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SOME STRUCTURAL CHANGES OBSERVED IN THE TRANSFORMATION OF WOOD INTO CHARCOAL¹

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ABSTRACT

On the basis of measurements of microtomed cubes of white oak and on resultant charcoal, dimensional changes occurring on the conversion of wood into charcoal are: tangential, -25.68%, radial, -15.45%, and longitudinal, -11.43%. Light microscopic examination of charcoal reveals residues of combustion present in cell cavities. Electron microscopic examination indicates that the original fibrillar arrangement of the cell wall has been replaced with a smooth, "amorphous-appearing" wall structure.

INTRODUCTION

Charcoal manufacture has been significant in Missouri since the advent of the iron industry about 1815 in Ironton. Missouri has 64 charcoal operations, which consume approximately 150,000 cords of wood annually (McGinnes 1965). In view of the contribution of the industry to the state's economy, studies are in progress to improve the efficiency of the process, hopefully with the objective of securing better control of charcoal quality and yield. Recent concern over environmental pollution has also given

emphasis to a research effort in pollution control in charcoal manufacture because it results in loss of volatiles to the atmosphere and the formation of tars and other non-gaseous byproducts. Charcoal was originally the byproduct of the destructive distillation-of-wood industry; today charcoal is the main product and the distillates are not marketed.

The most popular kiln in operation in the state is the Missouri-type shown in Fig. 1. It has a capacity ranging from 45-50 cords of wood. The design and construction details of the Missouri kiln are presented in the work of Jarvis (1960). A kiln of this design was used for the study of certain structural changes in the transformation of wood into charcoal.

Schaffer (1966) has presented an excellent survey of published information related

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FIG. 1. A Missouri-type charcoal kiln. Burn is in progress. Majority of charcoal in the state is made in a kiln of this design.

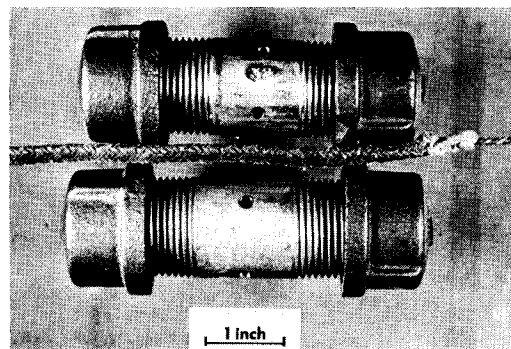


FIG. 2. One-inch-diameter steel pipe used to hold wood samples during charcoal run. Thermocouple wire was wrapped around the two capped pipes at each location.

to the charring of wood. However, no published studies pertaining to anatomical and ultrastructural changes of wood during commercial charring are available.

It is the intent of this paper: (1) to compare cross-sectional, radial, and tangential dimensions of wood and resultant charcoal; (2) to present a microscopic description of wood charcoal structure; and (3) to present exploratory use of X-ray diffraction techniques in describing charcoal structure.

EXPERIMENTAL

Cubes of white oak (*Quercus alba* L.) approximately one-half inch square on any face were cut on a band saw; all six faces were then sanded until the surfaces were smooth. Three adjoining faces (transverse, radial, and tangential) were cut with a sliding microtome to improve further the clarity of wood features. The samples were oven-dried and subsequent (X , R , T) dimensions were obtained from the microtomed surfaces of wood by photographing them with a millimeter scale and then projecting the slide upon a screen for measurement. Samples had an average moisture content of 3.7% when they were photographed.

Ten such cubes were prepared and then placed, one each, into ten perforated, one-inch-diameter pipes for charring. The capped pipes, with identifying sample num-

bers, are shown in Fig. 2. Two such samples were placed at each of five locations within a Missouri-type kiln and charred commercially. After charring, samples were dried, weighed, and rephotographed; and then X , R , and T dimensions of resultant coal were measured. The charcoal pieces had an average moisture content of 4.6% when they were photographed. During charring, temperature was recorded every six hours, using iron-constantan thermocouples; temperature was recorded throughout the kiln run from initial firing through the cooling-down period.

