STRUCTURAL CHANGES ON CHARRING WOODS OF DICHROSTACHYS AND SALIX FROM SOUTHERN AFRICA: THE EFFECT OF MOISTURE CONTENT

by

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Summary

Air-dried and saturated cubes of fully developed wood of *Dichrostachys cinerea* (Leguminosae) and *Salix subserrata* (Salicaceae) were charred for 60 minutes at 400°C. An initial increase in moisture content caused few structural alterations in *Salix* but in *Dichrostachys* it resulted in considerable ray distension and massive deformation of non-gelatinous fibres. An attempt is made to correlate these observations with the physical and chemical changes known to occur during wood pyrolysis.

*Keywords:* Pyrolysis, charcoal, fibres, Swaziland.

Introduction

During the past decade, scanning electron microscopy has been used increasingly to elucidate the structural details retained within charred plant remains which survive in many geological and archaeological deposits. A detailed analysis of Recent and Holocene charcoal fragments excavated from a rock shelter in northeastern Swaziland has recently been described (Prior & Price Williams, 1985). During the course of this work it became apparent that successful identifications of the ancient material were dependent upon a retention of the maximum number of anatomical features by charred modern comparative woods. The structural changes observed on charred two southern African woods of highly divergent anatomical character at a range of temperatures from 300 to 800°C are discussed by Prior and Alvin (1983). Since 1983, the routine temperature selected for charring reference material, either 400 or 450°C, has enabled damage to be minimised and to be clearly distinguished from anatomical characters of taxonomic significance.

It is the pyrolysis of wood, a process of thermal decomposition in the absence of oxygen, which results in the formation of charcoal. Recent research dealing with the industrial pyrolysis of wood shows that the type and extent of the reactions involved are dependent upon many factors apart from charring temperature, such as moisture content and the presence of inorganic salts or ash. Cope and Chaloner (1985) have applied some of this growing volume of research to an assessment of the origin of fossil charcoal from the geological record. An increased knowledge of the factors affecting wood pyrolysis should also enhance our understanding of conditions obtaining in ancient fires on archaeological sites.

The research described in the present paper seeks to investigate the relationship between the moisture content of *Dichrostachys* and *Salix* and the damage caused by charring. Reference woods charred in this laboratory to date have been air-dried and have therefore had moisture contents of approximately 8–13%. Dead branchwood still attached to the trees was probably the chief source of fuelwood on ancient Swaziland sites. This would be consistent with current practice in areas of southern Africa which still have adequate fuelwood supplies (Openshaw, 1974; Jelenic & Van Vegten, 1981). The variation in moisture content of such a fuel source may be considerable, according to whether wood was collected during the dry winter or wet summer season.

Materials and Methods

The differences in anatomy and habitat between the two woods have already been described (Prior & Alvin, 1983). The cubes used in the experiments reported in this paper were cut from branches approximately 3.5 cm in diameter. Both the *Dichrostachys* and *Salix* trees were growing in the bushveld of Swaziland, the former in semi-arid deciduous woodland and the latter on river alluvium. Cubes of 1 cm were cut from the same radius of the single branches of each wood, so that the anatomy was as comparable as possible. Gelatinous fibres, known to be common in *Dichrostachys*, vary considerably in both their transverse and longitudinal distribution throughout a branch (Hughes, 1965). In the case of *Dichrostachys*, where sap- and heartwood can be clearly distinguished, some of each was included in each cube. A total of twelve cubes were cut from each wood and their further treatment was as...
follows: 1) six cubes were placed in distilled water in a vacuum pump and air was removed by applying a vacuum of 100 kPa for 30 minutes; they were then saturated by replacing under a similar vacuum for a further hour; 2) six air-dried cubes were left untreated.

Three cubes from each batch were subsequently used for replicated moisture content, density and porosity determinations. Calculations were based on the following formulae conventionally used by wood technologists, as described by Siau (1984).

\[
\text{moisture content (MC)} = \frac{\text{weight of wood + water} - \text{oven dry weight}}{\text{oven dry weight}} \times 100\%
\]

\[
\text{density (D)} = \frac{\text{oven dry weight}}{\text{weight of displaced volume of water when sample is at its maximum volume}} \text{ g/cm}^3
\]

\[
\text{porosity} = 1 - D \ (0.667 + 0.01 \text{MC}) \times 100\%.
\]

The remaining three cubes from each batch were charred by embedding in crucibles of acid purified sand (40–100 mesh) to exclude air and heating in the centre of a Gallenkamp muffle furnace at 400°C ± 5°C for 60 minutes. The temperature of the furnace was attained at least an hour before the start of charring and after each 60 minute period the crucibles were removed and allowed to cool. The cold wood cubes were then removed from the sand, fractured and prepared for SEM as previously described (Prior & Alvin, 1983). Three fracture planes (TS, RLS and TLS) of all three replicated cubes were examined by SEM and sample areas photographed.

**Results**

The anatomical divergence of the two woods is clearly reflected in the results shown in Table 1. Air-dried *Dichrostachys* has a higher moisture content than *Salix*, due mainly to the strongly hydrophilic property of its abundant gelatinous fibres. The physical properties of the wall layers of such fibres together with the high density of the wood, account for the lower values obtained for saturation moisture content and porosity in *Dichrostachys*.

