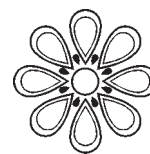




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REACHING THE UNREACHED: CHALLENGES FOR THE 21ST CENTURY

Activated carbon from Moringa husks and pods

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MORINGA OLEIFERA IS the most widespread species of the plant family *Moringaceae*. It is a rapidly growing tree native to the sub-Himalayan regions of north-west India and indigenous to many parts of Africa, South America and Asia, typically reaching a height of 3-4 metres, flowering and fruiting in one year from a 0.3 metre seedling even in poor quality soil (Sutherland, Folkard et al., 1994). It is known by different names around the world (Jahn, 1986), for example in India "Drumstick" or "Horseradish" tree. The tree produces large seed pods which can either be harvested when green for food or left to dry. The dried seeds can be crushed to produce a high quality vegetable oil and the resulting press-cake mixed with water and strained to form a coagulant for water treatment (McConnachie, Mtawali et al., 1994). The residue containing seed husks is currently discarded as waste. The research presented here has been carried out at the University of Edinburgh and the University of Malawi and demonstrates that a simple steam pyrolysis procedure can form high quality microporous activated carbons from both the waste husks of *Moringa oleifera* and the pods. The work is part of an overall assessment of the viability of the establishment of *Moringa oleifera* plantations in tropical areas for the benefit of rural communities and is being carried out in collaboration with the Engineering Department, The University of Leicester, U.K.

Raw materials

The seeds were produced in Moringa plantations in Malawi and the husks supplied as whole seeds or as a waste material from crushed and winnowed seeds. For the whole seeds it was possible to remove the kernels leaving only husk material. The crushing of seeds is carried out for water treatment as the kernels contain water soluble proteins which possess a net positive charge and act as an effective coagulant. The winnowing process essentially separates the less dense husk material although a proportion of the kernels is included.

Carbon production methods

Activated carbon has been produced by similar processes at the University of Edinburgh and the University of Malawi comprising heating the precursor (husk material or pods) in a furnace to eliminate volatiles (carbonisation) with concurrent activation from steam. This method has been shown to be economical in producing effective carbons from materials such as apricot stones, grape seeds and coconut shells (Gergova, Petrov et al., 1993)

compared with more elaborate methods such as two stage processing for carbonisation and activation or chemical activation methods. The overall aim was to establish a simple and least cost method which would be suitable for production of an effective carbon on a commercial scale.

- **At Edinburgh**, charges of the precursor (30g or so for the husk material and 10g for the pods) were placed in a quartz combustion boat to a depth of 10mm, inserted into a tube furnace of 65mm internal diameter and a flow of steam maintained at approximately 2mL (liquid water) per minute. The steam and evolved gasses were condensed at the outlet. Heating was at approximately 20°C per minute and the temperature of the air just above the sample was monitored by thermocouple probe. For the husks the final air temperature was either 500, 600, 700, 750 or 800°C (all $\pm 10^\circ\text{C}$) and the duration at the final temperature either 30, 60 or 120 minutes and for the pods, 500, 600, 650 or 700°C held for 30 minutes. Temperature higher than 700°C for the pods resulted in ash. After the furnace had cooled to between 200 and 300°C the carbonised material (char) was removed, allowed to cool for a few minutes and then weighed to determine the yield. The char was then stored in a desiccator. Each char was produced twice and before being tested for adsorptive capacity each pair was mixed, homogenised and sieved to $<250\ \mu\text{m}$ then dried in an oven overnight at 110°C. (Carbon of this nominal size can be regarded as in powdered form, granular being typically $>400\ \mu\text{m}$). Washing of selected carbons was undertaken to remove ash. Also, regeneration was carried out on the 800°C/30 minute husk carbon by repeating the heating sequence and its adsorptive properties then reassessed.
- **In Malawi** the husk material was placed in a steel container and then heated in a furnace. Initially there was no control on the thickness of the raw material in the container but it was decided that this may not give repeatable results and the depth was restricted to a uniform 10mm. Steam, generated by a domestic pressure cooker on a hot-plate, was injected continuously into the container throughout the sequence of furnace warming up and running at the desired temperature. Temperatures of 500, 600, 700 and 800 (all $\pm 10^\circ\text{C}$) were measured by a thermocouple probe outside the steel container and maintained for each of 30, 60 and 120 minutes. The container was removed after the furnace had cooled to 160°C. Initially, steaming was continued

while the container was further cooled with tap water on the outside. The resultant carbon was moist and to avoid any resulting irreproducible ash removal it was decided to improve and simplify the procedure by stopping the steam injection when the temperature fell to 200°C.