Microscopic examination of charcoal presents some difficulty because the material is extremely brittle and therefore hard to section. For light microscopy some success was obtained using polyethylene glycol (average molecular weight 1300–1600) as an embedding media. Sections were cut with a sliding microtome. For electron microscopy, a methyl cellulose film was formed on longitudinal faces of wood and charcoal, according to the technique of Lasko (1957). The dry film was stripped with a length of cellulose tape, and a carbon layer approximately 200 Å thick was deposited on the film. The plastic film was dissolved; the carbon replica was dried and shadowed with chromium at an angle of 5 to 7° for subsequent examination with the electron microscope.

X-ray analyses were made of graphite, activated wood charcoal, and conventional charcoal using a G. E. XRD-6 X-ray instrument. Filtered molybdenum radiation ($K\alpha$) of $\lambda = 0.710 \text{ \AA}$ was used for diffraction studies. Diffraction intensities were recorded continuously over an exposure angle range from 5 to 50° .

In this study, the term charcoal, or conventional charcoal, refers to wood charcoal manufactured for use in indoor or outdoor cooking either as lump charcoal or briquettes. The yield of such charcoal is usually about 40% of the original wood weight, whereas activated charcoal used for chemical purposes is usually obtained in about 16% yield (based on original wood weight).

RESULTS

At the five kiln locations (near the door in this run), maximum temperatures were reached from 142 to 160 hr after ignition of the charge. Yields, for these five locations, ranged from a high of 41.5% to 34.5%. Corresponding maximum temperatures obtained were 530 F and 750 F. This inverse relationship between maximum temperature and yield has been established in other studies. The maximum temperature range (530 F–750 F) and the actual high value (750 F) obtained for the samples are both somewhat lower than average because of sample location within the kiln. The samples were placed near the top of the charge, by the front door, to avoid damage from possible shifting of the charge during the burn and to allow their easy removal after the kiln was opened.

Dimensional change

The percentage changes for the tangential, radial, and longitudinal dimensions of the samples resulting from the conversion of wood to charcoal are: -25.68% , -15.45% , and -11.43% , respectively. Moisture contents of the samples at time of measurement were 3.7% and 4.6% for wood and charcoal, respectively. From the data on percentage shrinkage, the T/R shrinkage ratio is 1.73 on conversion of wood to coal. The T/R

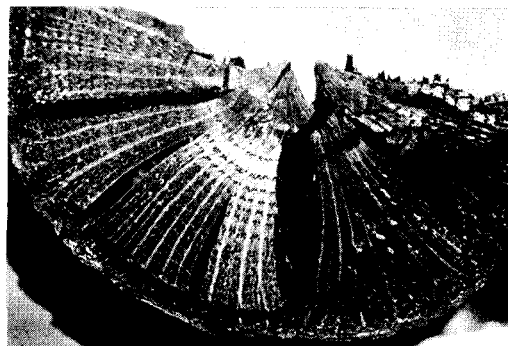


FIG. 3. Cross-sectional view of oak charcoal. "Bark" is still on sample.

ratio for shrinkage of white oak from green to oven-dry is 1.70 (Panshin, de Zeeuw, and Brown 1964). The similarity of these ratios is of interest, but may simply be a coincidence.

The transverse shrinkage of wood to coal, although 2 to 3 times that which occurs on moisture removal from oak, is not as striking as the longitudinal shrinkage. Longitudinal shrinkage on conversion of oak to charcoal approaches a 100-fold increase compared with longitudinal shrinkage of wood from the green to oven-dry state.

Volumetric shrinkage for the 10 samples averaged 44.6% and is representative of volumetric loss on conversion of oak to conventional charcoal.

Anatomical examination

That wood retains its gross features sufficiently for species identification on a macroscopic level is well known, as shown in Fig. 3. The most characteristic macroscopic feature, outside of color, is the presence of severe radial splits similar in appearance to those in honeycombed wood.

The normal circular shape of the large springwood pores in oakwood changes to an elliptical one, the small axis of the ellipse being in the tangential plane of the wood. This condition was present to a varying degree in all samples examined.

