PRESSURE-REFINED FIBER FROM LOW-GRADE SOUTHERN HARDWOODS[1]

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ABSTRACT

Pressure-refined fiber for medium-density fiberboard was made from five species of low-grade southern hardwoods. The fiber was evaluated for extractive content, pH, alkaline buffering capacity, ash content, bulk density, and morphology. Experimental variables included three refiner plate settings (0.064-, 0.127-, and 0.192-cm) and three raw material types. These were: 1) main stem with bark for each species, 2) main stem without bark for one species, and 3) whole tree for a mixture of all five species.

Results indicated that pH, alkaline buffering capacity, and ash content were independent of refiner plate settings. Species differences in these properties could be critical to the subsequent fiberboard processing. Although surface characteristics were similar for all five hardwood fibers, bulk density increased and the fiber became finer as refiner plate setting was reduced. Inclusion of bark in the furnish influenced the properties of the refined fiber.

Keywords: *Quercus falcata, Quercus alba, Liquidambar styraciflua, Carya tomentosa, Nyssa sylvatica,* pressure-refined fibers, pH, fiber bulk density, anatomy, fiber alkaline buffering capacity, extractive content, southern hardwoods.

INTRODUCTION

Medium-density fiberboard (MDF) has experienced a period of rapid growth in the past several years. This growth has been partly due to the wide range of acceptable wood residues that can be pressure-refined to produce a source of suitable fiber furnish. In conjunction with this, there is an urgent need for the South to remove economically and utilize a mixture of small, low-grade hardwoods growing on upland pine sites. Koch (1972) has estimated that 80 million upland acres classified as pine sites are stocked with these hardwoods.

The effect fiber properties have on processing variables is of primary importance in MDF manufacturing. Previous research has concentrated on the relationship between characteristics of the gross wood and the resulting reconstituted wood product. Very little information has been published on the relationships between the inherent properties of pressure-refined fiber and the quality of MDF.

The objective of this investigation was to evaluate those properties of pressure-refined fiber that may be critical in the manufacturing of MDF from five southern hardwoods. Experimental variables included three refiner plate settings (0.064-, 0.127-, 0.192-cm) and three raw material types. These were: 1) main stem with

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TABLE 1. *Data for species used in evaluating pressure-refined fiber.*

Species	Species Code	Specific Gravity[1]	Volume on Pine Sites[2] %	Mixture Composition (oven-dry weight) %
Southern Red Oak (*Quercus falcata* Michx.)	RO	0.52	24	30
White Oak (*Quercus alba* L.)	WO	0.60	17	24
Sweetgum (*Liquidambar styraciflua* L.)	S	0.46	21	22
Debarked Sweetgum	S[D]			
Mockernut Hickory (*Carya tomentosa* Nutt.)	H	0.64	10	15
Black Tupelo (*Nyssa sylvatica* Marsh.)	B	0.46	9	9
Five Species Mixture:				
Stem	M	—	81	100
Whole Tree	M[W]	—	81	100

[1] Values of solid wood based on weight when oven-dry and volume when green as reported by the Forest Products Laboratory, Forest Service, USDA, Agricultural Handbook No. 72. Values are for the xylem only.
[2] Data compiled by Forest Resources Research Work Unit, Southern Forest Experiment Station, New Orleans, LA. Hardwood species and their volumes on pine sites: Alabama, Louisiana, Texas, Oklahoma, 1963–1965.

bark for five hardwood species, 2) main stem without bark for one species, and 3) whole tree (main stem, limbs and bark) for a mixture of all five species. The fiber properties evaluated were extractive content, pH, alkaline buffering capacity, ash content, bulk density, and morphology.

MATERIALS AND METHODS

Material collection and preparation

The fiber investigated was generated from the five major hardwood species growing on pine sites in the Mid-South (Table 1). These species comprise approximately 81% by volume of the hardwoods growing on these sites. In addition to each species, a mixture was made up in proportion to the specific gravity and reported volume of each species growing on pine sites as determined by the Southern Forest Experiment Station (1974). The hardwood species groups are listed in Table 1. The effect of bark was evaluated by investigating sweetgum (*Liquidambar styraciflua* L.) both with and without bark.

A minimum of five trees was randomly selected from each species from the Noxubee National Wildlife Refuge located in North Central Mississippi. The trees were in the 15- to 20-cm class, and were cut to a 7.6-cm top. All woody materials were chipped with a Carthage, 99-cm chipper. Green chips were refined in a Bauer 418 pressurized refiner with a steam gage pressure of 689 kPa and a retention time of 5 min. Approximately 45 kg (oven-dry basis) of each chip type was refined at each of the three plate settings. After refining, all fiber types were dried to a uniform moisture content of 5% in a dry kiln.

Chemical properties evaluation

The solvent extractables in fiber refined with a plate setting of 0.127 cm were determined by conventional soxhlet extraction procedures. The solvent sequence was a 2:1 benzene/ethanol mixture (volume basis), ethanol, and distilled water. Approximately 20-gram fiber samples were extracted a minimum of 4 h with each solvent. The amount of extractives reported was the average of two extractions on an oven-dry, total-fiber basis.

