THE EFFECT OF SPECIES AND SUBSTRATE DENSITY ON THE FLEXURAL PROPERTIES OF EPOXY-IMPREGNATED BARKBOARD[1]

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ABSTRACT

Bark from red oak, yellow-poplar, soft maple, and beech was individually mixed with 6% (by weight) powdered phenolic resin and pressed into boards having nominal densities of 40, 50, 60, and 70 lb/ ft^3 (0.64, 0.80, 0.96, and 1.12 g/cm^3). Half of these boards were used as controls and half were impregnated with a solution of 100 parts by weight of epoxy resin, 25 parts styrene oxide, and 12.06 parts N,N-diethyl-1,3-propanediamine. Polymer retention was found to be linearly related to the substrate density and was somewhat dependent upon the species of bark. The impregnation process resulted in dramatic changes in the flexural properties of the barkboard. Increases in MOR of 3.0- to 4.0-fold and in MOE of 2.6- and 3.6-fold were observed over control values, with the largest improvements occurring in the red oak specimens. In contrast to the control specimens, MOR and MOE values for the epoxy-impregnated specimens were not well correlated. In addition, correlations of MOR with the substrate density and polymer mass loading were generally low or varied greatly with bark species. MOE correlations were notably higher, but variable.

Keywords: Barkboard, impregnation, epoxy, wood-polymer composites.

INTRODUCTION

The impregnation of porous systems with polymeric materials is an area of current industrial and academic interest. Most investigations of these materials

have dealt with either concrete, for applications such as the protection of bridge decks from corrosion (Whiting et al. 1974), or solid wood products, for applications such as hardwood flooring (Meyer 1977). To date, very little has been reported on other porous systems such as reconstituted wood products.

Beall et al. (1975) reported on the improvement in several properties of 42 lb/ft^3 (0.67 g/cm^3) aspen flakeboard following impregnation with methyl methacrylate (MMA) and subsequent *in situ* polymerization using Co-60 radiation. Mass loadings of approximately 60% were obtained, along with substantial improvements in a number of mechanical and physical properties. In particular, modulus of rupture (MOR) was increased by a factor of 1.62, and modulus of elasticity (MOE) by 1.75. The values for the latter compared favorably with the average MOE for hardwoods as a group, while the strength properties were still somewhat lower than values for solid wood. It was speculated that these improvements were due to the polymer distributing the applied stresses between the flakes.

More recently, Blankenhorn et al. (1977) reported on the compressive and flexural properties of impregnated red oak barkboard ranging in density from approximately 53 to 74 lb/ft^3 (0.84 to 1.19 g/cm^3) prior to impregnation. Two monomer systems were studied—one an acrylic solution of 100 parts by weight methyl methacrylate (MMA), 10 parts trimethylol propane trimethacrylate (TMPTMA), and 0.5 parts 2,2′-azobisisobutyronitrile (AZO); and the other an epoxy solution of 100 parts by weight diglycidyl ether of bisphenol A (Epon 828)[2], 25 parts styrene oxide, and 12.06 parts N,N-diethyl-1,3-propanediamine (DEA-PA). Mass loading of polymer was strongly correlated with board density, and ranged from approximately 17 to 34% for the acrylic impregnant, and from approximately 13 to 49% for the epoxy impregnant. Both resulted in substantial improvements in the measured flexural and compressive properties, but those associated with the epoxy impregnation were greater than those for the MMA system.

The present study was initiated to extend the work on epoxy impregnation of barkboard. The objectives were to determine flexural properties and to establish the relationships among the flexural properties, polymer mass loading, and density using bark from four species as substrates.

EXPERIMENTAL

Specimen preparation

A total of 160 specimens were prepared from bark of four species: red oak (*Quercus rubra* L.), soft maple (*Acer rubrum* L.), yellow-poplar (*Liriodendron tulipifera* L.), and beech (*Fagus grandifolia* Ehrh.). Half of the specimens served as controls, while the other half were ultimately impregnated with the epoxy system.