Light microscopic studies added little to anatomical studies of charcoal; in those instances where cross sections could be

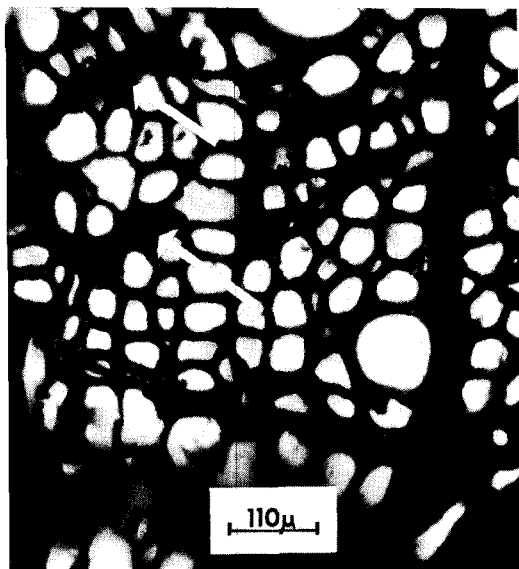


FIG. 4. Cross section of oak charcoal. Arrows indicate concentrated zones of residual "tars" within the former wood cell structure.

made, residual tars (or other byproducts of combustion) could be found within the cell structure. This condition is shown in Fig. 4. Cell-wall details are masked and not discernible.

The electron microscopic studies were more fruitful. The original fibrillar arrangement of wood has been destroyed, resulting in a smooth, amorphous-appearing wall structure as seen in longitudinal section. Pits are still discernible in charcoal; but these are the only wall features of the original wood still recognizable. Numerous folds in the cell walls are apparent. These may be both horizontal and vertical to the longitudinal axis of a cell and are assumed to be the result of the significant shrinkage involved. Figures 5 and 6 are representative of the features of pit structure and cell walls observed in this study. Results of the present study, plus the findings of Kollmann and Sachs (1967) on wood subjected to elevated temperatures, allow some speculation on the sequence of anatomical changes during the transformation of wood into charcoal. At first, there appears to be a preferential alteration (softening) of accessible, lignin-

like structures. As the charring temperature is increased to maximum, the microfibrillar structure of the wood is destroyed. This implies degradation of cellulose and the hemicelluloses accompanied by rearrangement of carbon into a structure approaching that of graphite. It is also at this time that shrinkage may become extensive. It is important to note, however, that the resultant charcoal retains a woodlike appearance despite such an intensive ultrastructural alteration of the original wood.

X-ray analyses

The carbon arrangement in graphite has been well established. Wood charcoal is actually microcrystalline in character with such crystallites (of graphite structure) oriented in random fashion (Moeller 1955). Conventional charcoal also includes residual tars and volatile matter. Transmitted-light microscopic examination of cross sections of wood charcoal, or incident light microscopy of wood charcoal surfaces, will frequently reveal such materials in cell lumens.

X-ray diffraction analyses may provide a research tool for differentiating varying qualities of charcoal produced in a Missouri-type kiln. The operation is a "batch-type" (as opposed to continuous) and results in a considerable spread of maximum temperatures within a kiln charge (400–1500 F based on current studies). Wood charcoal can have its random, microcrystalline structure ("amorphous") transformed to that of graphite with sufficient heating (Ubbelohde and Lewis 1960). Figure 7 presents X-ray diffraction patterns for conventional charcoal (oak), activated charcoal, and graphite. The change from "amorphous" (disordered) to "crystalline" (ordered) structure is apparent. Hopefully, future studies designed to improve charcoal yield and quality can utilize X-ray diffraction analyses, coupled with maximum temperature and per cent yield data, to describe the variation in charcoal properties manufactured in a Missouri-type kiln. One objective would be to reduce the spread in yields within a charge. Current findings indicate