Ash content in each fiber type was determined according to ASTM Standard D-1102-56 (ASTM 1975) with the exception that three determinations were made and the samples were not ground. Grinding was not feasible because of equipment limitations.

The pH and alkaline buffering capacity of the fibers were determined by titrating a 40:1 distilled water/fiber slurry (weight basis) with 0.025 normal sodium hydroxide solution and measuring the electromotive force across a glass-calomel electrode pair. The distilled water was boiled to remove dissolved carbon dioxide. The water/fiber slurry was allowed to soak for 1 h at 22 C prior to measuring the initial fiber pH and conducting the subsequent titrations.

Physical properties evaluation

Fiber bulk density was determined after conditioning each fiber type at 50% relative humidity and 22 C. The conditioned fiber was sifted through a ¼-inch mesh screen into a 1 ft^3 container and weighed. Bulk density was reported as the average of two measurements on an oven-dry basis. Fiber moisture content was determined with a Cenco moisture balance.

Fifteen-gram samples of each conditioned fiber type were classified using a Ro-Tap shaker equipped with 4-, 8-, 18-, 30-, 45-, 80-mesh screens and a pan (minus 80-mesh fraction). (Numbers refer to the US standard sieve sizes; a minus sign represents screen passed by sample and a plus sign represents screen on which sample was retained.) Shaking time was 5 min for each sample. Data for the screen classification were reported as the average values of duplicate samples.

Average fiber lengths for approximately 150 fibers were determined for the classified fiber retained on the 30-, 45-, and 80-mesh screens. A 20 × 28 cm photocopy of a random sample from each screen fraction, magnified 23×, was made with a microfiche reader-printer. Fiber lengths were measured on the photocopy (Short 1976).

Each fiber type was examined with a light microscope at low magnification to determine the general nature of the fiber. Fibers were then examined in a scanning electron microscope equipped with a Polaroid camera to determine minute surface characteristics.

RESULTS AND DISCUSSION

Chemical properties

Extractive content.—The extractive content for each fiber type is shown in Fig. 1. White oak fiber (WO, *Quercus alba* L.) had the highest extractive content, 10.6%, and sweetgum fiber without bark had the lowest content, 5.4%. Sweetgum fiber with bark (S) had a higher extractive content, 8.1%, than sweetgum fiber without bark (S^D). This would be expected considering that bark normally has a higher extractive content than wood (Browning 1963).

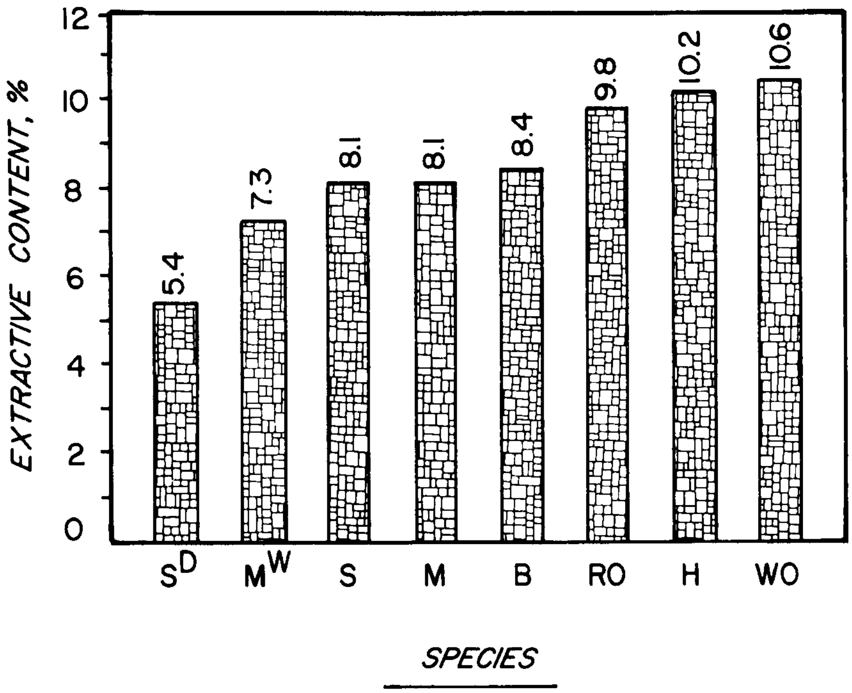

FIG. 1. Extractive content for pressure-refined fiber. Species code listed in Table 1.

