JOURNAL OF MATERIALS SCIENCE 30 (1995) 544–551

Scanning electron microscopic study of acacia and eucalyptus wood chars

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SEM studies of acacia and eucalyptus wood chars, prepared under different carbonization conditions, were undertaken to provide information on what happens in the transformation of wood to chars. The material normally lost as volatiles contributes totally to the formation of pores, cracks and pyrolytic carbon. Both woods exhibited similar devolatilization behaviour in pore structure development, crack formation and pyrolytic carbon deposition, showing a decrease in pore size with an increase in carbonization temperature and cracks/voids formation during rapid carbonization at higher temperatures (i.e. 800–1050 °C). Slow carbonization led to pyrolytic carbon deposition in resulting wood char structures and did not disturb the fibrous nature and cell structures of the wood, even at a high carbonization temperature of 1200 °C. Prolonged heating at carbonization (slow) temperatures of 800 and 1000 °C caused sintering of the adjacent fibres resulting in the formation of compacted mass.

1. Introduction

With increasing interest of metallurgists towards the utilization of wood char (a renewable and non-polluting energy source) for ironmaking, it seems necessary to know those characteristics of wood char which are influential in controlling its reactivity, strength, etc. Literature reveals that these properties of carbonaceous materials are sensitively dependent on their pore and surface characteristics. Thus, the knowledge of the pore and surface characteristics of wood chars, as affected by the carbonization conditions, is of utmost importance in order to use them more effectively for ironmaking.

In order to deduce some extensive remarks like petrographic analysis, pore structures, surface characteristics, etc., extensive scanning electron microscopic (SEM) photographs of coals, oil shale, chars, cokes, pitches, pyrolytic carbons, etc. have been published by Augustyn *et al.* [1], Singla *et al.* [2], Tomita *et al.* [3], Miura and Silveston [4] and others [5–8]. But, to our knowledge, very few studies have been made on changes in surface and pore characteristics of wood char with heat treatment temperature and other carbonizing parameters.

McGinnes *et al.* [9] have used SEM techniques to study the morphological changes (mainly shrinkage phenomena) occurring during the transformation of white oak, shortleaf pine and Eastern redcedar woods into their chars. During the investigation of Ehrburger and Lahaye [10] on the effect of carbonization on the porosity of beechwood, radial, transverse and tangential cuts of original and carbonized samples were studied by SEM. These studies indicated that the porous texture of carbonized material remained unchanged, modified by thinning down of cellular walls by heat treatment. From an SEM analysis, Standish and Tan Jung [8] concluded that the pore structure of wood char is fibrous with typical bimodal size distribution.

In this paper, observations of the SEM investigations of acacia and eucalyptus (known wood species for energy plantations under Indian conditions) wood chars prepared under different carbonization conditions are reported. The aim has been to study the changes in structural morphology (pores, fibres characteristics, etc.) occurring during the transformation of wood into char. Wood char is a friable product of wood pyrolysis and the use of SEM, due to its depth of focus feature, allows detailed observations of such a highly brittle material without preparation of thin sections (prerequisite for TEM studies) or polishing (prerequisite for optical microscopic studies).

2. Structural features in wood

Wood consists of a network of fibrous cells. The chief function of fibres in the woody plant is to give it mechanical strength and their position and distribution save the plant from various stresses and strains of environmental forces [11], e.g. strong winds. The woods are of two types: either soft or hard. The hardwoods are generally strong, dense and heavy, due to a high proportion of fibres. Arrangements of fibres, rays and vessels (present in wood) in three different planes (tangential, radial and transverse) of *Liriodendron tulipifera* wood, which is a typical hardwood species, are shown schematically in Fig. 1 [12], as an example. Vessels run parallel to the tree axis. They have thin walls and their cross-sections are either polygonal or elliptical. Fibres are also parallel to the

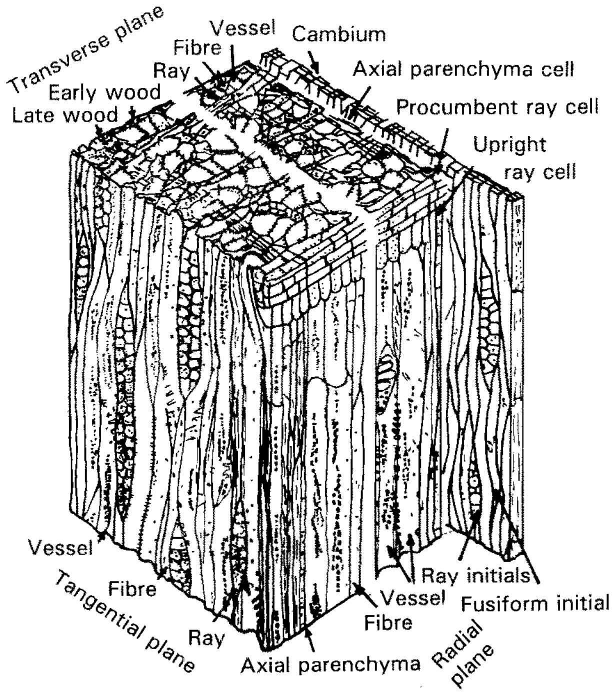


Figure 1 Block diagram of vascular cambium and wood of *Liriodendron tulipifera* (Tulip tree).