**Effects of charring**

There was good agreement between the replicates in that each cell type generally behaved in the same way. There was considerable variation, however, in the distribution of the tissues between the cubes. The following description applies to all cubes in the case of each wood.

*Dichrostachys cinerea* (L.) Wight & Arn. **Macroscopic appearance of the cubes.** — The marked difference in the appearance of the transverse faces of charred air-dried and saturated cubes is shown in Figures 1 and 2. Whereas saturation of the samples before charring causes additional shrinkage of at least 10% and a substantial increase in the number and extent of small radial fissures and larger radial splits, the air-dried samples show a somewhat uniform reticulum of small tangentially and radially orientated fissures. An increase in the moisture content of the wood before charring therefore results in far more severe distortion.

**Changes observed under SEM.** — The effect of an increased moisture content on the structure after charring can be seen in Figures 3–12 inclusive. Rays and fibres only are markedly affected, two forms of damage being clearly vis-

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Table 1. Moisture content, density and porosity determinations: average values obtained from three replicates.

<table>
<thead>
<tr>
<th>Species</th>
<th>Type</th>
<th>MC (%)</th>
<th>D (g/cm³)</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>Dichrostachys cinerea</em></td>
<td>air-dried</td>
<td>10.9</td>
<td>1.0</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>saturated</td>
<td>30.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td><em>Salix subserrata</em></td>
<td>air-dried</td>
<td>7.9</td>
<td>0.5</td>
<td>65</td>
</tr>
<tr>
<td></td>
<td>saturated</td>
<td>139.3</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
ible. Firstly, the gaping radial splits apparent in cubes charred after saturation are typically associated with rays, as noted in the previous paper (Prior & Alvin, 1983). The rays, which are most commonly multiseriate, no doubt represent planes of weakness along which rupture occurs in response to contraction of the abutting fibrous tissue. In cross fracture the large splits sometimes appear to occur entirely within fibrous tissue, as seen in Figures 9 and 10. In tangential view, however, it can be seen that the splits initially opened alongside or through the rays, but were sufficiently large to extend well above and below them (Fig. 8). The less extensive fissures seen in the air-dried material seldom extend in this way (Fig. 7). The small tangential fissures apparent in all cubes tend to occur through vessels and the associated aliform or subconfluent parenchyma (Fig. 10). Secondly, in all cubes, ray damage is most frequent and severe in those areas where the rays concerned are flanked by fibres which are predominantly non-gelatinous (Figs. 3, 4, 9). This suggests that the contraction of non-gelatinous fibres is greater than that of gelatinous ones and that this contraction is increased by a high moisture content of the wood before charring. The observations are confirmed by Figures 5, 6, 11 and 12. In those cubes charred in an air-dried condition, both fibre types generally retain their individuality (Figs. 5, 11) although a few non-gelatinous fibres in close proximity to distended rays show somewhat more distortion than those elsewhere (Fig. 3). An increase in the moisture content of the wood prior to charring results in massive contraction of the non-gelatinous fibres with extreme fusion and homogenisation of the walls such that the individuality of the cells is virtually indiscernible (Fig. 6). The charring of gelatinous fibres on the other hand, is comparatively unaffected by high initial moisture content (Fig. 12). Their distortion is somewhat increased and small disruptions more frequently occur (Figs. 9, 12). The outer wall layers become thicker and the cell lumina are more frequently obliterated (Fig. 12) but extreme cellular deformation never occurs. In Figures 4 and 6 individual gelatinous fibres can still be clearly identified flanking the ray at the top of the photograph.

**Salix subserrata** Willd.

Differences between cubes charred in an air-dried and saturated condition are far less evident than in *Dichrostachys*. An increase in the moisture content causes a slight but probably significant increase in the overall degree of shrinkage. This is reflected microscopically in an increase in the number of vessels per unit area as seen in cross fracture (Figs. 13, 14). No other structural differences are apparent.

Fig. 1–2. *Dichrostachys cinerea.* – 1: Transverse plane of cube of wood charred air-dry; x c. 6. – 2: Similar view of a specimen cut from the same branch charred saturated; x c. 6.
Discussion and Conclusions

Two main points emerge from the results described:

1. The two woods react very differently though in both cases, saturation before pyrolysis increases shrinkage.

2. Charring of saturated Dichrostachys cubes at 400°C for 60 minutes results in the formation of large radial splits associated with the rays together with anatomical changes in the fibrous tissue. The changes conform closely to some of those described by earlier workers using SEM techniques to elucidate charcoal structure, notably Kollman and Sachs (1967), McGinnes et al. (1971, 1974) and Prior and Alvin (1983).

Since the two woods responded differently when oven-dried cubes were subjected to different temperatures of charring (Prior & Alvin, 1983), then the assumption must be that it is primarily characteristics of the wood which govern charring behaviour and it is the interaction between these and factors such as time and temperature of charring, initial moisture content of the wood and size of the cubes which determines the changes observed.