Carbon characterisation

Two adsorbates, phenol and methylene blue, were used to establish the porosity characteristics of the Moringa carbons. Two commercial carbons, Acticarbone and Eurocarb, were also phenol tested. Conventionally the pores of activated carbon are divided into three groups, micropores of less than 2 nano-metres in "width", mesopores of 2-50 nm and macropores of greater than 50 nm. Phenol molecules can penetrate down to the lower range of micropores whereas methylene blue requires pores of about 1.3nm so can only enter the largest micropores and the mesopores.

For the adsorption tests, carbon samples (0.5 or 0.2g - see Figure 2) were added to 100mL of 0.01M solution of the methylene blue or the phenol adsorbate in 250mL Erlenmeyer flasks, sealed with Suba-seals, then shaken at 90 strokes per minute in a temperature controlled shaker bath at 25°C for 24 hours. The flask contents were then vacuum filtered through Whatman No. 542 filter paper, acidified with a drop of 1M HCl and the residual adsorbate concentrations found by ultra-violet spectrophotometry at 665 nm for the methylene blue and 270nm for the phenol. All determinations were at least duplicated.

For the Edinburgh carbons, in addition to the phenol and methylene blue tests, nitrogen adsorption analysis for surface area characterisation and pore size distribution was carried out as well as iodine number determina-

tion to assess each carbon's ability to remove low molecular weight compounds (Warhurst, McConnachie et al., in press), (Warhurst, McConnachie et al., in press).

Results

The results of the tests are given in Figures 1 and 2. Each carbon has been identified as, for example, EH700/30 which is carbon derived from husk material used at Edinburgh and heated at 700°C for 30 minutes; M denotes carbon from husk material used at Malawi, and P is carbon from pods.

The process of steam pyrolysis gives reproducible yields with duplicate runs differing by less than 1 per cent. Yield tends to reduce as the heating temperature increases. The duration of heating (soak time) has little effect on yield at 500 or 600°C but tends to give reduced yield for longer times at the higher temperatures. The pods were turned to ash at temperatures >700°C whereas the husks ashed above 800°C and also at 800°C when the time was 120 minutes.

Both the methylene blue and the phenol results show that the capacity for a carbon to adsorb impurities will tend to improve as the temperature and the soak time of production are increased. A compromise must be reached between obtaining a high yield and maximising the adsorptive capacity and these are compared in Figure 2. The cost of production of the carbon is likely to be the governing factor which requires assessment of the power requirements of the particular furnace used.

One difficulty which arose when carbon which had been produced from the pods at 500 and 600°C was used in the adsorption tests was that a brown colour was imparted to the sample which prevented reliable spectrophotometer readings. Acid washing of the carbon coun-