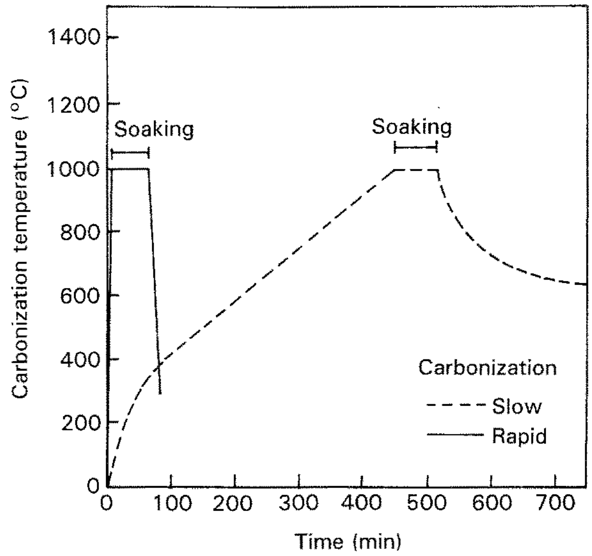


Figure 2 Typical time–temperature profile for slow and rapid carbonization.

tree axis. Their walls are rather thick and the inside radii range from 1 to 18 μm as opposed to 5–60 μm in vessels [10].

3. Experimental details

3.1. Preparation of wood chars

In the present investigation, preparation of wood chars has been carried out by carbonizing wood samples under two different heating–cooling cycles, namely, slow and rapid. Fig. 2 shows the typical time–temperature profile for slow and rapid carbonization processes adopted in the present investigation.

While carbonizing according to the slow schedule, the air dried wood pieces (15 mm^3), placed in a stainless steel retort, were kept inside the muffle furnace and heated slowly from room temperature to predetermined carbonization temperatures of 250, 400, 600, 800, 1000 and 1200 °C at the rate of ca. 4 °C min^{-1}. Samples were soaked at their final carbonization temperature for a period of 1 h and then allowed to cool in the furnace itself. To examine the influence of soak time on resultant wood char properties and structure, the wood samples were carbonized slowly at temperatures of 800 and 1000 °C and the soaking time varied from 1 to 5 h.

In carbonization following rapid schedule, the air dried wood pieces (15 mm^3) were placed in the stainless steel retort, which was then inserted into the muffle furnace maintained at the required carbonization temperatures of 400, 600, 800 and 1050 °C. The retort was kept in the furnace for 1 h, then removed and cooled in air.

3.2. Scanning electron microscopic observations

Scanning electron micrographs to assess the morphology of wood chars were obtained using PSEM-500 (magnification capacity 6× to 80 000×) and JEOL-840A (magnification capacity 15× to 1 30 000×) scanning electron microscopes. In this study, specimens (approximately cubic in shape, with edges less than 0.78 cm) were mounted on aluminium studs and coated with gold to produce a conductive path and were examined at magnifications increasing to 6500×.

4. Results and discussion

4.1. Scanning electron microscopic views of woods

Typical SEM of acacia and eucalyptus woods are presented in Figs 3 and 4; they clearly indicate the presence of dense fibrous cells of cellulose in both woods, which means they are hard and strong and ultimately useful for char making. Such a fibrous nature can be noted only on viewing through tangential or radial plane of wood. The rays (rich in resin content) present in acacia and eucalyptus woods are clearly evident in these figures.

As seen in the micrographs of these woods (Figs 3 and 4), the fibre walls are approximately free from pits or pores and the structure is highly directional (i.e. fibrous), which is a general characteristic of wood.

4.2. Wood char structure along transverse and tangential planes

The cell cross-section can be noted by viewing the char in the transverse section. SEMs of eucalyptus wood char prepared under rapid carbonization at 1050 °C, taken by viewing along different planes, are shown in Fig. 5. On viewing this wood char piece along its transverse plane (Fig. 5a) the cell cross-sectional structure is clearly seen and the cell wall thickness can be determined. This wall thickness gives strength to the structure. Fig. 5b is the SEM view of this wood char at low magnification, showing transverse and tangential planes in combination. On viewing this char piece in the tangential plane (Fig. 5c) the fibres and vessels can be seen clearly (no ray cells).

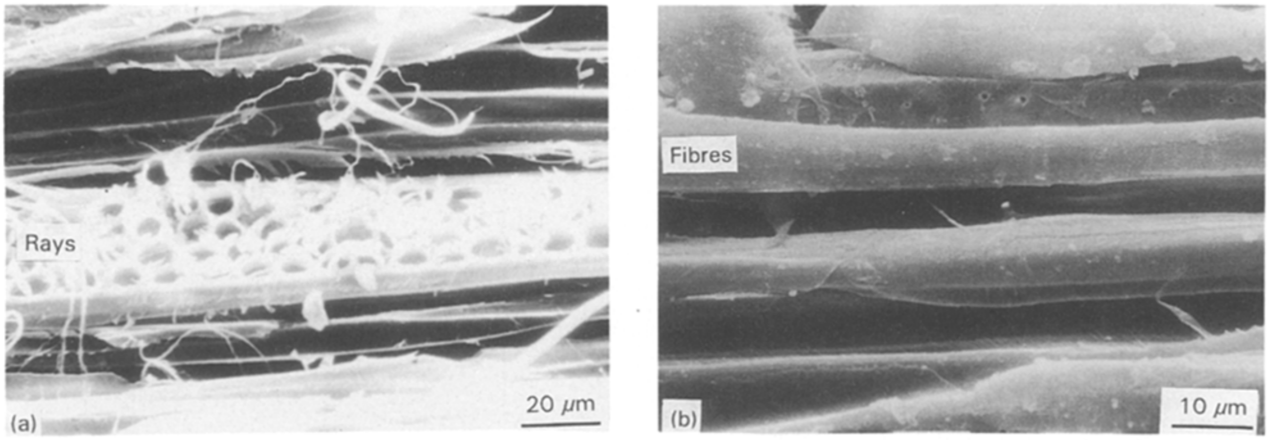


Figure 3 SEM photographs of acacia wood viewed along tangential section.