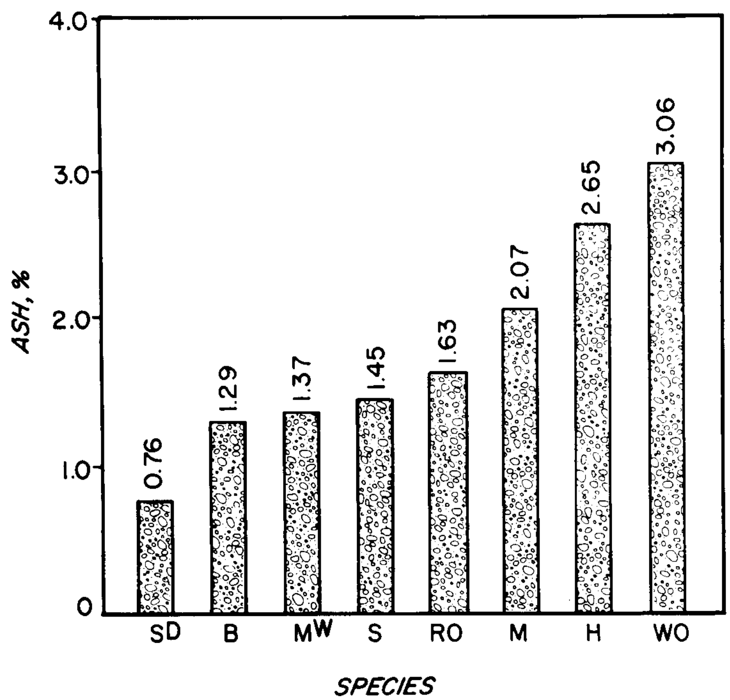

FIG. 2. Ash content for pressure-refined fiber. Species code listed in Table 1.

Results imply that the pressure-refining conditions used in this investigation did not remove a substantial amount, if any, of the extractives of the fiber. One consequence of this finding is that many of the ethanol- and water-soluble extractives of these hardwood fibers have an effect on the gel time of a typical urea-formaldehyde resin used in MDF (Albritton and Short 1977). Thus, fiber types with high amounts of these extractives may cause difficulties in developing sufficient adhesive forces with the urea-formaldehyde resin used in fabricating MDF.

The extractive content of the stem mixture (M) was 8.1% (Fig. 1). The behavior of mixtures often follows the rule of mixtures, which states that the contribution of each constituent to mixture behavior is in proportion to its weighted average. The calculated weighted average for the individual fiber components was 9.5%. Thus, the rule of mixtures overestimated the extractive content by 17%.

Ash content of fiber.—Ash contents for the various fiber types are shown in Fig. 2. No relationship between ash content and refiner plate setting was detected. Values reported are average ash contents for all three refiner plate settings. Ash contents were extremely high for all species. White oak fiber had the highest ash content, 3.06%, and sweetgum fiber without bark had the lowest, 0.76%. The ash content of the debarked sweetgum was lower than the ash content of the sweetgum with bark, 1.45%. This difference was expected because bark normally has a higher ash content than wood.

The cause of the relatively high ash contents of the pressure-refined fiber is not known, but its effect on the abrasiveness of MDF would be substantial. Choong et al. (1974) reported that the mineral contents in bark tissue of hardwoods appear to be much higher than those of conifers. They reported an average ash content of about 0.5% for debarked sweetgum, and an average value of 8.0% for sweetgum bark. Considering that sweetgum has a bark content of 13.4% (Woodson 1976), ash content values reported in Fig. 2 are reasonable.

The ash content of the fiber mixture was 2.07% and the calculated weighted average for the five species was 2.06%. Thus, the rule of mixtures predicted the experimental value with less than 0.5% error.

Fiber from the whole tree mixture had a lower ash content, 1.37%, than observed for the stem mixture. This implies that the limbs and leaves could have contributed additional fiber weight but with proportionally less ash than stem material. Additional research needs to be done to substantiate this implication.

pH and alkaline buffering capacity of fiber.—pH values for the various fiber types are illustrated in Fig. 3. Results indicated that the pH of the pressure-refined fiber is independent of the refiner plate settings. Thus, the reported values are the average measurements for all three settings.

The hardwood fiber types had pH values that could be separated into two distinct groups. Hickory (H, *Carya tomentosa* Nutt.) and black tupelo (B, *Nyssa sylvatica* Marsh.) had the highest pH values, 6.5 and 6.3, respectively. These will be defined as low acidic fiber types. White oak, sweetgum, and red oak (RO, *Quercus falcata* Michx.) had the lowest pH values, 5.1, 5.2, and 4.9, respectively. These will be defined as high acidic fiber types.

The pH of the fiber mixture was 5.1 and the calculated weighted average for the five species was 5.4, a 6% difference.

