Pertanika J. Sci. & Technol. 1(2):169-177 (1993)
ISSN: 0128-7680

Activated Carbon from Mangrove Wood (*Rizophora apiculata*): Preparation and Characterization

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Received 6 January 1992

ABSTRAK

Arang teraktif telah disediakan dari kayu bakau (*Rizophora apiculata*) melalui persulingan memusnah di dalam keadaan vakum. Suatu siri masa pemanasan dan suhu telah dipilih bagi penyediaan karbon teraktif. Nombor iodin bagi karbon teraktif yang disediakan telah ditentukan dengan kaedah jerapan dalam larutan akuas. Plot nombor iodin sebagai fungsi suhu pembakaran menunjukkan bahawa nombor iodin maksimum diperolehi dengan pembakaran pada 500°C selama 3 jam. Penentuan unsur-unsur surih, saiz liang dan topografi permukaan juga telah dilakukan.

ABSTRACT

Activated carbon was prepared from mangrove wood (*Rizophora apiculata*) by destructive distillation in vacuum. A series of heating times and temperatures were selected for the preparation of activated carbon. The iodine number of the activated carbon was determined by absorption method in aqueous solution. The plot of iodine number as a function of heating temperature revealed that the maximum iodine number was attained at 500°C after heating for 3 hours. Determination of trace elements, pore sizes and surface topography was also carried out.

Keywords: activated carbon, mangrove, iodine adsorption method.

INTRODUCTION

A lot of work has been done on activated carbon with respect to preparation and characterization that has enabled it to reach a significant level of achievement and usage. However, physical and chemical properties differ from one to the other (Mattson and Mark 1971) due to the difference in the base materials and the way they have been prepared. Activated carbon prepared from coconut shell for example, differs from that of palm fruit shell or rice hull even though they were prepared by the same method. Due to this variation, not all activated carbons are suitable for a specific application. The activated carbon prepared from a particular source may only be useful for a particular application.

Potential application of activated carbon from a particular source can be realized by detailed studies of its properties. The activated carbon

should be characterized to identify its properties and abilities to match to a particular application.

Thus mangrove wood was selected to identify the characteristic features of the activated carbon prepared from this particular species, in order to gain a new source of activated carbon. Furthermore, mangrove is an abundant natural resource which has been used commercially as fuel in charcoal-based manufacturing industries and also as chromatographic stationary phase (Jamaludin 1989). A wider range of potential usage can be realized by more detailed studies.

In addition, the method of preparation mentioned in this report is capable of producing larger pieces of activated carbon with higher value of iodine number than those currently available in the market. Activated carbon produced by this method has been successfully used as working electrode and porous junction for reference electrodes (Badri *et al.* 1989).

MATERIALS AND METHODS

Preparation

Mangrove trunks (*Rizophora apiculata*) were cut into small pieces and dried in an oven for one week at 100°C. The dried pieces were again cut into smaller pieces approximately 2.5 × 6 cm^2. Two or three pieces were put into the heating tube and heated in vacuum (0.5 - 0.01 mmHg). The activated carbon was prepared according to the method described earlier (British Patent 1984). Temperature and heating period were varied from 200-600°C and 1 - 4 hours respectively.

Iodine Number

The powdered form of activated carbon was obtained by sieving, using 300 μm mesh sieve. The iodine number was measured by the method as previously described by Puri and Bansal (1965).

0.5 g of ground activated carbon was added into 50 ml of 0.15 M N I_2 in 2.1 M KI solution (mole ratio of $I^-/I_2 = 14$). The mixture was shaken for a few minutes to allow proper wetting. It was kept for 3 days at room temperature (25°C) with occasional shaking. A set of controls was also prepared.

The mixture was filtered through glass wool and the filtrate was titrated against standard sodium thiosulphate solution with starch as an indicator. The iodine number was calculated based an the fact that 1 mg I_2 is adsorbed by 1 m^2 activated carbon as suggested by Puri and Bansal (1965).

To ensure the reversibility of iodine adsorption process and its physical nature the recovery analysis was conducted. This was done by washing the residue trapped in the glass wool with water, followed by benzene. The

amount of iodine in each solution was determined by titration against sodium thiosulphate as mentioned above.