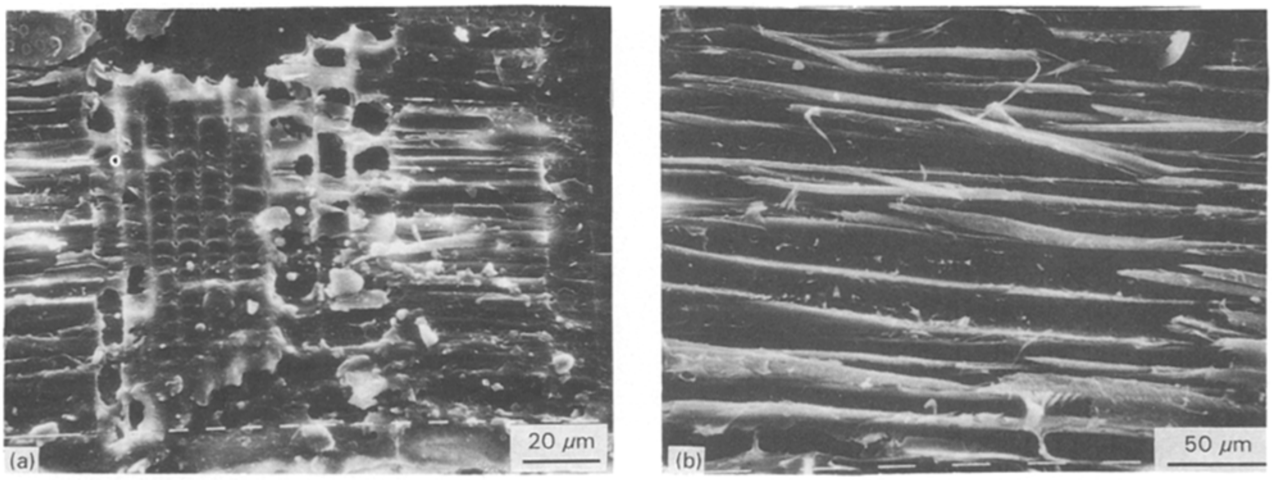
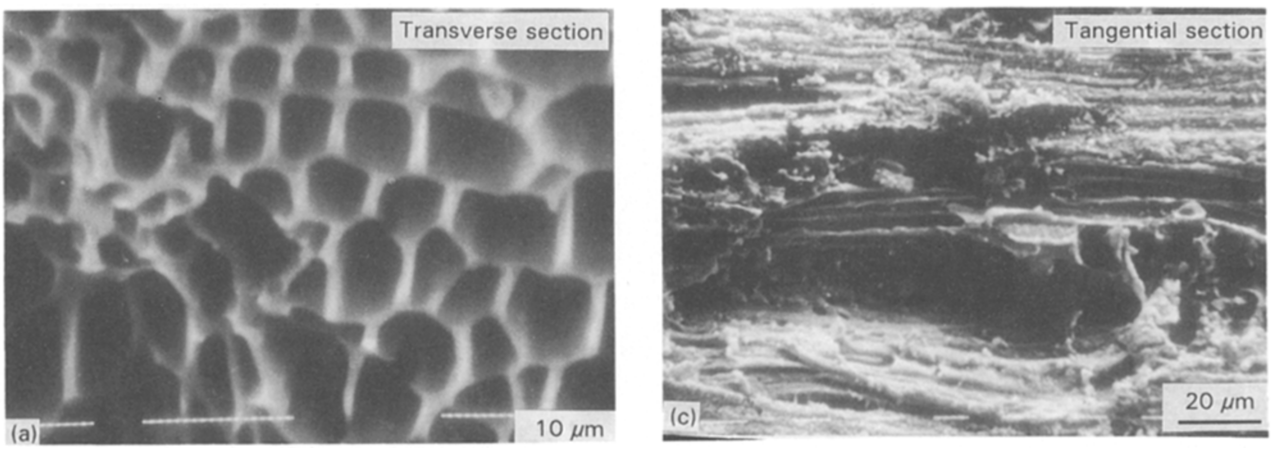


Figure 4 SEM photographs of eucalyptus wood when viewed along (a) radial section (b) tangential section.



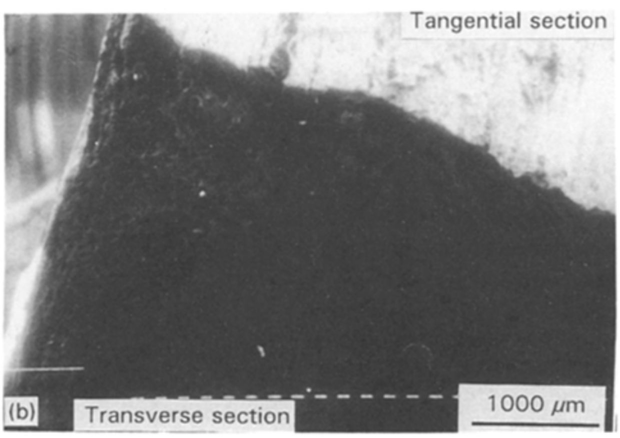


Figure 5 SEM photographs of eucalyptus wood char prepared at 1050 °C under rapid carbonization. Soaking time = 1 h.