Bark from each of the species was hammermilled and classified, using a mechanically operated sieve shaker. Material passing a 0.25-inch (0.64-cm) mesh screen and retained on a 0.0625-inch (0.16-cm) mesh screen was collected for use in the specimens. Approximate distribution of the sizes obtained was reported by Bartolomucci (1979). This bark was dried to approximately 5% moisture content

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[2] Epon 828 resin, a product of Shell Chemical Company.

(oven-dry basis) and mixed with 6% by weight powdered phenolic resin (Durez #28693). Appropriate amounts of the resulting mixtures were then weighed in anticipation of yielding specimens having nominal densities of 40, 50, 60, and 70 lb/ft³ (0.64, 0.80, 0.96, and 1.12 g/cm³). All were pressed to stops, using a press with a platen temperature of 177 C (350 F) and a cycle time of 15 min.

Specimen impregnation

The rough specimens were first conditioned at 30% relative humidity and 22 C for 6 weeks. They were then machined to approximately 0.75 inch × 0.75 inch × 12.0 inch (1.9 cm × 1.9 cm × 30.5 cm) and returned to the conditioning chamber for an additional 3 weeks. After moisture equalization, their densities were determined, using the water immersion method (ASTM D2395-69).

The 80 controls were dried in a forced-air oven at 105 C for 24 h and weighed to determine their oven-dry weights. The remaining 80 specimens were dried under vacuum for 24 h at 105 C and then weighed. Prior to impregnation, the specimens were stored under vacuum at 23 C to preclude any possible moisture uptake.

The impregnant used in all cases was a solution of 100 parts by weight diglycidyl ether of bisphenol A[2], 25 parts styrene oxide reactive diluent, and 12.06 parts N,N-diethyl-1,3-propanediamine (DEAPA)[3]. This system was identical to the epoxy system used by Blankenhorn et al. (1977).

A vacuum-pressure-soak method was used to impregnate the specimens. They were placed in an impregnation chamber, and a vacuum of about 400 microns was held for 30 min. The epoxy solution was then backfilled into the chamber, and the pressure was increased to atmospheric. An overpressure of 200 psig (1,379 kPa) was subsequently applied for 4 h using nitrogen. At the end of this pressurization period, the specimens were removed, wiped free of excess resin, and permitted to cure for 24 h at 23 C (B-stage cure). Specimens were then placed in a forced-air oven at 75 C for an additional 24 h to complete the crosslinking process. The cured specimens were weighed, and the polymer mass loading (P_{ML}) for each was calculated from the following:

$$\text{mass loading} = \frac{\text{impregnated weight} - \text{substrate weight}}{\text{substrate weight}} \tag{1}$$

Additional details concerning this procedure may be obtained from Bartolomucci (1979).

Flexural tests

All specimens were tested in the oven-dry condition. A three-point loading was used for the specimens with a span of 10.5 inches (26.7 cm) and a crosshead speed of 0.1 inches/min (0.254 cm/min). Load versus deflection was continuously plotted. MOR and MOE values were calculated in accordance with ASTM D 1037-72a for wood-based fiber and particle panel materials.

