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IV-A ASPEN FLAKEBOARD TREATED WITH DISODIUM OCTABORATE TETRAHYDRATE

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ABSTRACT

In this project, we investigated mechanical properties and fire performance of aspen flakeboards manufactured with the fire-retardant chemical disodium octaborate tetrahydrate (DOT). Flakeboards were prepared using two levels of adhesive loading (5% and 7% methylene diphenyl diisocyanate (MDI)) and three levels of fire-retardant treatments (6%, 9%, and 12%). DOT is a well known fire-retardant treatment for cellulosic materials. In this study, DOT powder was added to the blend of adhesive and flakes during manufacture of flakeboard. To evaluate fire performance, specimens were tested in a cone calorimeter. Mechanical property tests included static bending, internal bond, and T-nut withdrawal. Three parameters of density, adhesive, and fire-retardant treatment had a statistically significant effect on the modulus of rupture, modulus of elasticity, internal bond strength, and T-nut withdrawal strength. Adhesive level was a statistically significant predictor of the 180 s and 300 s averages for the rate of heat release (p-values less than 0.05). The density was a statistically significant predictor of the 60 s heat release rate average, total heat release, and ignition times at the 0.10 significance level. Except for ignition times, fire-retardant treatment improved fire performance results. Except for the smoke extinction area,

improvements in results from the cone calorimeter tests were poorer than expected for a viable fire-retardant-treated wood product. The literature suggests that better results for fire performance can be obtained by combining DOT with other fire-retardant chemicals or treating wood flakes prior to panel manufacture.

INTRODUCTION

The objective of this study was to aid development of new products by using recycled wood residue and a fire-retardant additive to improve existing products and markets. The goals include development of mechanical property data, demonstration of product performance, and demonstration to potential customers. Use of fire retardants could provide added value for chairs in offices and public transportation where fire spread can be a major concern. When this project was initiated by Michigan Technological University (MTU, Houghton, MI), a small business in Hancock, MI, was producing high quality molded chair parts for major furniture manufacturers. The plant utilized 100% aspen in their products. This study was an opportunity to expand markets and create more jobs by finding new products from trim material left in molded chair-part production.

Initial phases of the study focused on determining the viability of producing acceptable materials from mill residues. Evaluation of panels manufactured using recycled plant residues was limited to the mechanical properties of static bending, internal bond strength, and T-nut withdrawal. Internal bond strength and T-nut withdrawal strength of panels manufactured using 20% recycled plant residues were comparable to available data for panels made in production. The modulus of elasticity (MOE) from static bending tests was slightly less than reported values for the production panels. For molded chair components manufactured using the mill residues, T-nut withdrawal strength values were slightly less than reported values for production panels.

This paper reports results of the final phase of the project in which performance of aspen flakeboards treated with disodium octaborate tetrahydrate was examined. No mill residues were used in the manufacture of experimental materials for this phase of the study.

EXPERIMENTAL MATERIALS

Flakeboards were produced with standard aspen flakes, the adhesive methylene diphenyl diisocyanate (MDI), and the fire-retardant disodium octaborate tetrahydrate (DOT). Treatment levels were 0%, 6%, 9%, and 12% of the oven-dry weight of the aspen flakes. The DOT was a fine white powder (tradename Polybor®) obtained from U.S. Borax, Inc. (Rio Tinto Borax, Valencia, CA) and added to the blend of flakes and adhesive during manufacture of the flakeboard. The chemical formula for DOT is

$\text{Na}_2\text{B}_8\text{O}_{13} \cdot 4\text{H}_2\text{O}$. Two levels (5% and 7% of dry weight of flakes) of the adhesive were used. Two target panel densities (640 and 690 kg/m^3) were also part of the experimental design.