4.3. Pores in wood char

Due to the evolution of volatile matter during carbonization of wood, pitting of the vessel surface occurs which leads to the development of pores all along the walls of the vessels. Such small pores can be seen in the SEM photographs of acacia wood char prepared at 600 °C under rapid carbonization, shown in Fig. 6. As evidenced by Fig. 6a, the vessel has a much wider diameter in comparison with that of fibre cells. The pitted walls of the vessel, on magnification, reveal the

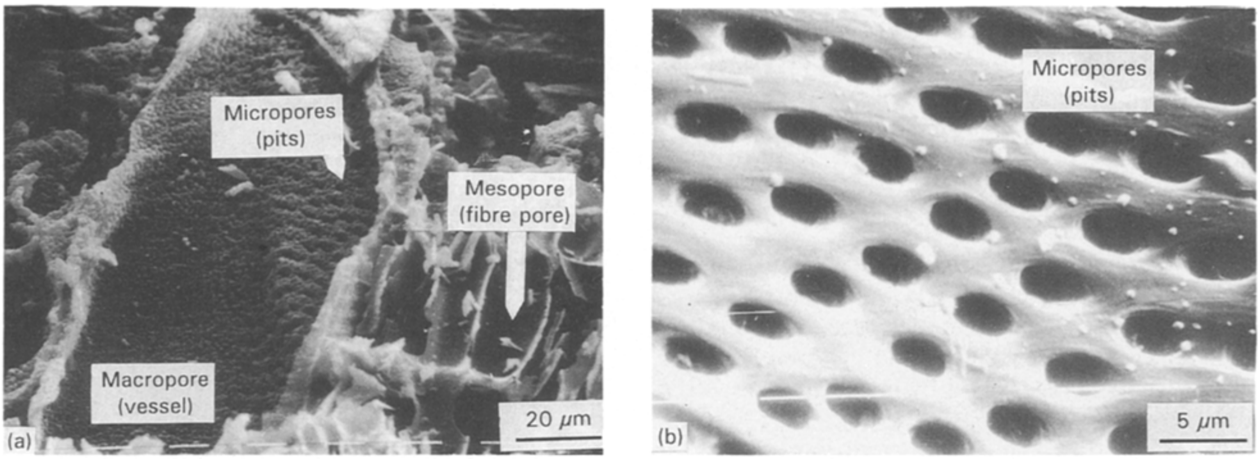


Figure 6 SEM photographs of acacia wood char prepared at 600 °C under rapid carbonization. Soaking time = 1 h.

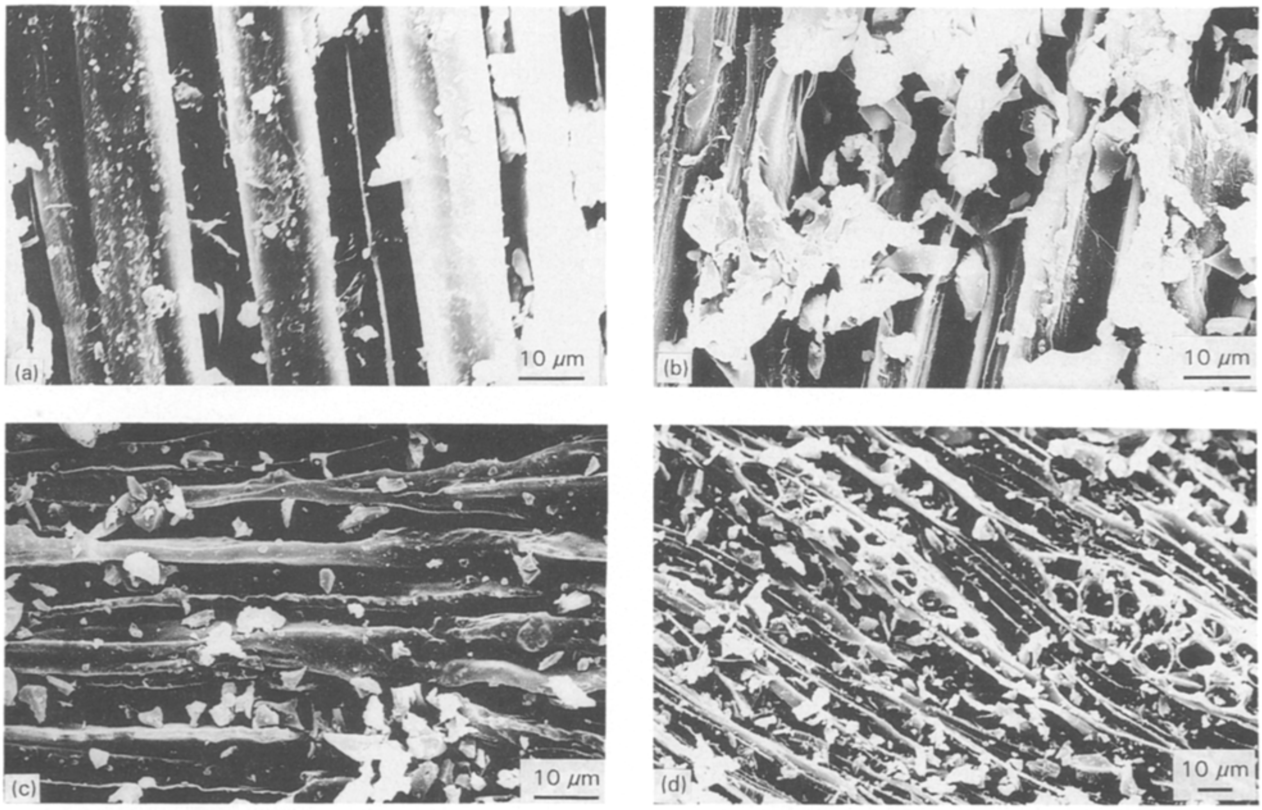


Figure 7 SEM photographs of acacia wood chars prepared under slow carbonization at temperatures (°C) (a) 260; (b) 400; (c) 600; (d) 1000; (e) 1200. Soaking time = 1 h.