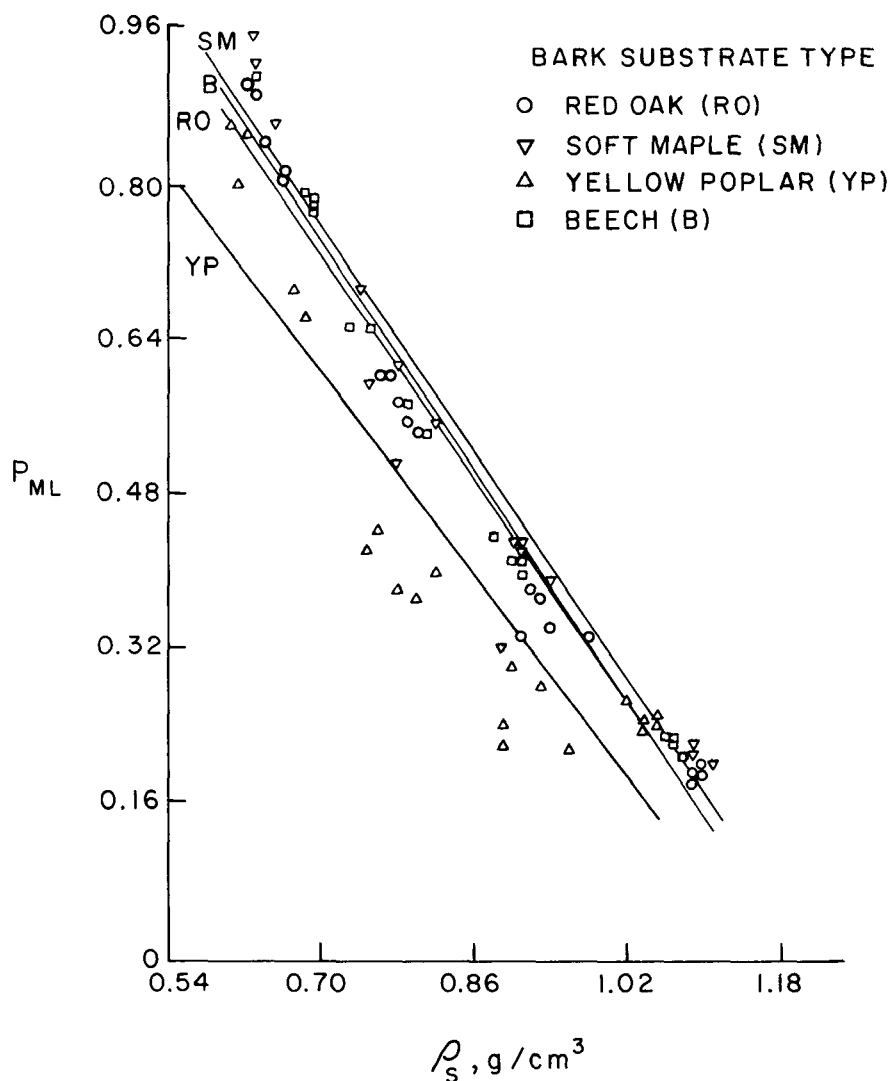[3] Curing Agent A, a product of Shell Chemical Company.

FIG. 1. Polymer mass loading as a function of substrate density for four species of bark.

RESULTS AND DISCUSSION

Polymer mass loading

The mass loading results for each of the four species and nominal substrate densities are presented in Fig. 1, and their least squares regression equations are given in Table 1. These equations are significant at the $\alpha = 0.01$ level. As one might suspect, the higher density substrates result in proportionately less polymer retention because of their lower interparticle void volumes. Results for red oak, soft maple, and beech are very similar. The reason for the lower values exhibited by the yellow-poplar specimens compared to the other specimens is not clear at this time, although a reasonable explanation is possible if one assumes that the