Scanning Electron Microscopy

Pore sizes and surface topography of the activated carbon were estimated using a scanning electron microscope (SEM).

Neutron Activation Analysis (NAA)

Impurity content due to the presence of trace elements in the base material was obtained by neutron activation analysis. Prior to the analysis the sample was first refluxed with 1 M HCl and then washed with deionized water in Soxhlet apparatus for one week. A sample without any treatment was also prepared as a control, for comparison.

RESULTS AND DISCUSSION

Adsorption isotherm of iodine on the activated carbon prepared from mangrove wood at several heating temperatures is shown in *Fig.1.* In general, maximum adsorption was achieved after 3 days of soaking. The curve at 500°C showed that the adsorption did not change with the increase in soaking time. However at 400 and 600°C, adsorption started to decrease after 3 days of soaking. This may be due to the chemical change to the iodine in contact with activated carbon.

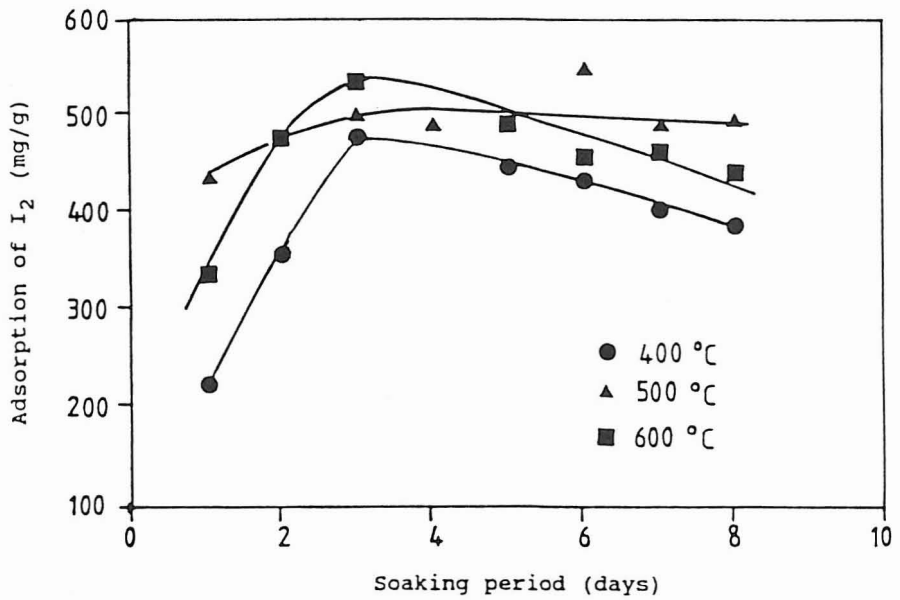

Fig. 1. Plot of iodine adsorption on the surface of activated carbon from mangrove wood against soaking period at various heating temperatures.

Fig. 2 shows that the magnitude of adsorption decreased as the I^-/I_2 ratio was increased and started to level off when the ratio reached about 14. At lower concentrations of I^-, the interaction of iodine with the solvent is less and hence the interaction with the surface of activated carbon is strong. This leads to the so-called "cooperative adsorption" (Kipling *et al.* 1964), i.e. the adsorption of more than a single layer of iodine on the activated carbon surface. Increasing the relative concentration of I^- resulted in an increase in the solvent-iodine interaction and the interaction with the surface also started to decrease. Finally, an equilibrium is achieved between both interactions and the adsorption reaches a constant value.