cross-section of pits, as seen in Fig. 6b. The wood char thus appears to contain three different types of pores:

1. macropores (widths exceeding 50 μm) originating from vessels;
2. mesopores (widths 2–50 μm) originating from fibres;
3. micropores (widths less than 2 μm) formed on the walls of the vessels giving pits.

The development of such enormous and such a large variety of pores in wood char renders a high surface area for chemical reaction to occur.

4.4. Effects of carbonization conditions and wood species on char structure

The basic structural morphology of wood initially gets affected during carbonization and the extent of change depends upon the carbonization conditions.

4.4.1. Carbonization temperature

The SEMs of acacia wood chars prepared under slow carbonization at five different temperatures, 260, 400, 600, 1000 and 1200 °C are presented in Fig. 7a to e, respectively. From these SEMs it can be seen that the fibrous structure, characteristic of original wood, has been conserved during carbonization, even at a high temperature of 1200 °C.

The SEMs showing the presence of pits (micropores) on vessel walls of acacia wood chars, prepared under slow carbonization at temperatures of 1000 and 1200 °C, are shown in Fig. 8a and b, respectively. The size of the pit, as measured by image analyser (VIDS III), was found to decrease by ca. 25% with an increase in carbonization temperature from 1000 to 1200 °C. A similar effect was also noticed under rapid carbonization, Fig. 9a and b, respectively. Here, the pit size (measured by the same image analyser) decreased by about 30% when the carbonization temperature was increased from 600 to 1050 °C. In addition to the development of pores, the wood chars made under rapid carbonization at temperatures of 800 and 1050 °C showed the presence of cracks and voids in their structures, which can be seen in SEMs in Fig. 10. These micrographs also reveal an increase in the extent of crack formation with an increase of carbonization temperature (i.e. heating rate). The wood chars prepared under rapid carbonization at low temperatures of 400 and 600 °C, however, had well preserved fibrous structures and were free from any cracks and voids. It appears that rapid heating at higher temperatures resulted in rapid evolution of volatile matter, rupturing the structure under excessive pressure.

4.4.2. Carbonization programme

The programme of carbonization (i.e. heating–cooling cycle) markedly influenced the structural characteristics of the wood chars produced. The wood chars produced by slow carbonization showed structural morphology different to those obtained by rapid carbonization. As evidenced by the SEMs shown in Fig. 11, the woody fibrous structure is more preserved in wood char made under slow carbonization (Fig. 11b), and these chars are practically free from any cracks and voids, whereas the micrographs of wood chars prepared under rapid carbonization at temperatures of 800 °C (Figs 10a and 11a) and 1050 °C (Fig. 10b) showed the evidence of breaking and distortion of fibres leading to the formation of cracks and voids. The preserved fibrous structure in wood chars is believed to provide them with strength and rigidity to enable them to withstand various strains caused during their transfer from one place to another. This would be of great significance in the use of wood chars as fuel/reductant for blast furnace iron making, where they have to resist the effects of numerous handling operations in their transit from the carbonization kilns to the blast furnace.

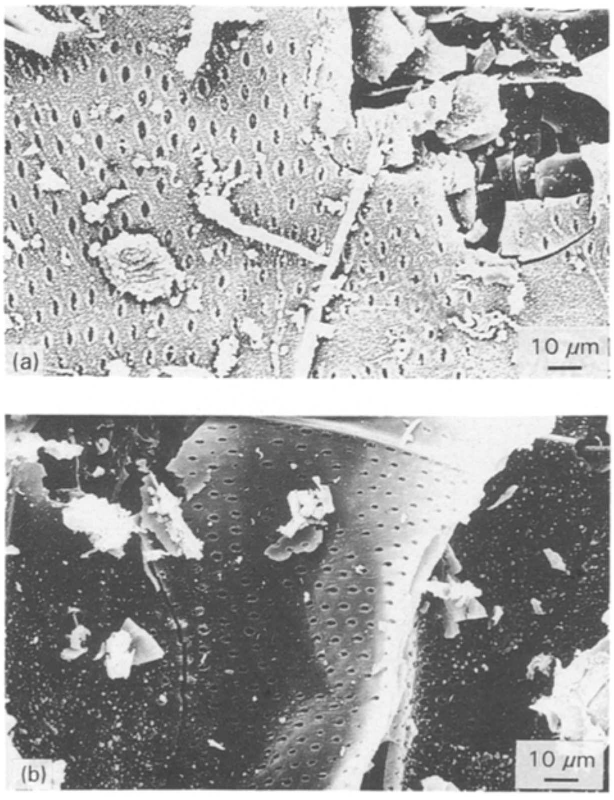


Figure 8 SEM photographs of acacia wood chars prepared under slow carbonization at temperatures (°C) (a) 1000; (b) 1200. Soaking time = 1 h.