BARK – PLASTIC
COMPOSITE SUBSTRATES

A_I – RED OAK

B_I – YELLOW POPLAR

C_I – SOFT MAPLE

D_I – BEECH

NON FILLED CONTROL
SUBSTRATES

A_0 – RED OAK

B_0 – YELLOW POPLAR
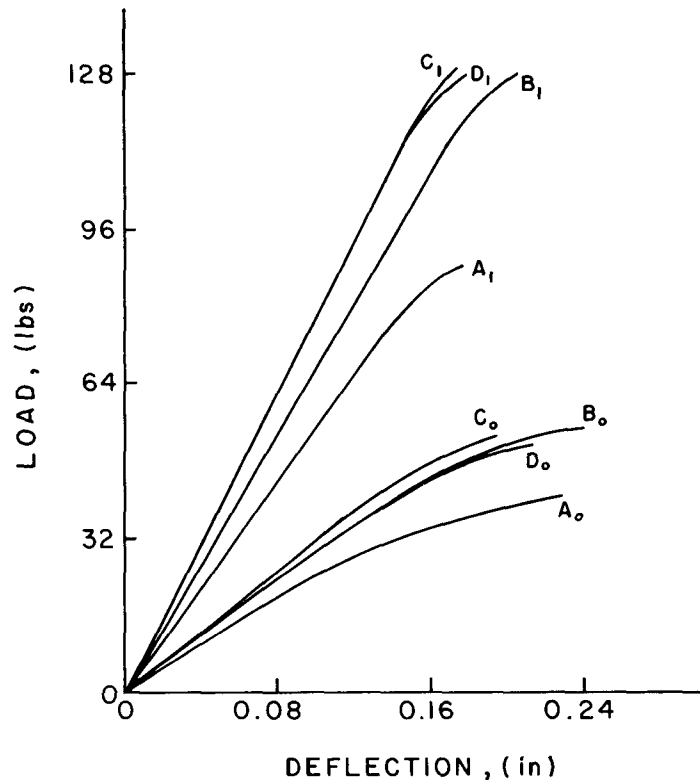
C_0 – SOFT MAPLE

D_0 – BEECH

FIG. 2. Typical load-deflection curves for unimpregnated barkboard and impregnated barkboard.

impregnant primarily occupies the interparticle, as opposed to intraparticle, void space. The yellow-poplar rhytidome can have a rather low density compared to the other species used in this study (Martin and Crist 1968). This would imply that for a given barkboard density, less interparticle void space exists for the yellow-poplar than for the others, resulting in a lower polymer mass uptake for the yellow-poplar barkboard.

Flexural data (control specimens)

The flexural strengths and moduli of the control specimens are summarized in Table 2. As in often typical of reconstituted wood products (Kollmann et al.

TABLE 1.   *Regression equations for least-squares fit of polymer mass loading as dependent upon non-filled bark substrate oven-dry density.*

Substrate type	Regression equations* for polymer mass loading, P_{ml}	$R^{2}**$
Red oak	$P_{ml} = 1.73 - 1.44\rho_s$	0.970
Soft maple	$P_{ml} = 1.80 - 1.49\rho_s$	0.934
Yellow-poplar	$P_{ml} = 1.52 - 1.31\rho_s$	0.815
Beech	$P_{ml} = 1.73 - 1.43\rho_s$	0.975

* All equations are significant at the $\alpha = 0.01$ level.
** Adjusted for degrees of freedom.

1975), MOR and MOE values increase with an increase in the oven-dry density. The least squares regression equations describing this relationship are given in Table 3. Greater than 75% of the variability in the flexural strength data for each species can be explained by these equations. The regression equations for the modulus of elasticity also show strong dependence on oven-dry density. In this case, they account for greater than 83% of the variability in the data for each species.

Analysis of variance for both the MOR and MOE showed that there were significant differences attributable to the species themselves. The yellow-poplar controls exhibited the highest overall flexural strengths and moduli, while the red oak controls had the lowest average values.

Flexural data (impregnated specimens)

The impregnation process greatly altered the flexural characteristics of the barkboard. Typical load-deflection curves for the controls and the impregnated specimens in the high density range of 0.96 to 1.11 g/cm³ are illustrated in Fig. 2.