Salix, a light wood of low lignin content, has thin-walled vessels, homogeneous, comparatively thin-walled fibres, numerous uniseriate rays and rather little axial parenchyma. Saturation of the wood, resulting in a very high moisture content of 139.3%, suggests that all the tissues took up a large amount of water. On charring, the narrow rays and homogeneous fibrous tissue ensured that the stresses set up were even, resulting in a general shrinkage without much fissuring or splitting.

Dichrostachys in contrast, has thick-walled vessels containing gum, thick-walled fibres of two types, a comparatively large amount of paratracheal axial parenchyma and rays which are predominantly multiseriate. The saturated wood has a low moisture content of 30.7%, due to the density of the fibrous tissue. Furthermore, since a high proportion of the fibres are gelatinous and therefore retain a considerable amount of water in the air-dried state, they cannot absorb much more. On charring, water will probably be lost first from the vessels and the thin-walled ray and axial parenchyma cells. The uneven stresses thus set up will produce the initial fissuring in the tangential and radial planes. The position of the major splits which may occur later is related to the distribution of large blocks of non-gelatinous fibres which, as a tissue, contract more than the gelatinous fibres. Although the sequence of events occurring within the different regions of the Dichrostachys cubes during the 60 minutes of charring are no doubt complex, changes seen in the cellular structure of the fibre walls can be discussed in the light of some recent work on pyrolysis. Literature concerning the thermal degradation of wood components is reviewed by Beall and Eickner (1970) and the chemistry of pyrolysis has been described most recently by Shafizadeh (1984). As Slocum et al. (1978) point out, wood behaves as a mixture of its three major polymers in response to heat. Early in the pyrolytic process (100–200°C), water is evolved. Hemicelluloses decompose between 200 and 300°C, followed by celluloses above 240°C. Lignin, which merely undergoes a rearrangement of functional groups within this temperature range, begins to decompose at temperatures above 280°C. Under the charring regime described in this paper, all three constituent polymers will undergo some decomposition and some of the volatiles produced will presumably condense on or near the surface of the cubes on cooling. Since changes in the appearance of fibre walls are seen in freshly exposed fractured planes, they cannot be explained by secondary deposition alone but must also be due to physical and chemical changes. As Parham and Gray (1984) point out, when considered as a percentage of total wood, gelatinous fibres have more cellulose, less lignin and fewer xylose residues than normal wood. On charring cubes of Dichrostachys at 400°C, the gelatinous layer pulls away from the remainder of the wall and being high in celluloses and hemicelluloses, depolymerisation and shrinkage of this layer is considerable. The remainder of the heterogene-

Fig. 3–8. Dichrostachys cinerea. — 3: Transverse fracture of specimen charred air-dry showing relatively restricted radial ray distortions; x c. 100. — 4: Similar view of specimen charred saturated showing massive radial cracks and associated highly contracted and homogenised non-gelatinous fibres; x c. 100. — 5: Non-gelatinous fibres of specimen charred dry seen in transverse fracture; x c. 800. — 6: Predominantly non-gelatinous fibres from specimen in Figure 4; note that the gelatinous fibres (adjacent to ray at top) retain the individuality of the cells much better than the non-gelatinous fibres; x c. 400. — 7: Tangential longitudinal fracture of specimen charred dry showing radial openings typically confined to the rays and confined within their height; x c. 100. — 8: Similar view of specimen charred saturated; note wide openings not restricted to height of rays; x c. 100.
ous wall is comparatively thin and is therefore capable of retaining a certain amount of plasticity when subjected to stresses associated with distortion of the surrounding tissues. Conversely, the non-gelatinous fibres of *Dichrostachys* have thick, less flexible, more highly lignified walls, so that this tissue, on contraction, will exert more strain on adjacent cells. The influence of the differing chemistry of fibre walls on subsequent charring damage is also clearly shown by Connor and Salazar (1985). When earlywood and latewood samples of *Eucalyptus delegatensis* with an initial moisture content of 10.6% were charred in a flow of preheated nitrogen at 400°C, it was the thicker walled, more highly lignified fibres of the latewood which deformed most. Analysis of the lignin residues from similar samples in a partially pyrolsed condition indicated that changes in the lignified walls were due either to partial lignin breakdown or to a reaction between lignin and some of the breakdown products of celluloses and hemicelluloses.

One final factor may contribute some of the responses to charring in *Dichrostachys*. The wood is known to contain considerable amounts of prismatic crystals believed to be calcium oxalate, within axial strands of chambered parenchyma cells. Gmelin (1961) states that decomposition of the oxalate to calcium carbonate and carbon monoxide begins to occur at 370°C. The addition of sodium and potassium carbonates to wood samples before pyrolysis is known to cause enhanced cellular degradation and an increase both in the gasification and also in charcoal yield (Hawley et al., 1983; Zaror et al., 1985). In the context of the present investigations it may well be that the maximum charring damage in *Dichrostachys*, noticeable at temperatures above 700°C using oven-dried wood (Prior & Alvin, 1983) and at 400°C using saturated wood, may have been considerably enhanced by the breakdown products originating from the crystals.

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References