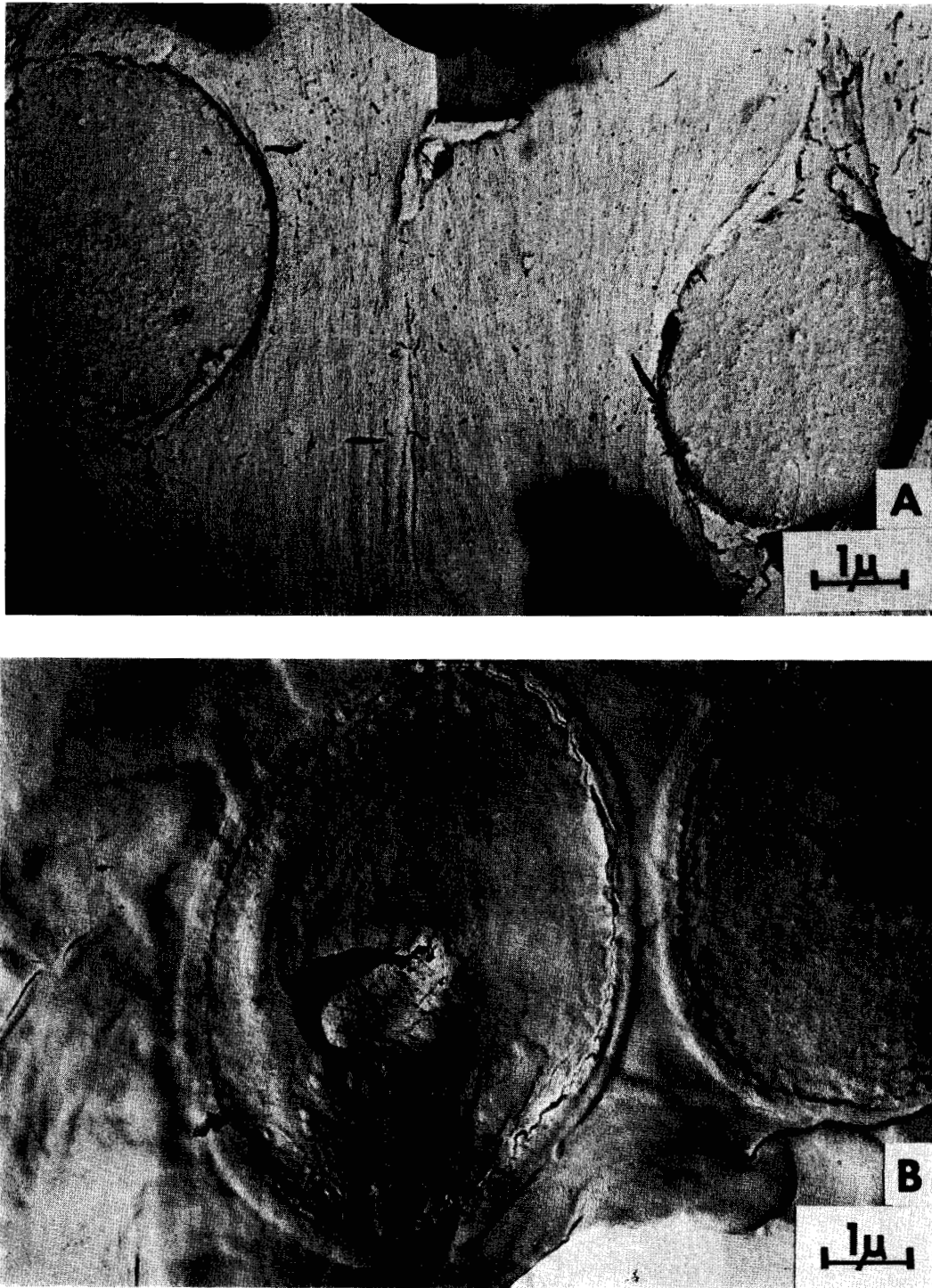


FIG. 5. Electron micrographs of radial view of oak wood (A) and charcoal (B) showing transition from fibrillar structure of wood cell wall to the "amorphous" wall structure of charcoal. Pits are still identifiable in charcoal.