Bending strengths (Table 4) among the four impregnated populations showed increases ranging from 3.0 to 4.0 times the average strength of the controls, and

TABLE 2.   *Flexural properties of the control specimens.*

Bark species	Nominal density* g/cm³ (lb/ft³)	Actual density range ρ_s (g/cm³)	Number of specimens	MOR (kPa × 10³) average	MOE (kPa × 10⁶) average
Red oak	0.641 (40)	0.670–0.739	5	1.32	0.24
	0.801 (50)	0.777–0.817	5	6.06	1.05
	0.961 (60)	0.897–0.961	5	7.47	1.38
	1.121 (70)	1.06–1.09	5	15.60	2.80
Yellow-poplar	0.641 (40)	0.629–0.678	5	7.06	1.40
	0.801 (50)	0.714–0.778	5	11.70	1.94
	0.961 (60)	0.867–0.929	5	16.50	2.59
	1.121 (70)	0.963–1.07	5	20.40	3.17
Soft maple	0.641 (40)	0.633–0.692	5	4.56	0.84
	0.801 (50)	0.702–0.789	5	9.86	1.81
	0.961 (60)	0.851–0.881	5	14.10	2.36
	1.121 (70)	1.01–1.08	5	19.20	3.29
Beech	0.641 (40)	0.584–0.684	3	2.83	0.52
	0.801 (50)	0.773–0.793	5	7.91	1.36
	0.961 (60)	0.873–0.919	5	10.70	1.74
	1.121 (70)	1.04–1.08	5	17.70	2.88

* 1 lb/ft³ = 0.016018 g/cm³.

TABLE 3. *Regression equations for control specimens.*

Bark species	Regression equations*	R^2**
Red oak	MOR = $-(2.23 \times 10^4) + (3.43 \times 10^4)\rho_s$	0.843
	MOE = $-(4.05 \times 10^6) + (6.21 \times 10^6)\rho_s$	0.883
Yellow-poplar	MOR = $-(1.52 \times 10^4) + (3.52 \times 10^4)\rho_s$	0.878
	MOE = $-(1.56 \times 10^6) + (4.64 \times 10^6)\rho_s$	0.927
Soft maple	MOR = $-(1.70 \times 10^4) + (3.48 \times 10^4)\rho_s$	0.756
	MOE = $-(2.70 \times 10^6) + (5.75 \times 10^6)\rho_s$	0.879
Beech	MOR = $-(2.09 \times 10^4) + (3.61 \times 10^4)\rho_s$	0.757
	MOE = $-(3.19 \times 10^6) + (5.65 \times 10^6)\rho_s$	0.862

* All equations are significant at the $\alpha = 0.01$ level. ρ_s equals nonimpregnated oven-dry substrate density, and MOR and MOE values are in kPa units.
** Adjusted for degrees of freedom.

the degree of increase was dependent upon the type of bark. The red oak specimens showed the largest percent increase, although the average strength value for this group was the lowest for the four impregnated populations. The average moduli of the epoxy-impregnated systems were greater than those of the controls by factors of 2.6 to 3.6. The degree of increase was again dependent upon the type of bark, with the red oak specimens having the largest increase.

In contrast to the strong correlation found between flexural strength and density for the control specimens, relatively weak least squares correlations (Tables 5 and 6) were found between impregnated specimen flexural strength values and initial substrate density or impregnated density values. The density values after impregnation were similar for all species and this fact may have contributed to the similarity in MOR values for all species after impregnation (Table 5). Poor correlation also existed between flexural strength and polymer mass loading. Although perhaps contrary to intuition, this was consistent with data reported by Blankenhorn et al. (1977) on red oak barkboard impregnated with the same epoxy system used in this study. Modulus of elasticity, on the other hand, correlated much better with the nonimpregnated oven-dry substrate density (ρ_s), impregnated specimen density (ρ_c), and polymer mass loading (P_{ml}), although the degree of correlation varied among the species—for example R^2 for MOE versus substrate density ranged from a high of 0.905 for the impregnated soft maple specimens to a low of 0.507 for the yellow-poplar specimens. This was a much wider range than that of the control series. In addition, there was no combination of any two of these variables $(\rho_s, \rho_c,$ and $P_{ml})$ that simultaneously yielded the highest degree of correlation common to each of the four impregnated species. Maximum R^2 improve-