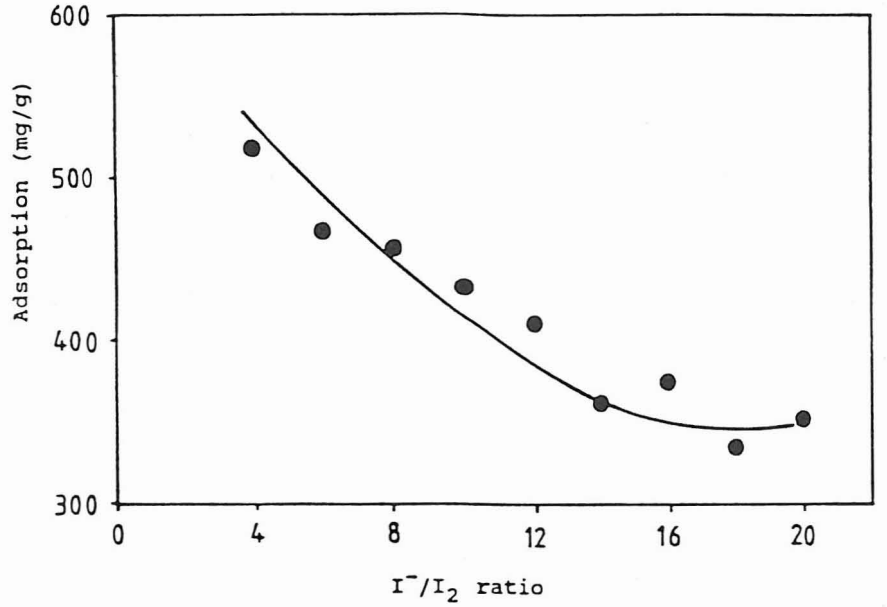

Fig. 2. Plot of iodine adsorption against I/I_2 ratio of activated carbon from mangrove wood.

In employing this method, it was assumed that the change in iodine concentration is only due to physical adsorption of the free iodine (I_2) on the surface and the wall of the pores of the activated carbon. However, there is a possibility that reduction of iodine by activated carbon takes place. Puri and Bansal (1965) obtained recovery percentage of around 96 to 100%, which supports the former rather than the latter case. They also found that only a small amount of iodine (∞ 3 mg/g) changed into hydroiodic acid. The percentage of recovery obtained in this work is about 88%.

Fig. 3 shows that the iodine number increases as the heating temperature increases up to a maximum value. However, increasing the temperature further resulted in a decrease in the iodine number. The maximum iodine number of about 503 m^2/g was obtained for activated carbon prepared by heating at 500°C. The maximum iodine numbers after 3 and 4 hours of heating were also obtained at about the same temperature. The value of these are 449 and 466 m^2/g respectively. Thus it is clear that 500°C is the optimum heating temperature.

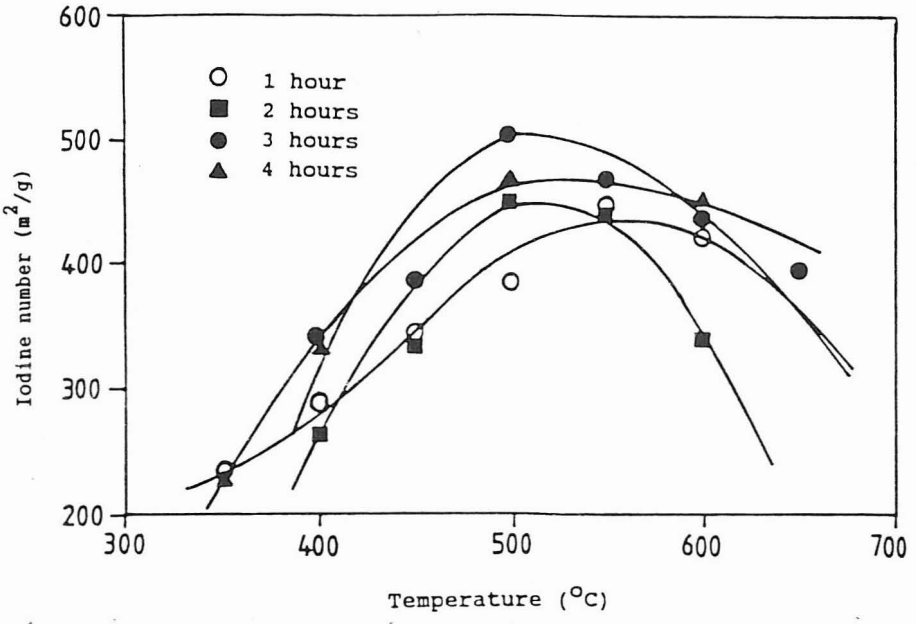

Fig. 3. Plot of surface area against heating temperature of activated carbon from mangrove wood prepared at several heating times.

