rate of nitrogen and carbon dioxide are kept at 150 and 50 mL/min, respectively. The activation dwell time is fixed at 0.5 h with variying ramp rate from 5, 10, 20, 30, 40 °C/min

for all the five samples. The synthesized activated carbon was characterized by calculating Brunauer-emmett-teller (BET) surface area, pore size (PS), pore volume (PV), scanning electron microscope (SEM). Fig. 1 shows the scheme of the process.

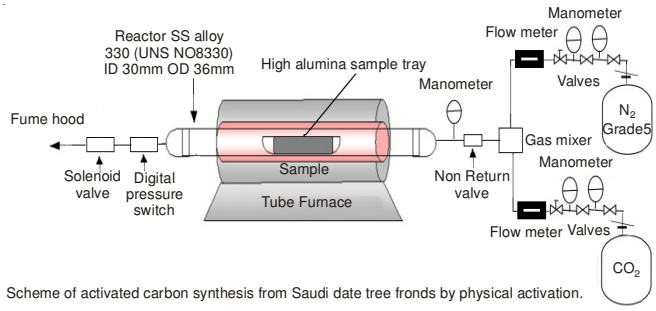

Fig. 1. Scheme of process

RESULTS AND DISCUSSION

Brunauer-Emmett-Teller surface area, pore size and pore volume: Brunauer-Emmett-Teller surface areas, pore size and pore volume of the activated carbons were determined by N_2 gas adsorption at 77 K with an automated adsorption instrument (GEMINI VII, 2390 Micromeritics). Prior to the determination, about 0.02 g of sample degassed for 1 h at 150 °C under nitrogen to remove moisture and other volatiles from the sample. The BET surface area of activated carbon prepared at single activation temperatures (850 °C) is shown in Table-1. The highest surface area 1094 m^2g^{-1} and the pore volume 0.4382 $cm^3\ g^{-1}$ achieved for activated carbon prepared at 850 °C is obtained at a ramp of 10 °C/min. 18.75 % constant yield is observed at a ramp of 10 and 20 °C/min wheras an increase in yield from 18.75 to 20 % was observed at a ramp of 30 °C/min.

TABLE-1
SURFACE AREA, PORE VOLUME, PORE SIZE AND YIELD OF ACTIVATED CARBON

S. No.	Sample name	Surface area ($m^2\ g^{-1}$)	Pore volume ($cm^3\ g^{-1}$)	Pore size (Å)	Yield (%)
1	Raw Date Frond	2	–	–	–
2	SAC^a-850^b-5^c-30^d	818	0.3525	17.23	20.30
3	SAC^a-850^b-10^c-30^d	1094	0.4382	16.09	18.75
4	SAC^a-850^b-20^c-30^d	1020	0.4181	16.38	18.75
5	SAC^a-850^b-30^c-30^d	565	0.2423	17.14	20.00
6	SAC^a-850^b-40^c-30^d	784	0.3308	16.99	16.00

Note: a-b-c-d denotes sliced activated carbon-activation temperature (°C)- heating ramp rate (°C/min)-activation dwell time (min)

Increasing the heating ramp rate from 5 to 10 °C/min increases the BET surface area where as further increase from 10 to 30 °C a linear decreasing trend was observed again the increase in surface area was observed at a ramp rate of 40 °C/min. A higher heating ramp rate would transform into a shorter contact time between the char and the CO_2 gas during the temperature ramp-up stage. This can be perceived by collating the activated carbons subjected to heating ramp rates of 10 and 20 °C/min; both activated carbons have nearly the same pore surface area but the activated carbon at 10 °C/min has 276 m^2/g more BET surface area than that for 5 °C/min, 529 m^2/g more than 30 °C/min and 310 m^2/g more than 40 °C/min. Hence, the activated carbon prepared at a heating ramp rate of 10 °C/min has a larger pore surface area than that of the other ramp rates.The effects of heating ramp rate on the surface area, pore volume and pore size of the activated carbons are shown in Figs. 2 and 3, respectively.