TREATMENTS OF COMPOSITES

A mixture of ammonium chloride and borax was a treatment for cellulosic fabric identified by Gay-Lussac in 1821 (Browne 1958). Boron treatments are both a fire-retardant treatment and a wood preservative. Reviews of fire-retardant treatments for wood include Browne (1958), LeVan (1984), and Kozłowski and Władyka-Przybylak (2001). Fire performance and treatment of strandboard were discussed by White and Winandy (2006). Common fire-retardant chemicals for lignocellulosic boards include boric acid; ammonium phosphates and borates; ammonium sulphate and chloride; zinc chloride and borate; phosphoric acid; dicyanodiamide; and sodium and antimony oxide (Kozłowski and Władyka-Przybylak 2001). Kozłowski and Władyka-Przybylak (2001) concluded that fire-retardant chemicals are most commonly introduced as a powder during the forming stage of partially glued particles, and treatment levels of fire-retardant chemicals are in the range of 5% to 10% of dry wood mass. Hirata and Kawamoto (1990) suggested using different fire retardants on the interior and outer portions of the panel cross section depending on the mechanisms of fire retardancy. One such combination suggested by Hirata and Kawamoto (1990) was phosphates applied to the interior and boron compounds to the outer zone (Kozłowski and Władyka-Przybylak 2001). Laufenberg and others (1986) investigated treatments for particleboards including a DOT–boric acid mixture in which flakes were soaked in treating solution. Treatment levels of 8.5% to 44% dry wood weight were obtained. Syska (1969) found application of salts in solution to green wood particles was more effective than adding dry salts in the manufacture of boards. Several examples of boron compounds being combined with other chemicals to treat wood composites were listed by LeVan (1984). In a recent study of Winandy and others (2008), strands were treated with boric acid and organic phosphate fire retardants prior to manufacture of the strandboard.

Commercial fire-retardant-treated particleboard and fiberboard products with the Class A flame-spread rating based on ASTM E84 are available on today's market. Applications listed in the U.S. Borax's product profile sheet for their DOT product called Polybor® (available from www.borax.com) included treatment of wood composites, cellulosic insulation, and cotton batting used in mattresses. As discussed by LeVan (1984), DOT is made from borax and boric acid. The use of Polybor® as an ingredient for treating particleboards for fire retardancy can be found in the patent literature (Surdyk 1975, Coyle 1977). At least one of the commercial Class A medium density fiberboards is treated with Polybor.®

METHODS

The experimental design was two average densities (640 and 690 kg/m³), two adhesive loadings (5% and 7%), and four levels of fire-retardant treatment (0%, 6%, 9%, and 12%). Only one density (690 kg/m³) was used for untreated specimens. Tests included static bending, internal bond strength, T-nut withdrawal, and cone calorimeter. Four panels of each density–adhesive–fire-retardant combination were produced. Panels and all test specimens were approximately 13 mm thick. The total number of tests for each density–adhesive–fire-retardant combination was as follows: density and static bending, 8; T-nut withdrawal, 16; internal bond, 12; and fire, 4.

Mechanical properties tests were conducted at MTU. Static bend testing was conducted according to ASTM D1037. Specimens for the static bending tests were 76 mm wide and 365 mm long. A 38-mm round radius-bearing block and end supports were used to apply the load to the center of the 305-mm test span. The load was applied at a rate of 6 mm per minute. Each specimen was tested to failure. Internal bond specimens were cut to 51 mm by 51 mm. All specimens were sanded on both faces with a stationary belt sander. Aluminum blocks were glued to both faces of each specimen using commercial hot-melt glue; the specimens were then allowed to cool. Testing was conducted according to ASTM D1037. Maximum load was recorded for each specimen. For the T-nut withdrawal tests, specimens were cut from one end of the broken static bend specimens. Specimens were 76 mm wide and 127 mm long. Specimens did not include any fractures from the bending tests. Prior to testing, a 6-mm bolt was threaded into the T-nut. The specimen was placed into a base fastened to the test machine with an opening or slot that was 51 mm wide. The speed of testing was 2.5 mm per minute. The head of the T-nut was pulled through the thickness of the panel until it reached a maximum load.

Fire performance was evaluated at the U.S. Forest Service Forest Products Laboratory (FPL) using the cone calorimeter method for determining the heat release rate (HRR) as described in ASTM E1354. The horizontal 100-mm square specimens were exposed to a constant external heating flux of 50 kW/m². The standard retainer frame (without the wire grid) was placed over the test specimen.