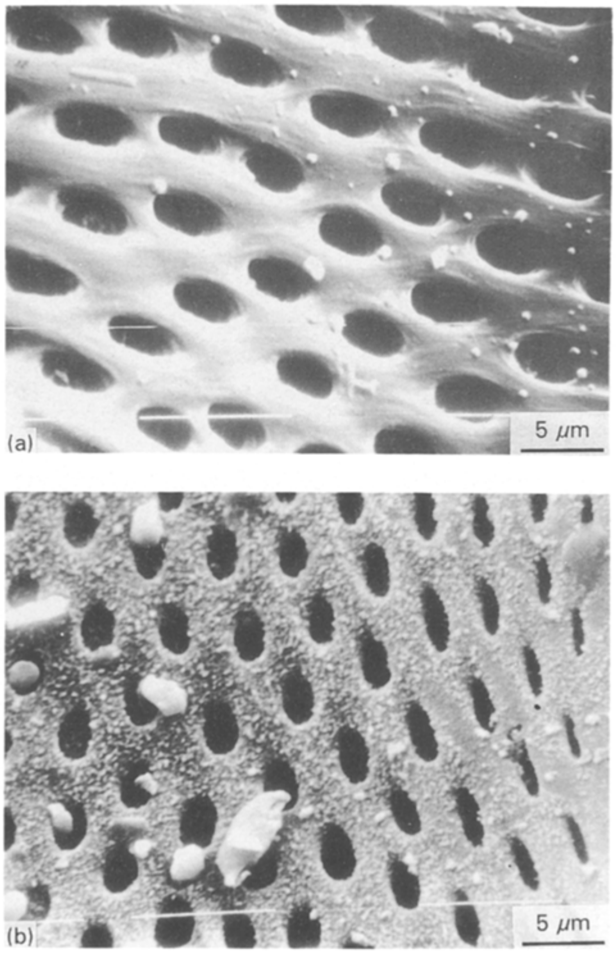


Figure 9 SEM photographs of acacia wood chars prepared under rapid carbonization at temperatures (°C) (a) 600; (b) 1050. Soaking time = 1 h.

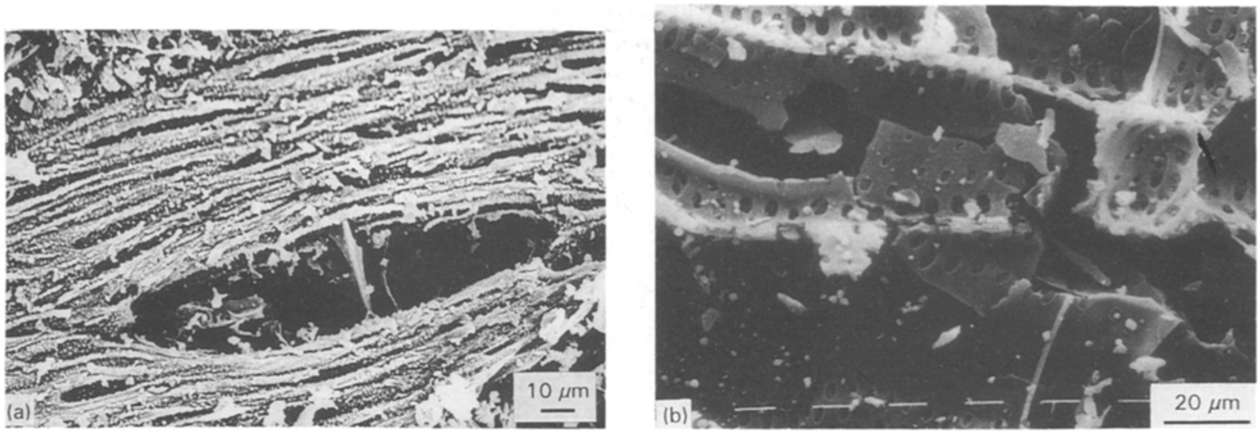


Figure 10 SEM photographs of acacia wood chars prepared under rapid carbonization at temperatures (°C) (a) 800; (b) 1050. Soaking time = 1 h.

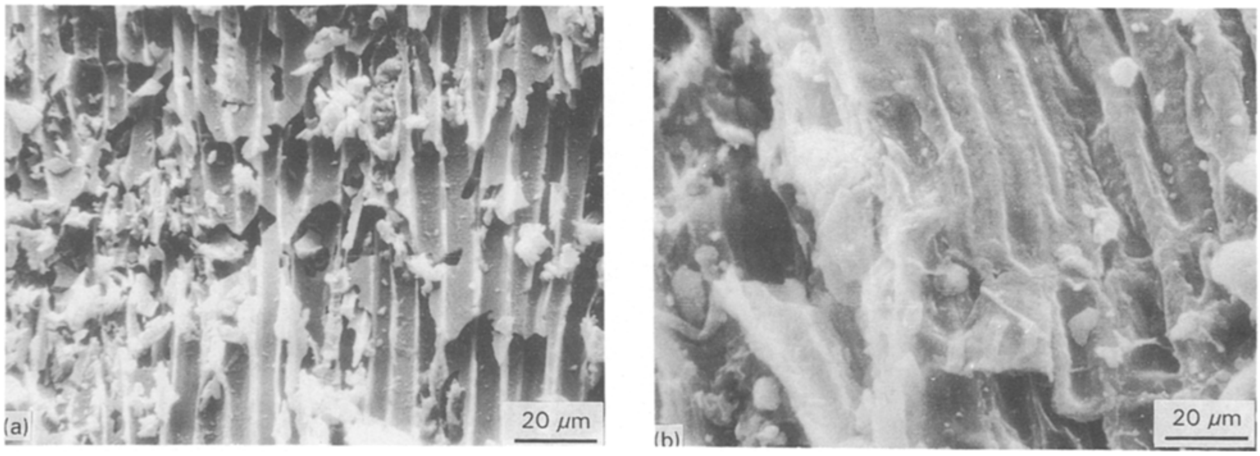


Figure 11 SEM photographs of eucalyptus wood chars prepared at 800 °C under (a) rapid carbonization; (b) slow carbonization. Soaking time = 1 h.