Previous research has indicated the significance of fiber pH and resin type used in fabricating a fiber product. Matsuda and Sano (1971) found a negative rela-

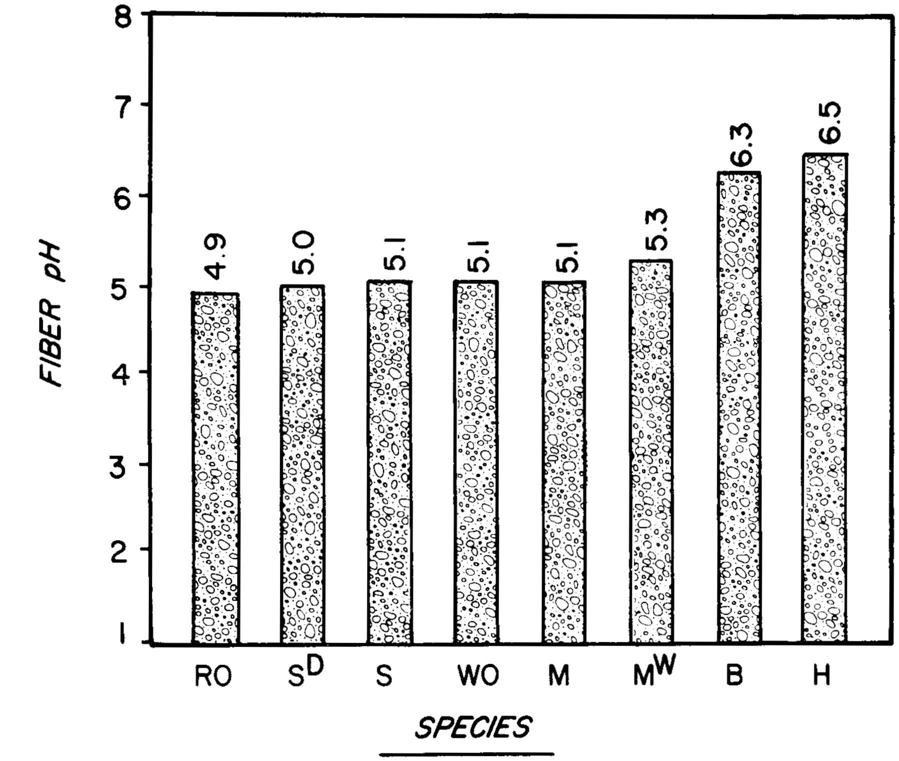

FIG. 3. pH values for pressure-refined fiber. Species code listed in Table 1.

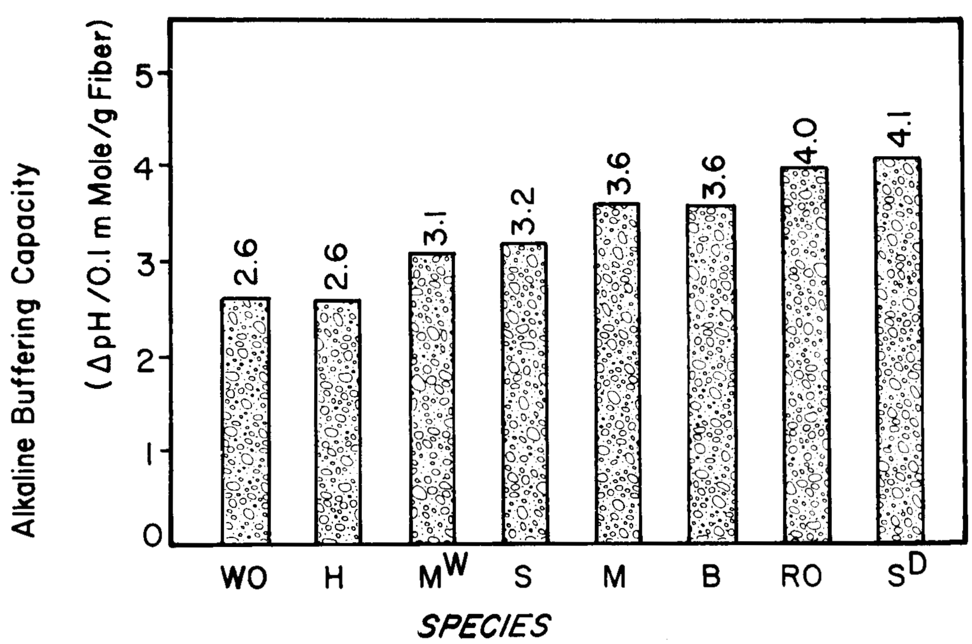

FIG. 4. Alkaline buffering capacity for pressure-refined fiber. Species code listed in Table 1.

tionship between pulp pH and the strength properties of dry-formed hardboard produced with an acid-curing resin. This is important since urea-formaldehyde resin, which is the resin used in processing MDF, is an acid-curing resin. Nelson (1973) found a positive relationship between pulp pH and the properties of hardboard processed with phenolic resin, which exhibits optimum curing at alkaline pH. Thus, it is expected that when considering only fiber pH, the high-acidic fiber types (white oak, sweetgum, and red oak) would be better suited for attaining high quality MDF than the low-acidic fiber types when urea-formaldehyde is used as an adhesive.

The alkaline buffering capacity of the various fiber types was independent of refiner plate settings. Values for the refiner plate setting of 0.127 cm are shown in Fig. 4. Results indicate that both white oak and hickory fiber types had the greatest resistance to increasing pH with the addition of a strong alkali. Red oak and sweetgum fiber types had the least alkaline buffering capacity.