Static bending, internal bond, and T-nut withdrawal tests were conducted shortly after pressing without conditioning the specimens. Moisture contents of specimens for the bending tests ranged from 3.5% to 5.7%. Cone calorimeter specimens were conditioned at 23°C, 50% RH prior to testing.

RESULTS

MECHANICAL PROPERTIES RESULTS

Regression fits of mechanical property data (Table 1) indicated that adhesive, fire-retardant treatment and density were statistically significant factors (p-values less

than 0.05) affecting modulus of rupture (MOR), internal bond strength (IB), and T-nut withdrawal maximum load (TNW). For modulus of elasticity (MOE), the p-values were less than 0.0001 for density but only less than 0.10 for adhesive and fire-retardant treatment.

Regression models were used to examine practical effects of changes in the predictors on the responses. In each case, average values were used for two of the predictors and the third predictor was changed. Average values used in these calculations were 665.5 kg/m³, 6%, and 6.75% for density, adhesive level, and fire-retardant treatment level, respectively. For MOR, a change in adhesive level from 5% to 7% caused an increase of 2.6 MPa and a change in density from 641 to 690 kg/m³ caused an increase of 4.0 MPa, whereas the addition of 12% fire-retardant treatment decreased MOR by 8.7 MPa. For MOE, a change in adhesive level from 5% to 7% caused an increase of 229 MPa; a change in density from 641 to 690 kg/m³ caused an increase of 693 MPa; and the addition of 12% fire-retardant treatment decreased MOE by 404 MPa. For IB, a change in adhesive level from 5% to 7% caused an increase of 134 kPa and a change in density from 641 to 690 kg/m³ caused a decrease of 106 kPa, whereas the addition of 12% fire-retardant treatment decreased the IB by 398 kPa. For TNW, a change in adhesive level from 5% to 7% caused an increase of 294 N and a change in density from 641 to 690 kg/m³ caused an increase of 500 N, whereas the addition of 12% fire-retardant treatment decreased TNW by 656 N.

TABLE 1

**. RESULTS FOR MECHANICAL PROPERTIES.
RESULTS ARE THE MEANS AND (STANDARD DEVIATIONS)**

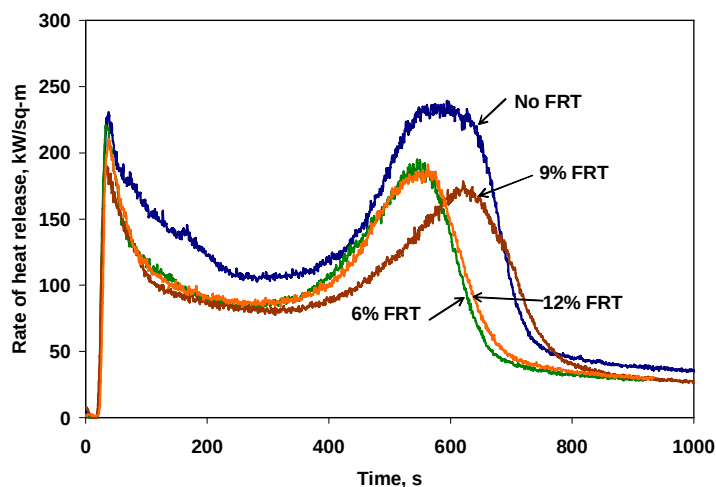
Adhesive Loading (%)	Fire - Retardant Loading (%)	Board density (kg/m³)	Static Bending		Internal Bond Strength (kPa)	T-Nut Withdrawal Maximum Load (N)
			Modulus of Rupture (MPa)	Modulus of Elasticity (MPa)		
5	0	718 (26)	45.6 (2.3)	6476 (772)	839 (228)	5262 (924)
	6	694 (16)	36.4 (7.9)	6307 (990)	813 (260)	4652 (812)
		659 (15)	37.1 (9.1)	6050 (1299)	742 (252)	4718 (1050)
	9	698 (13)	37.1 (7.6)	6538 (868)	666 (174)	4811 (619)
		656 (13)	30.0 (4.2)	5235 (535)	892 (213)	4294 (561)
	12	706 (18)	33.9 (3.8)	5974 (890)	707 (119)	4858 (703)
665 (14)		30.9 (6.0)	4985 (1473)	599 (163)	4292 (930)	
7	0	701 (41)	44.1 (10.5)	6446 (1556)	1215 (347)	5730 (1240)
	6	692 (22)	37.8 (7.1)	6116 (935)	855 (274)	5119 (677)
		646 (23)	36.7 (5.4)	5788 (592)	1157 (219)	4459 (1286)
	9	709 (19)	41.6 (8.8)	6591 (1117)	773 (162)	5414 (798)
		653 (18)	35.4 (5.2)	5823 (848)	861 (260)	4573 (803)
	12	700 (26)	36.8 (5.7)	6332 (1033)	722 (239)	4614 (682)
660 (21)		33.1 (4.1)	5569 (623)	691 (216)	4422 (768)	