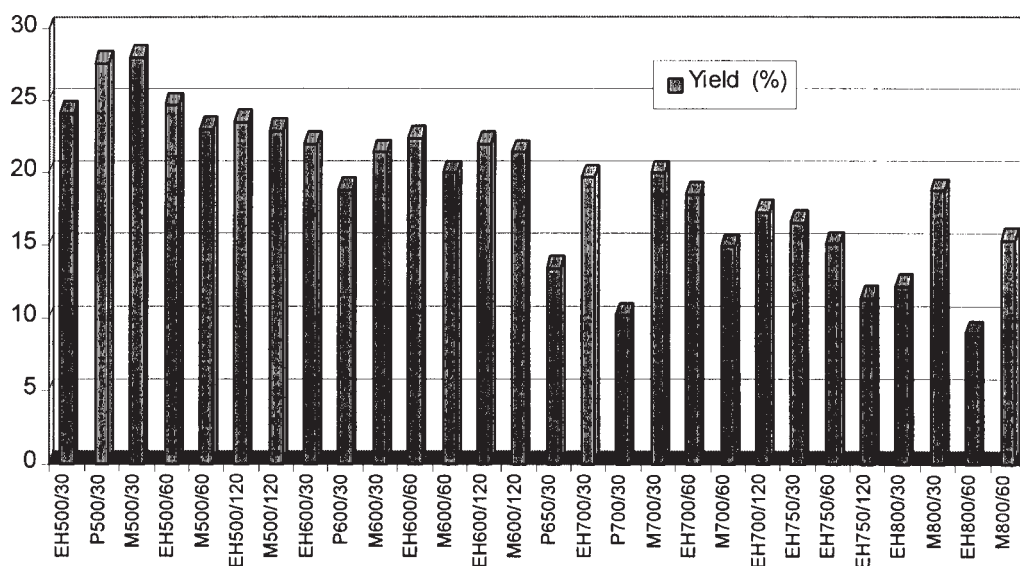
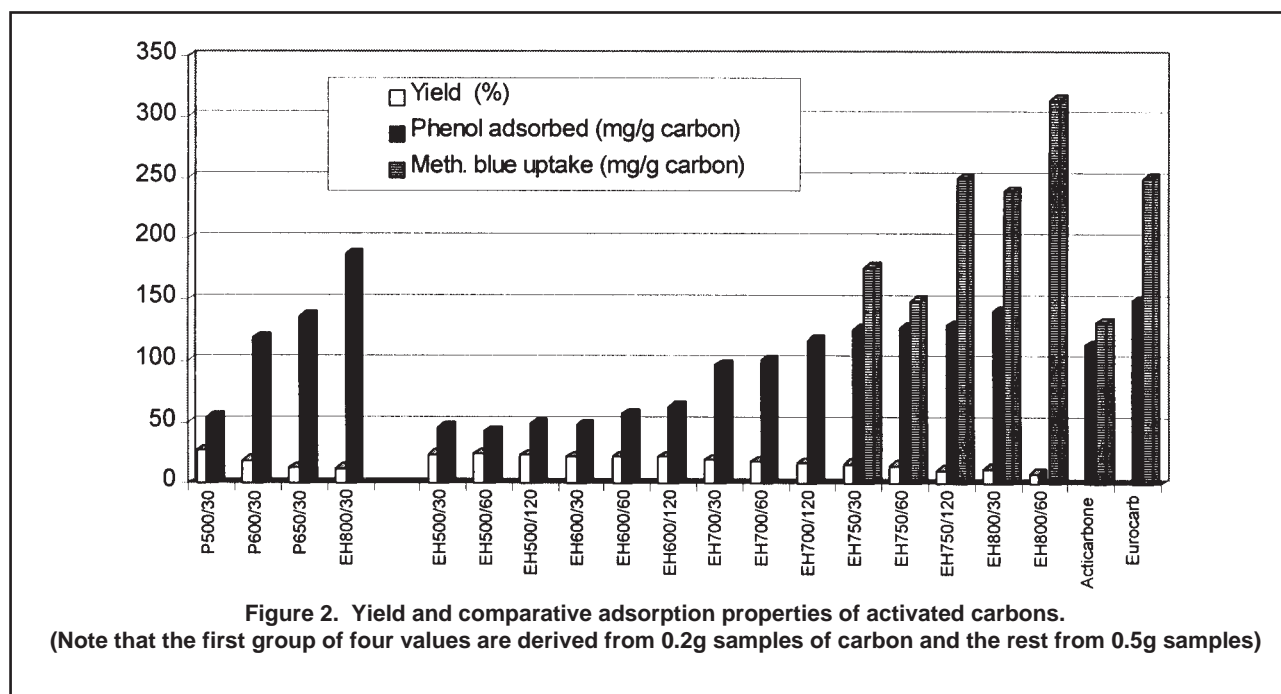


Figure 1. Activated carbon yield as percentage of original mass.



teracted this problem and the results shown for the pods in Figure 2 are from carbons treated in this way.

The BET surface areas derived from the nitrogen adsorption isotherms for EH800/30 was $713\text{m}^2/\text{g}$ and after rinsing, $931\text{m}^2/\text{g}$ (Warhurst, McConnachie et al., in press).

Waste product

The composition of the derived gases and condensate has not been determined so far but other work on steam pyrolysis volatiles from apricot stones and coconut husks (Razvigorova, Goranova et al., 1994) has identified the presence of derivatives of phenol, guaicol, syringol, resorcinol, free fatty acids and the esters of fatty acids. Unless a use can be found for this material it will have to be disposed of. Fortunately it is likely to be readily biodegradable.

Applications

The potential applications for activated carbon include the adsorption of excessive concentrations of iron and manganese from groundwater, taste and odour compounds, bacterial and viral pathogens as well as the tertiary treatment of wastewaters.

Seasonal problems of taste and odour in drinking water from a number of lake sources in Malawi arise from algal blooms, mainly of the species *Oscillatoria* and *Anabaena*, which are difficult to remedy and it is planned to assess the effectiveness of carbon treatment at the Blantyre Water Treatment Works. Current treatment there is a sequence of microstraining, addition of potassium permanganate as an algicide and to remove iron and manganese, addition of chemical coagulant, flow through a mixing channel then rapid gravity filters. Chlorination is applied at various points along the pipe distribution network. The filter medium is sand obtained locally.

Conclusions

Activated carbons can be produced from *Moringa* husks and pods which compare favourably with commercial carbons using a one stage steam pyrolysis process.

For husks the most economical process is likely to be at 800°C for 30 minutes and for the pods, 650°C for 30 minutes.

For some applications a rinsing stage of treatment for the carbon might be beneficial.

Further work

Aspects of the carbon work still to be undertaken are,

- power requirements for various combinations of process temperature and soak time
- adsorption capacity of granular carbons after use and regeneration
- application of carbon in treating Malawi drinking water supplies for removal of taste and odour
- maximisation of carbon production.

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