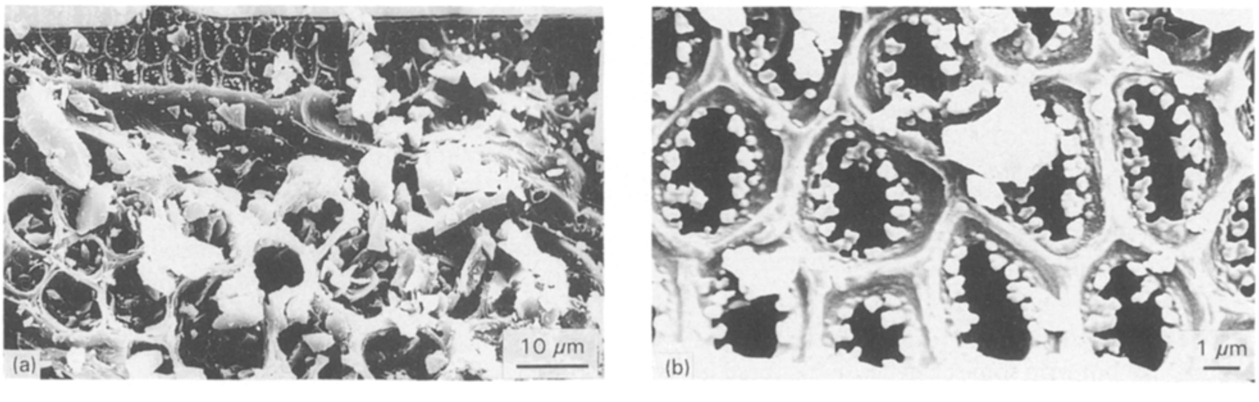


Figure 12 SEM photographs of acacia wood char prepared under slow carbonization at 1000 °C. Soaking time = 1 h.

In case of carbonization of acacia and eucalyptus wood species under a slow heating–cooling cycle, some embedded particles (expected to be pyrolytic carbon resulting from the cracking of hydrocarbons) were found to be present in the resulting char structures, as seen in the micrographs presented in Figs 11b and 12. In contrast, no sign of the presence of such deposited particles was noticed in the microstructures of wood chars prepared under rapid carbonization (Figs 5, 9, 11a and 13).

The micrographs of eucalyptus wood chars prepared under two different heating–cooling cycles, namely slow and rapid, at a temperature of 800 °C are given in Fig. 11a and b, respectively. The distinct difference can be noticed in the appearance of fibre surfaces. The fibre morphology of wood char made by

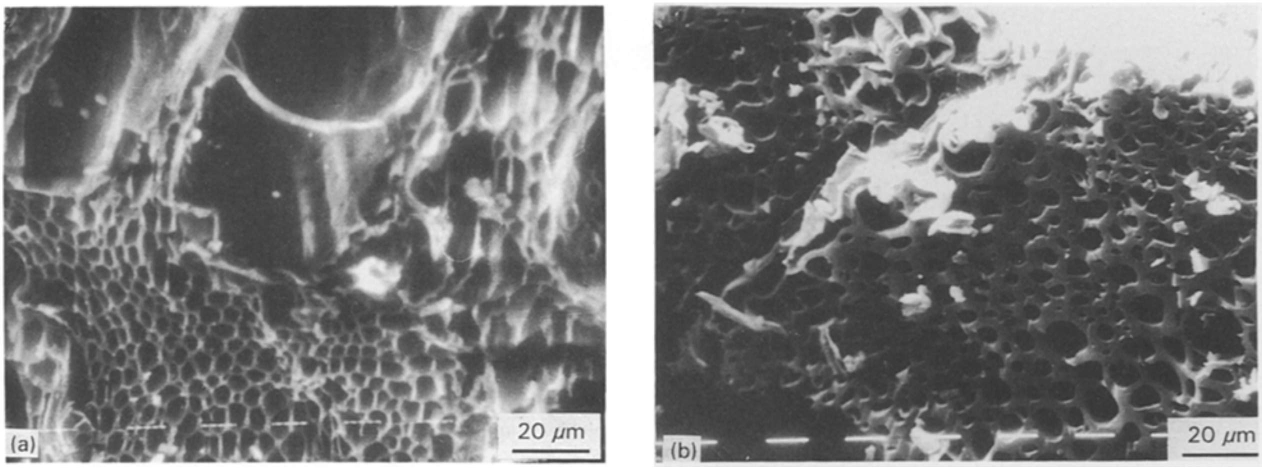
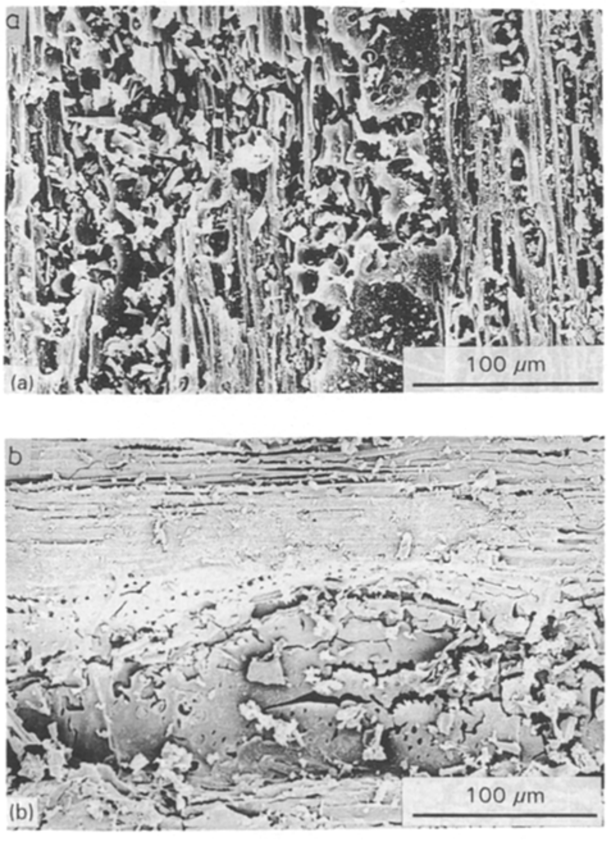