A comparison between the buffering capacity of sweetgum with and without bark showed that the included bark improved the alkaline buffering capacity of the pressure-refined fiber. Apparently, the higher extractive content of bark compared to wood (Fig. 1) is beneficial to the alkaline buffering capacity of the fiber types.

The significance of the alkaline buffering capacity manifests itself with the curing of urea-formaldehyde resin in MDF. Hickory fiber with its high pH of 6.5 and excellent alkaline buffering capacity could cause undercure of the urea-formaldehyde resin and adversely affect MDF properties.

It is to be noted that although white oak and hickory had the best alkaline buffering capacities, white oak was a high-acidic fiber type and hickory was a low-acidic fiber type (Fig. 3). Thus, initial pH values of pressure-refined fiber are not indicative of the fibers' alkaline buffering capacity. The practical aspect of these findings is that an MDF manufacturer must custom tailor the resin formation to insure maximum compatibility between fiber and resin.

The alkaline buffering capacity of the fiber mixture was 3.6 and the calculated weighted average for the five species was 3.2. The rule of mixtures underestimated the alkaline buffering capacity of the pressure-refined hardwood fiber by 11%.

Physical properties of fiber

Fiber bulk density.—Bulk density (oven-dry basis) for the various fiber types is shown in Fig. 5. A statistical analysis, based on a one-way chi-square distribution (95% confidence level), indicated that the smallest refiner plate setting, 0.064 cm, produced fiber with the highest bulk density. Although the largest refiner plate setting, 0.192 cm, produced fiber with the lowest bulk density for all species except sweetgum, there was no significant difference between the bulk densities of fiber refined at the other settings. This may be explained by results of the screen classification, which showed that the smallest refiner plate setting generated a finer fiber.

Results indicated that bulk density was dependent on raw material type. Hickory fiber had the lowest bulk density, 15.55 and 19.08 kg/m^3, for refiner plate settings of 0.192 and 0.064 cm, respectively. Sweetgum fiber with bark had the highest bulk density, 24.69 and 28.37 kg/m^3, at the same plate settings. Sweetgum

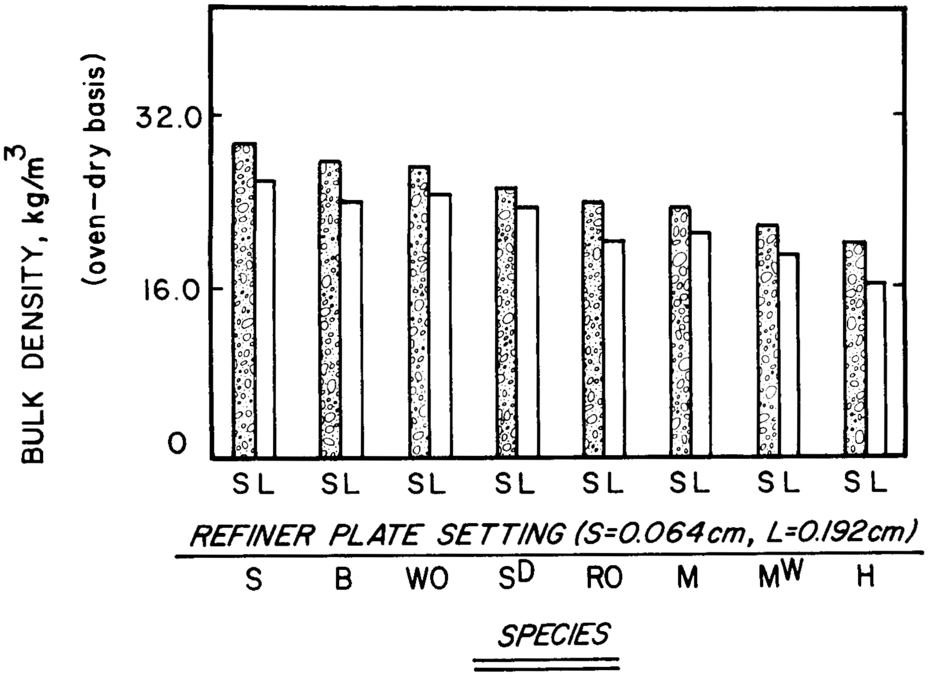

FIG. 5. Bulk density for pressure-refined fiber at two refiner plate settings. Species code listed in Table 1.