As shown in *Fig 4.* maximum iodine number is obtained for activated carbon prepared by heating for 3 hours. Thus 3 hours is clearly the optimum heating period.

Since activated carbon is prepared from natural resources it is expected to contain traces of inorganic substances. Neutron activation analysis revealed that some major elements such as calcium, potassium, chlorine and manganese are present. Listed in table 1 are some of the major elements in activated carbon prepared at optimum temperature and time (500°C for 3 hours). Calcium is the highest, followed by sodium and potassium. The elemental content was found to be reduced to about 70% after washing. The increase of chlorine concentration in the latter is expected as HCl was used.

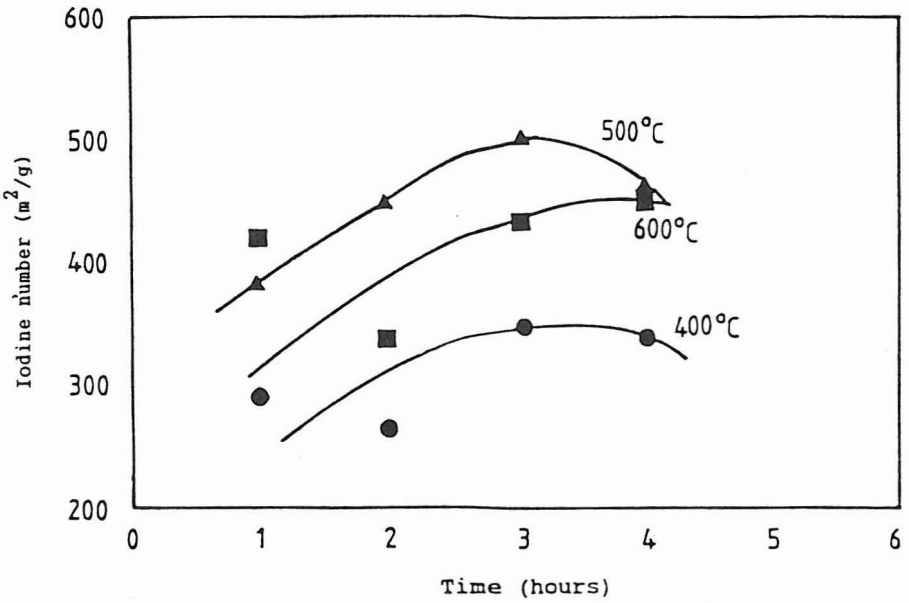

Fig. 4. Plot of surface area against heating time of activated carbon from mangrove wood at various temperatures

TABLE 1
Major elements in mangrove wood activated carbon. A is the sample without treament and B is the sample after washing in 0.1 M HCl and water.

Element (unit)	Concentration	
	A	B
Na (%)	0.299 ± 0.011	0.067 ± 0.003
K (%)	0.155 ± 0.003	0.020 ± 0.002
Ca (%)	1.15 ± 0.02	0.330 ± 0.04
Cl (%)	0.026 ± 0.004	1.208 ± 0.013
Mn (ppm)	38 ± 2	50 ± 2

Pore sizes were measured by scanning electron micrograph (Plate 1). Pores for longitudinal section lie between 40 - 60μ length and 20 - 40μ width. On the other hand the pore sizes of cross-section samples were found to be distributed over a larger range. The pore size can be divided into 3 groups according to the diameter, that is 80 - 100 μm, 10 - 20 μm and 1-2 um. Generally the pore sizes for activated carbon from mangrove wood are larger than those of coconut shell (< 10 μm) which can be used as porous junction for reference electrodes (Badri *et al.* 1989).