TABLE 4. *Ratio of bark-polymer composite population mean property values in bending to corresponding mean property values of bark controls.*

Bark substrate type	Ratio of bark-polymer composite bending strength to oven-dry control strength in flexure	Ratio of bark-polymer composite modulus to oven-dry control modulus
Red oak	3.98	3.60
Yellow-poplar	2.95	2.62
Soft maple	3.54	3.22
Beech	3.52	3.51

TABLE 5. *Flexural characteristics of the epoxy-impregnated specimens.**

Bark species	Nominal density** g/cm³ (lb/ft³)	Actual substrate density range ρ_s (g/cm³)	Polymer mass loading P_{ml}	Impregnated specimens density range ρ_c (g/cm³)	MOR (kPa × 10⁴) average	MOE (kPa × 10⁶) average
Red oak	0.641 (40)	0.619–0.664	0.798–0.892	1.17–1.19	2.71	3.85
	0.801 (50)	0.763–0.803	0.540–0.604	1.22–1.23	3.12	4.70
	0.961 (60)	0.912–0.979	0.333–0.375	1.26–1.31	3.06	5.11
	1.121 (70)	1.09–1.11	0.181–0.199	1.29–1.32	3.24	6.00
Yellow-poplar	0.641 (40)	0.601–0.679	0.662–0.859	1.10–1.15	3.88	5.50
	0.801 (50)	0.753–0.821	0.372–0.437	1.07–1.15	3.37	5.31
	0.961 (60)	0.886–0.963	0.215–0.298	1.08–1.19	4.06	5.57
	1.121 (70)	1.01–1.05	0.237–0.265	1.28–1.31	4.75	7.46
Soft maple	0.641 (40)	0.633–0.664	0.855–0.945	1.22–1.23	4.08	5.76
	0.801 (50)	0.746–0.820	0.511–0.692	1.17–1.27	4.07	6.32
	0.961 (60)	0.886–0.936	0.320–0.434	1.17–1.38	4.16	6.70
	1.121 (70)	1.10–1.11	0.201–0.219	1.32–1.33	4.54	8.00
Beech	0.641 (40)	0.632–0.691	0.722–0.914	1.21–1.23	3.41	5.34
	0.801 (50)	0.727–0.809	0.541–0.652	1.20–1.25	3.45	5.56
	0.961 (60)	0.884–0.928	0.375–0.435	1.27–1.28	3.64	6.07
	1.121 (70)	1.06–1.08	0.209–0.231	1.30–1.31	4.34	7.56

* Data are for an average of five specimens.
** 1 lb/ft³ = 0.016018 g/cm³.

ment techniques (SAS 1979) for determining the best (in terms of R^2) two variable equations did not significantly improve R^2 values for MOE or MOR.

The relatively high R^2 values for the MOE of impregnated red oak barkboard contrasted with those of Blankenhorn et al. (1977), in which low correlations were found for both MOR and MOE and the parameters, ρ_s, ρ_c, and P_{ml}. A possible explanation for this difference may be the physical size of test specimens. In the study by Blankenhorn et al. (1977), the size of the bark particulate used was approximately that of the width and thickness of the specimens. In the present