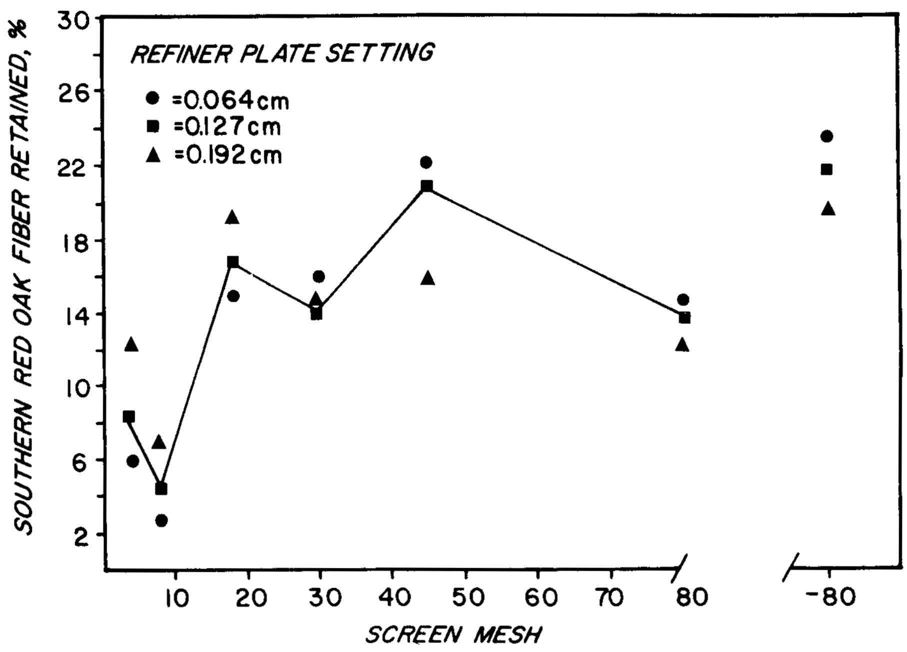

FIG. 6. Tyler Ro-Tap screen classification of pressure-refined southern red oak.

TABLE 2. *Tyler Ro-Tap screen classification of pressure-refined hardwood fiber.*

Species Code[1]	Refiner Plate Setting[2]	U.S. Standard Mesh Designation[3]						
		+4	-4/+8	-8/+18	-18/+30	-30/+45	-45/+80	-80
WO	S	6.9	2.6	13.7	10.9	19.1	17.6	29.2
	M	8.4	3.6	16.5	12.5	18.7	16.2	24.1
	L	7.5	7.0	18.4	10.8	14.8	16.0	25.5
RO	S	6.0	2.7	14.9	15.9	22.1	14.5	23.6
	M	8.0	4.5	16.8	14.1	20.9	13.9	21.8
	L	12.2	6.8	19.1	14.4	15.9	12.1	19.5
H	S	10.5	3.7	18.1	13.8	18.0	14.4	21.6
	M	17.4	6.0	18.6	10.9	14.5	12.8	19.8
	L	25.6	8.5	18.4	9.1	11.5	11.0	16.0
B	S	0.4	2.1	15.2	24.7	17.6	16.2	23.8
	M	1.2	3.9	19.4	19.8	12.7	18.5	24.6
	L	2.2	8.4	23.6	14.2	8.7	17.7	25.1
S[D]	S	0.2	2.9	17.9	25.0	22.1	15.8	16.2
	M	1.1	6.6	22.8	20.9	22.7	12.0	14.0
	L	1.6	5.1	25.5	20.9	16.7	15.2	15.1
S	S	0.1	2.9	16.0	20.5	25.3	15.7	19.6
	M	0.0	3.5	18.8	23.9	20.1	15.4	18.4
	L	0.2	7.7	23.9	18.7	20.5	14.5	14.6
M	S	5.7	3.0	15.0	17.4	19.8	15.0	24.1
	M	10.3	6.7	19.5	15.9	16.0	13.1	18.5
	L	9.5	9.5	17.0	11.1	16.1	14.7	22.1
M[W]	S	7.9	4.3	16.0	17.1	20.5	12.7	21.6
	M	4.8	3.3	15.2	18.1	21.1	14.6	23.1
	L	10.9	7.7	20.7	14.7	14.7	12.7	18.7

[1] Species code listed in Table 1.
[2] S = 0.064 cm, M = 0.127 cm, L = 0.192 cm.
[3] Minus indicates fiber passing through screen; plus indicates fiber retained by screen. Values are the weight percentages.

fiber without bark had substantially lower fiber bulk densities compared to sweetgum fiber with bark. This result could indicate that the bark fiber has a higher density than the wood fiber and/or the refining of bark compared to wood generates a larger fines fraction as evidenced by the screen classification.

A statistical analysis indicated that there is no correlation between the specific gravities of the wood of the various hardwoods investigated and the resulting pressure-refined fiber bulk densities (95% confidence level). The specific gravities of the woods used in the analysis are listed in Table 1. Although the specific gravities used in this analysis were of the wood only, it is believed that inclusion of bark would not affect the observed trend. These findings suggest that the morphology of the pressure-refined fiber is independent of the specific gravity of the whole wood, although fiber morphology is specific for each species. Woodson (1976), using a larger sample size, did find that bulk density of pressure-refined hardwood fiber was greater for the higher specific gravity woods (0.05 level of significance).

The significance of the variations in fiber bulk density of the different furnish types lies in the relationship between bulk density and fiberboard properties.