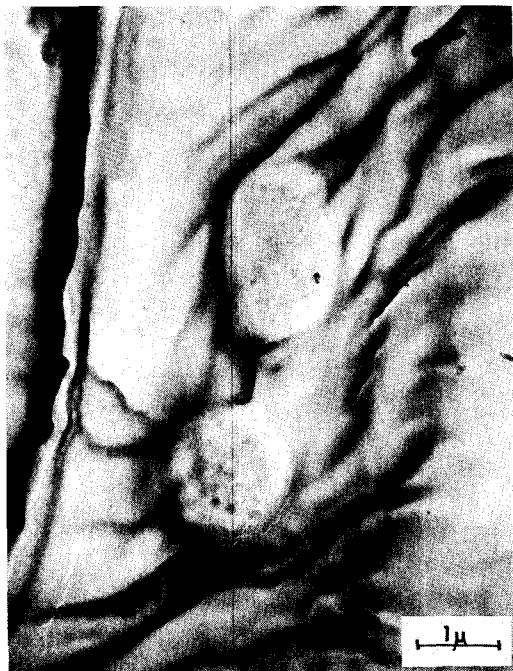


FIG. 6. Electron micrograph, radial view, of wood charcoal. Pit structures still apparent. Microfibrillar structure of original wood cell wall is replaced by smooth, "amorphous" wall displaying marked folds as a possible result of shrinkage on conversion of wood to charcoal.

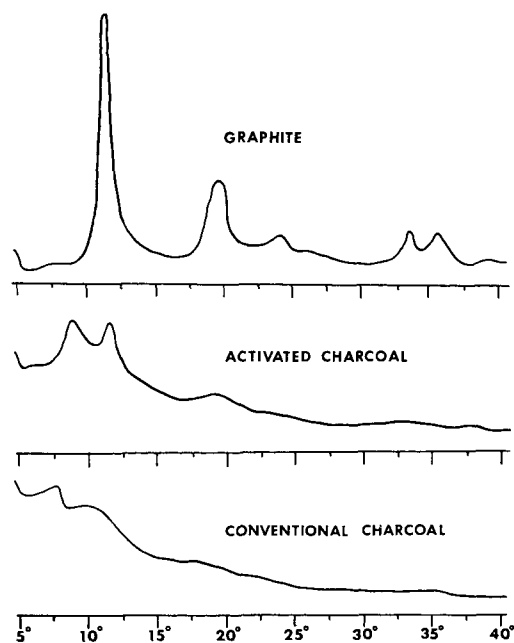


FIG. 7. Three X-ray diffraction diagrams of graphite, activated charcoal, and conventional charcoal over exposure angle range of 5° to 40°. Vertical axis omitted as it compares samples on a relative intensity basis only. Note similar locations of "peaks" (vicinity of 10° and 20° angle) for graphite and activated charcoal.

that both small quantities of coal (19% yield) approaching the properties of activated charcoal as well as some charcoal (50% yield) in early stages of conversion from brands may be present in the same run.

SUMMARY AND CONCLUSIONS

On the basis of measurements of microtomed cubes of white oak and on resultant charcoal, dimensional changes occurring on the conversion of wood to charcoal are presented for the tangential, radial, and longitudinal planes of wood. Loss in dimension for these three planes was: -25.68%, -15.45%, and -11.43%, respectively. The T/R ratio of 1.73 is quite similar to T/R of 1.70 for drying of white oak. Most marked dimensional change, in comparison with drying of wood, is in the longitudinal plane.

Light microscopic studies indicate the presence of residual tars and other residues of combustion in cell cavities. Some residues are probably in the cell-wall structure also. This condition exemplifies conventional charcoal (yield about 40%). The electron microscope reveals that the original microfibrillar orientation of the cell wall has been destroyed in charcoal manufacture, and replaced with a smooth "amorphous-appearing" wall structure. Folds running either parallel to, or across, the longitudinal cell-axis are also visible. These are assumed to be a result of the large shrinkages, in both transverse and longitudinal planes, of wood in conversion to charcoal.

X-ray data of two different forms of wood charcoal and of graphite indicate that diffraction studies could be a useful tool in differentiating charcoal qualities.

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