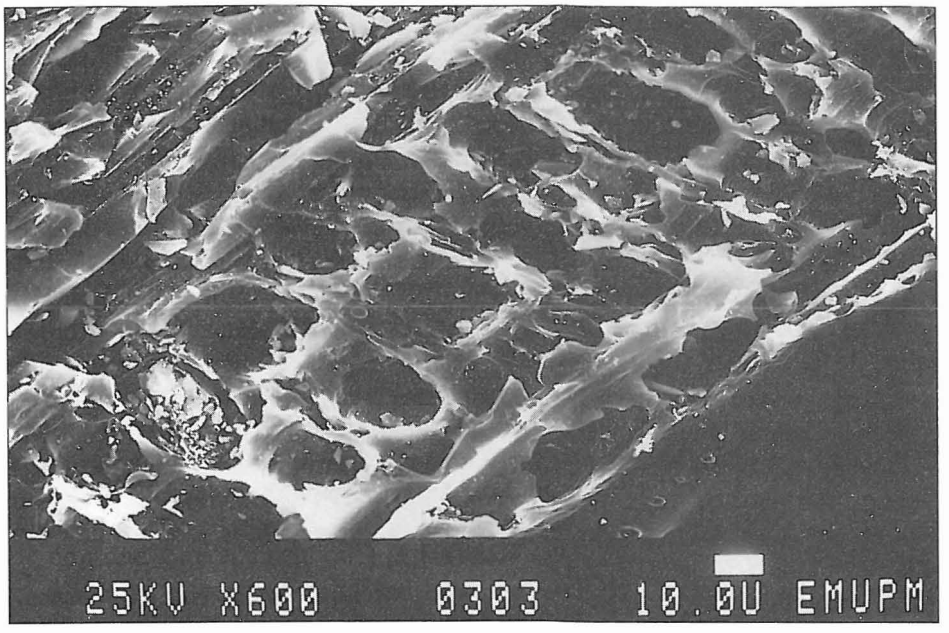

Plate 1a. Longitudinal section

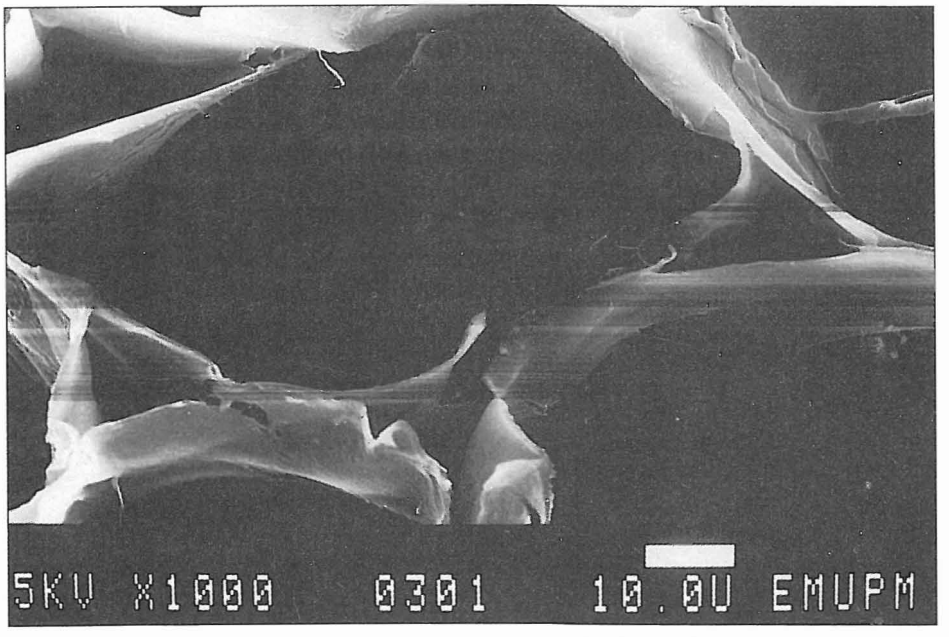

Plate 1b. Higher magnification image of longitudinal section

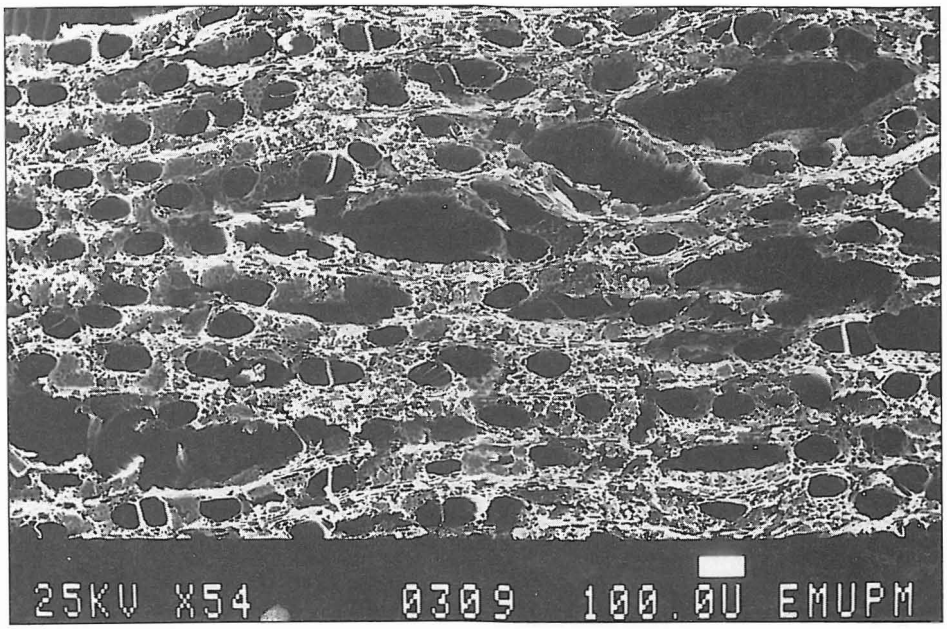

Plate 1c. Cross-sectional section

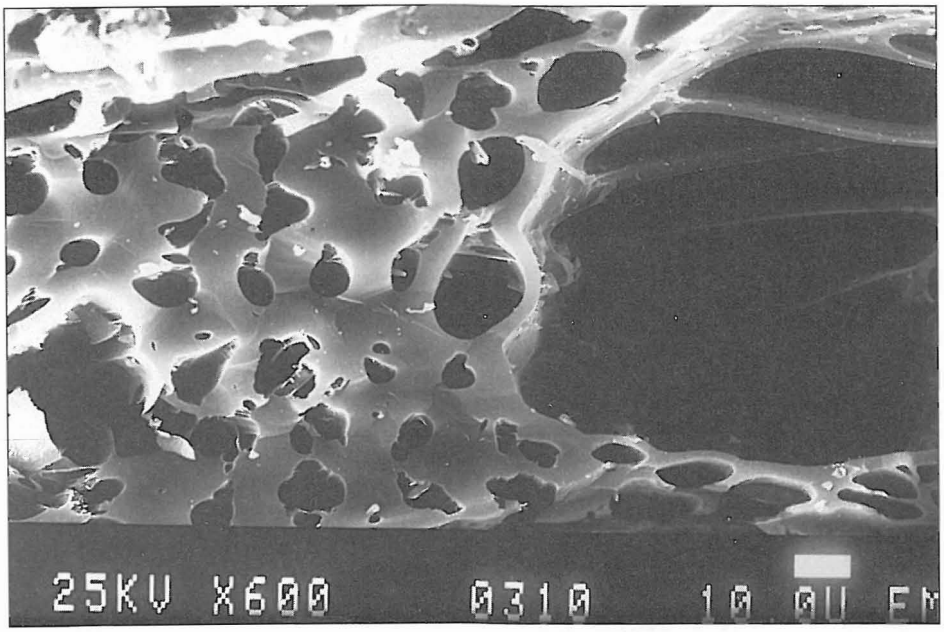

Plate 1d. Higher magnification image of cross-sectional section

CONCLUSION

Activated carbon prepared from mangrove wood showed quite a high value of iodine number compared to that of cocount shell. However this value is mainly determined by heating time and temperature, as different temperature or time may result in different value of iodine number. For a particular application which needs a certain value of iodine number, a specific temperature and time must be chosen to suit the requirements. For activated carbon prepared from mangrove wood, a maximum value of iodine number may be obtained by heating at 500°C for 3 hours.

Neutron activation analysis showed that traces of some elements are present and the pore sizes of the activated carbon prepared from mangrove wood are generally larger than those of coconut shell.

ACKNOWLEDGEMENTS

We are grateful to MPKSN (2-07-05-009) for funding this project, the Forest Department, Sepang for identification of the species of the mangrove wood, *Rizophora apiculata,* Unit Tenaga Nuklear, Bangi for NAA, Fakulti Kedoktoran Veterinar dan Sains Penternakan UPM for SEM and Mr. B. Steven.

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