TABLE 3. *Lengths of fibers (S) and fiber bundles (B) of the various pressure-refined hardwoods retained on three screens. Values in millimeters. Refiner plate setting was 0.127 cm.*

Species Code[1]	Particle Type	U.S. Standard Mesh Designation[2]		
		−18/+30	−30/+45	−45/+80
RO	S	1.5	1.5	1.1
	B	2.1	1.6	1.0
S[D]	S	2.2	1.7	1.2
	B	2.8	1.7	1.0
S	S	1.7	1.7	1.3
	B	1.8	1.6	1.2
WO	S	1.5	1.3	—
	B	2.3	1.4	—
H	S	1.7	1.4	1.0
	B	2.6	1.4	1.0
B	S	1.9	1.6	1.5
	B	2.3	2.0	1.5

[1] Species code listed in Table 1.
[2] Minus indicates fiber passing through screen; plus indicates fiber retained by screen.

Woodson (1976) indicated that bending strength and tensile strength for MDF were negatively correlated to fiber bulk density. Thus, low bulk density fiber types could be expected to produce superior panel products compared to high bulk density fiber types when all other processing variables are equal.

The fiber mixture had an average bulk density of 20.84 kg/m^3 compared to the calculated weighted average bulk density of the five species of 24.05 kg/m^3, a 15% difference.

Fiber screen classification.—The fiber classification data are presented in Table 2. The classification for southern red oak is illustrated in Fig. 6 as a typical example of the results. White oak refined at the smallest plate setting, 0.064 cm, produced the finest fiber type. Approximately 66% of this particular fiber type passed a 30-mesh screen. When refined with a plate setting of 0.192 cm, only approximately 56% of the white oak fiber passed a 30-mesh screen. A listing of the fiber types from the finest to the coarsest is white oak, black tupelo, red oak, sweetgum, and hickory.

If each species had a similar fiber morphology for each fiber screen size, then the coarsest fiber types should have generated the lower fiber bulk densities. Although hickory fiber had the coarsest fiber type and the lowest fiber bulk density, white oak fiber had the finest fiber type but not the highest bulk density (Fig. 5 and Table 2). Variation in fiber density, as well as morphology and/or experimental variation, could account for this weak relationship between coarseness of fiber type and fiber bulk density.

Sweetgum refined with bark generated a finer fiber type compared to sweetgum refined without bark at all three refiner plate settings. Thus, it can be concluded that the fiber type contributed by the bark is of a finer morphology compared to that contributed by the wood.

There was no significant difference between the classification of fiber generated from the whole tree and the stem mixture. This implies that fiber fractions gen-

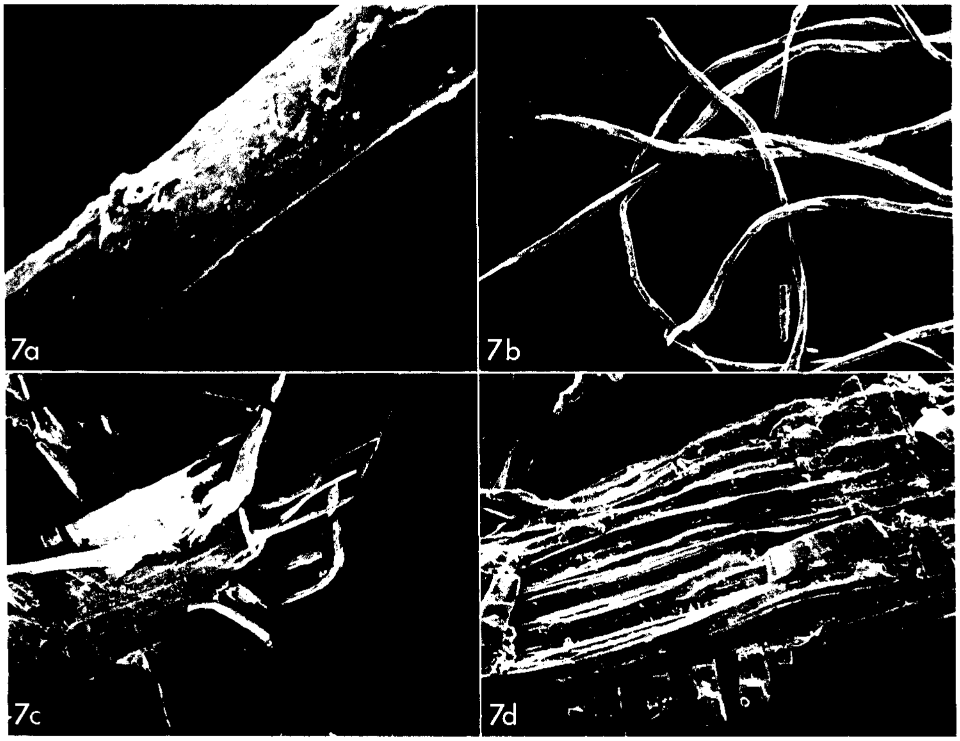

FIG. 7. A. Fiber surface of pressure-refined white oak—1500×. B. Fiber morphology of pressure-refined white oak—150×. C. Frayed fiber bundle of pressure-refined black tupelo—85×. D. Fiber bundle of pressure-refined white oak with attached parenchyma fragments—350×.

erated from pressure-refined branch wood and bark have a morphology similar to that of pressure-refined stem wood and bark. This was verified by microscopic examination as discussed later.