TABLE 6. *Regression equations for impregnated specimens.*

Bark species	Regression equations*	R^2**
Red oak	MOE = (6.42 × 10⁶) + (3.09 × 10⁶)P_{ml}	0.820
	MOE = (1.02 × 10⁶) + (4.50 × 10⁶)ρ_s	0.816
	MOE = −(1.43 × 10⁷) + (1.54 × 10⁷)ρ_c	0.751
Yellow-poplar	MOE = (2.14 × 10⁶) + (4.53 × 10⁶)ρ_s	0.507
	MOR = −(2.26 × 10⁴) + (5.39 × 10⁴)ρ_c	0.411
	MOE = −(6.73 × 10⁶) + (10.9 × 10⁶)ρ_c	0.887
Soft maple	MOE = (8.27 × 10⁶) − (3.01 × 10⁶)P_{ml}	0.779
	MOE = (2.46 × 10⁶) + (4.94 × 10⁶)ρ_s	0.905
	MOE = −(6.31 × 10⁶) + (1.03 × 10⁷)ρ_c	0.443
Beech	MOR = (4.52 × 10⁴) − (1.60 × 10⁴)P_{ml}	0.539
	MOE = (8.06 × 10⁶) − (3.85 × 10⁶)P_{ml}	0.779
	MOR = (1.66 × 10⁴) + (2.40 × 10⁴)ρ_s	0.576
	MOE = (1.23 × 10⁶) + (5.74 × 10⁶)ρ_s	0.824
	MOR = −(8.28 × 10⁴) + (9.53 × 10⁴)ρ_c	0.534
	MOE = −(21.8 × 10⁶) + (22.2 × 10⁶)ρ_c	0.725

* Equations reported are those which were significant at the $\alpha = 0.01$ level (ρ_s = nonimpregnated oven-dry substrate density, ρ_c = impregnated specimen density and P_{ml} = polymer mass loading).
** Adjusted for degree of freedom.

TABLE 7. *Correlations between MOR and MOE for controls and impregnated specimens.*

Specimen	Bark species	Regression equation*	R^{2**}
Controls	Red oak	$MOE = (3.72 \times 10^4) + (1.75 \times 10^2)MOR$	0.974
	Yellow-poplar	$MOE = (5.43 \times 10^5) + (1.25 \times 10^2)MOR$	0.938
	Soft maple	$MOE = (3.19 \times 10^5) + (1.47 \times 10^2)MOR$	0.911
	Beech	$MOE = (2.23 \times 10^5) + (1.44 \times 10^2)MOR$	0.952
Impregnated	Red oak	Nonsignificant	
	Yellow-poplar	$MOE = (1.63 \times 10^6) + (1.08 \times 10^2)MOR$	0.551
	Soft maple	$MOE = (1.03 \times 10^6) + (1.34 \times 10^2)MOR$	0.310
	Beech	$MOE = -(2.03 \times 10^5) + (1.71 \times 10^2)MOR$	0.694

* MOE and MOR in kPa and equation is significant at the $\alpha = 0.01$ level.
** Adjusted for degrees of freedom.

study, a ratio of specimen thickness to particle size equal to or greater than three was used. This minimized edge effects and precluded the possibility of an occasional discontinuity in the impregnant matrix at any given cross section of the beam. The larger specimen sizes would therefore be expected to reduce the observed variability in the test results.

Of most striking interest in the present study was the lack of correlation between MOR and MOE which occurs as a result of impregnation (Table 7). In all species of the control specimens, R^2 values greater than 0.90 existed for a linear relationship between these two parameters. For the impregnated specimens, R^2 values were extremely variable, ranging from a low of 0.157 for the red oak specimens to a high of 0.694 for the beech specimens. This underscored the mechanistic distinction between modulus and strength and emphasized the difference in character resulting from the impregnation process.

SUMMARY

The effects of bark species and the substrate density on epoxy-impregnated barkboard have been examined. In all species and densities studied, the impregnation process substantially improved MOR and MOE. Red oak specimens exhibited the largest improvements in these parameters. However, red oak had the lowest values of MOR and MOE of the four species examined.

While the impregnation process improved the flexural properties, it also changed the nature of these properties. In the unimpregnated substrates of all species, strong correlation was found between MOR and MOE, MOR and substrate density, and MOE and substrate density. In the impregnated specimens, the density values after impregnation were similar, and this fact may have contributed to the similarity in the MOR values after impregnation. In addition, poor correlation was found between MOR and polymer mass loading. Somewhat higher correlation was found between MOE and the above parameters, but this was strongly dependent upon species.

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