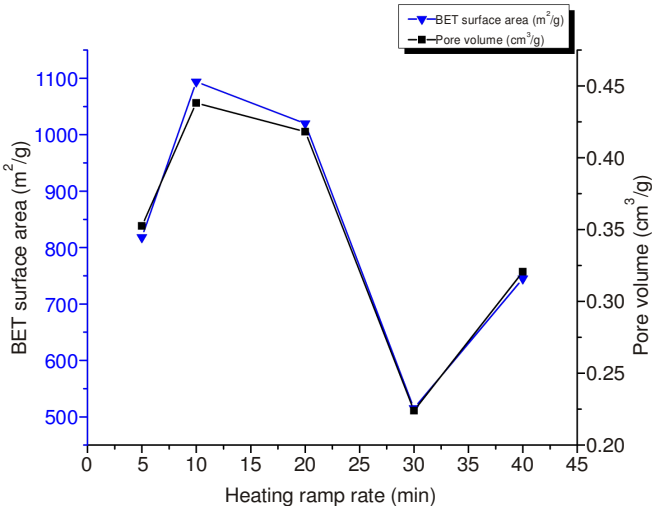

Fig. 2. Effect of heating ramp rate on BET surface area and pore volume

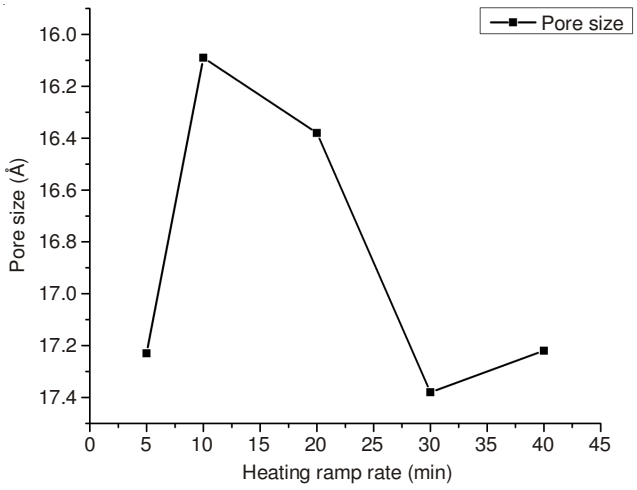

Fig. 3. Effect of heating ramp rate on pore size

Scanning electron microscope analysis: Scanning electron microscope analysis was carried out by using Jeol JSM-6380 LA instrument. Fig. 4 shows the scanning electron microscope micrographs for the raw date fronds in which the surface is curvy due to cellulose, hemicelluloses and lignin and with less slit like fractures or cracks. The micrograph in Fig. 5 is obtained at 1000 × magnification and 10 µm. In Fig. 5, SAC^a-850^b-10^c-30^d prepared at the activated temperature of 850 °C pores growth can easily be visualized.

Conclusion

Mainly mesoporous activated carbon prepared at a ramp of 10 °C/min has a larger surface area and has a larger adsorption capacity compare to other ramp rates. Moreover the pressure swith and solenoid valve helps to attain the reproducibility of results by maintaing the pressure inside the vessel at 0.25 bar.

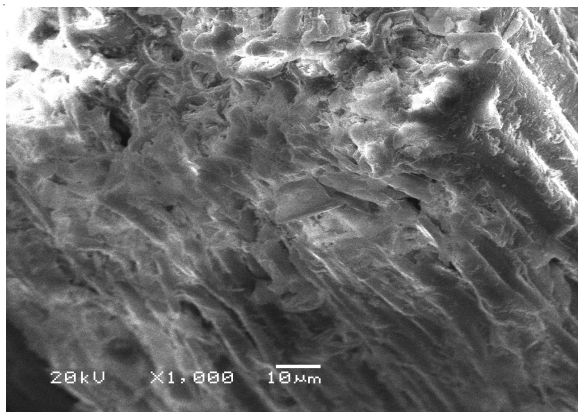

Fig. 4. Sliced precursor date palm tree frond

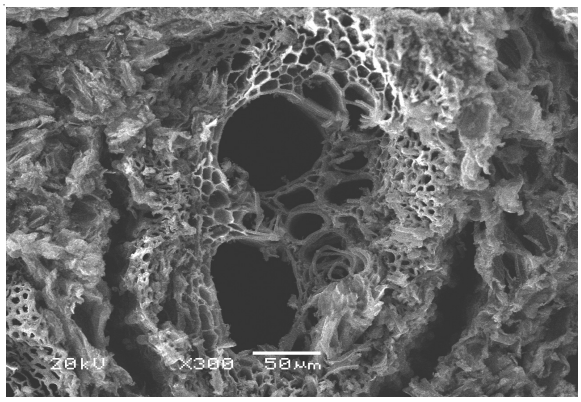

Fig. 5. Sliced activated carbon

ACKNOWLEDGEMENTS

This research was supported by King Saud University, Deanship of Scientific Research, College of Science Research Center.

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