The mixture of the five species had a fiber type with 53.1% passing an 18-mesh screen. This value is similar to the calculated weighted average of 54.9% for the same fiber fraction of each species.

Fiber length.—Table 3 contains a listing of the average lengths of fibers and fiber bundles for the pressure-refined hardwoods refined with a plate setting of 0.127 cm. An analysis of the fiber length data indicated that there was little difference in the fiber or fiber bundle length for material generated at either of the three refiner plate settings. Also, both fiber and fiber bundle length decreased as the screen classification mesh size increased. The average hardwood fiber length ranged between 1.0 mm and 2.2 mm.

Generally, only slight differences in either fiber or fiber bundle length existed between material of the five hardwood fiber types retained on either the 30- (passing 18 mesh), 45-, or 80-mesh screens. Considering the mechanical screening classification procedure used, similar fiber and fiber bundle lengths of a particular screen fraction for all the hardwood fiber types would imply that the fiber morphology for each fiber type would also be similar. This considers only the fiber fractions retained on 30- (passing 18 mesh), 45-, and 80-mesh screens. Thus, it is suggested that differences in fiber bulk density between the five species (Fig. 5) could be due to differences in fiber morphology of material larger than 30 mesh and smaller than 80 mesh, or to differences in the amounts of each fiber size for the five fiber types.

Fiber morphology.—Microscopic examination of the various pressure-refined fiber types revealed that no observable differences existed between the surface characteristics of the various fiber types. The individual fibers had rather smooth surfaces with some surface debris (Fig. 7-A). The fibers did have an overall helical twist (Fig. 7-B). The only noticeable differences in the nature of the fiber types were the relative amounts of fragmented parenchyma and other cell debris included with the fibers and fiber bundles as shown in Fig. 7, frame C and D. Black tupelo especially seemed to have a large amount of fiber bundles with protruding fibers producing a "fuzzy" appearance. This "fuzzy" component would influence the fiber bulk density and could affect the strength properties of MDF.

Results of the microscopic examination are consistent with the findings from the fiber screen classification and fiber length determination. This emphasizes the fact that pressure refining reduces the various hardwoods into fibers of similar physical nature, but that the amount of each fiber mesh size is unique for each hardwood species. Thus, the yield of uniform, whole fibers or fiber bundles ideally suited for MDF from pressure refining is species specific.

Additional research needs to be done to determine the characteristics of the larger fiber mesh sizes (+30 mesh), which have a tendency to form clusters of fibers or fiber bundles. Differences with respect to the morphology of these clusters could account for some of the observed differences in fiber bulk densities.

CONCLUSIONS

Several inherent properties of pressure-refined fiber from five southern hardwoods that are critical to the processing of MDF have been elucidated by this investigation. The conclusions are summarized as follows:

1. Pressure refining does not substantially affect the extractive content of the hardwood fibers evaluated.
2. Ash content of the pressure-refined hardwoods (bark included) is extremely high, ranging from 1.29% for black tupelo to 3.06% for white oak.
3. The pH of pressure-refined hardwood fiber ranges from 4.9 for southern red oak to 6.5 for mockernut hickory and is not influenced by the refiner plate settings used in this study.
4. Pressure-refined hardwood fiber exhibits a wide range of alkaline buffering capacities. Buffering capacity of sweetgum increased with the inclusion of bark. Fiber pH is not indicative of its alkaline buffering capacity.
5. Pressure-refined fiber bulk density ranges from 15.55 kg/m^3 for mockernut hickory to 28.37 kg/m^3 for sweetgum with bark. The exclusion of bark decreased the fiber bulk density of sweetgum to 22.77 kg/m^3. The smallest refiner plate setting used, 0.064 cm, generates fiber with the highest bulk density for each of the five hardwood species.
6. The smaller the refiner plate setting, the finer the fiber generated by pressure refining. White oak has the finest fiber, and mockernut hickory has the coars-

est fiber. Sweetgum bark is pressure-refined to a finer material compared to sweetgum wood.

7. Lengths of pressure-refined fibers and fiber bundles retained on 30- (passing 18 mesh), 45-, and 80-mesh screens are independent of refiner plate settings and species. Fiber length decreases from 2.2 mm to 1.0 mm, and fiber bundle length decreases from 2.8 mm to 1.0 mm as the screen mesh size increases from 30 mesh to 80 mesh.
8. A microscopic evaluation of the refined fiber reveals that, generally, no observable differences in surface characteristics exist between the five hardwood fiber types. Differences seem to occur in the relative amounts of cell fragments.
9. Generally, the rule of mixtures, which relates the behavior of each component to the behavior of the mixture, provided good estimates of the inherent properties of the pressure-refined hardwood fiber tested. Fiber length was excluded from this evaluation.

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