Figure 13 SEM photographs of wood chars prepared under rapid carbonization from (a) eucalyptus wood at 1050 °C; (b) acacia wood at 600 °C. Soaking time = 1 h.



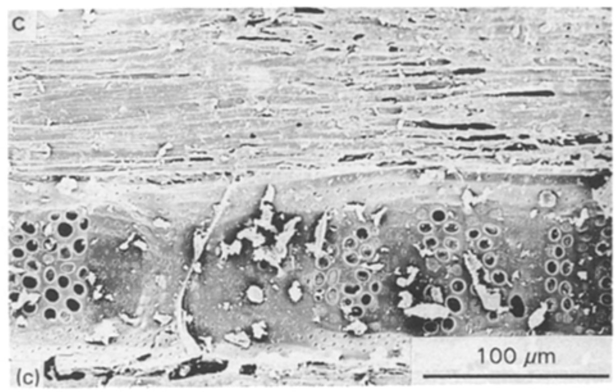


Figure 14 SEM photographs of 1000 °C eucalyptus wood chars prepared after slow carbonization for varying soaking periods (h) (a) 1; (b) 3; (c) 5.

rapid carbonization (Fig. 11a) looks clean and clear of any deposit but with some evidence of fractured fibres, whereas the surface looks dull in the case of wood char obtained from slow carbonization (Fig. 11b), as if some fine particles have been deposited over it. Such deposits are more clearly visible in the SEM of a transverse section of 1000 °C acacia wood char prepared under slow carbonization, as shown in Fig. 12.

4.4.3. Soaking time

An increase in soaking time while carbonizing wood samples under a slow heating–cooling cycle at temperatures of 800 and 1000 °C was found to affect the surface characteristics of the wood chars produced. The SEMs of chars produced after slow carbonization of eucalyptus wood samples at a temperature of 1000 °C for different soak periods, in the range 1–5 h, are shown in Fig. 14. Fusion (sintering) of the adjacent fibres resulting in the formation of compacted (densified) masses are clearly visible. Comparison of these micrographs indicates that the extent of compactness (i.e. fusion of fibres) increased with an increase in soaking time. Similar results were also obtained for carbonization at 800 °C. The decrease in porosity with an increase in soaking time for 800 and 1000 °C slowly carbonized acacia and eucalyptus woods, reported in [13], can be explained by the shrinkage of pore structure occurring through sintering of the fibres.

4.4.4. Wood species

In the present SEM study the surface characteristics (pores, fibre morphology, etc.) of wood chars appeared to be unaffected by the change in plant species (i.e. type of wood). Both the wood species, namely acacia and eucalyptus, exhibited similar behaviours in the development of pores, cracks and voids, etc., during carbonization. Almost all the morphological properties of acacia and eucalyptus wood chars, prepared under identical carbonization conditions, were quite similar, but the appearance (shape) of their cell cross-sections under SEM looked different. In Figs 5a and 13a, a polygonal cell cross-section in eucalyptus wood chars can be seen, whereas Figs 12b and 13b show an (approximately) elliptical cell cross-section in acacia wood chars.

5. Conclusions

The carbonization process of acacia and eucalyptus woods is characterized by the change in their structural morphology, with the extent of change depending on the carbonization conditions. The main observations made from the present study are:

1. under identical carbonization conditions, both the acacia and eucalyptus wood species, in general, underwent similar morphological changes;
2. the fibrous structure of wood is conserved during the process of slow carbonization even at a high temperature of 1200 °C, while the rapid carbonization at temperatures above 600 °C breaks the fibrous structure;
3. both carbonization processes, in general, decreased the pore size in resultant chars with an increase of carbonization temperature;
4. rapid carbonization at temperatures of 800 and 1050 °C led to the production of chars with cracks and voids in their structures, and the extent of crack formation increased with an increase in temperature, whereas the chars prepared from slow carbonization were found to be free from cracks and voids;
5. slow carbonization resulted in pyrolytic carbon deposition in the resultant wood char structures, whereas no such deposited particles were noticed in the wood char structures prepared under rapid carbonization;
6. at a particular carbonization temperature, the extent of fusion of adjacent fibres increased with an increase in soaking time;
7. eucalyptus wood chars were found to have a polygonal cell cross-section, whereas acacia wood chars had an (approximately) elliptical cell cross-section.

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Received 25 June 1992
and accepted 16 December 1993