FIRE PERFORMANCE RESULTS

The typical curve for wood products was observed in these tests and the HRR of treated specimens was less than that of the untreated specimen (Figure 1). For reporting purposes, results from the curves include initial peak heat release rate (PHRR), the average of the heat release rate (AHRR) for various periods (60, 180, and 300 s) (AHRR-60, etc.) after observation of sustained ignition of the test specimen. Total heat release (THR) is the cumulative heat release over the duration of the test. For the duration of the test, AHRR, mass loss rate (AMLR), and effective heat of combustion (AEHOC) of the burning specimen were determined. Obscuration of a laser beam in the exhaust duct was also recorded as a measure of the visible smoke development from the burning specimen. Average specific extinction area (ASEA) (m^2/kg) was computed from smoke obscuration data. Ignitability was determined by observing the time for sustained ignition (TSI) of the specimen.

Fire-retardant treatments resulted in reductions in the HRR (Table 2). Regression models were developed with adhesive level, density, and fire-retardant treatment level as the predictors. At a 0.05 significance level, adhesive was a statistically significant predictor of HRR-180 and HRR-300. At a 0.10 significance level, density was a statistically significant predictor of HRR-60, THR, TSI, and β . At a 0.001 significance level, fire-retardant level was a statistically significant predictor of all cone calorimeter results other than TSI. Only density was a statistically significant (at a 0.10 significance level) predictor of TSI. A quadratic fire-retardant term was also statistically significant for the cone calorimeter responses other than PHRR, ASEA, and TSI.

FIGURE 1
TYPICAL HRR CURVES.



As with mechanical properties, regression models were used to examine the practical effects of changes in the predictors. Except for TSI, changes in these responses from the change in adhesive from 5% to 7% or the change in density from 641 to 690 kg/m³ were all small compared to changes from a 12% fire-retardant treatment. Reductions in heat release rates for the 12% treatment were 23, 39, 40, 36, and 26 kW/m² for PHRR, HRR-60, HRR-180, HRR-300, and AHHR, respectively. Effective fire-retardant treatments normally significantly reduce the initial PHRR. For example, 16-mm thick maple lumber treated with approximately 58 kg/m³ of borax/boric acid had an initial PHRR rate of 85 kW/m² compared to 209 kW/m² for the untreated lumber specimen when tested in a cone calorimeter (without retainer frame or grid; a heat flux of 50 kW/m²) (White and Dietenberger 2004). The reduction in AHRR-300 for the borax/boric acid-treated maple of a previous study was from 146 kW/m² for the untreated specimens to 64 kW/m² for the treated lumber specimens (White and Dietenberger 2004).

Only density was a significant factor in the regression of the TSI data (Table 3). The regression model using averages for the other parameters showed an increase of 2.5 s for the change in density from 641 to 690 kg/m³ and an increase of only 1.1 s for the 12% fire-retardant treatment. The regression model using averages for the other parameters showed a decrease in AEHOC (Table 3) of 1.6 MJ/kg for the 12% fire-retardant treatment. In contrast, reduction in AEHOC for the borax/boric acid-treated maple of a previous study was from 12.0 MJ/kg for untreated specimens to 7.2 MJ/kg for treated lumber specimens (White and Dietenberger 2004). The regression model using averages for the other parameters showed a decrease in ASEA (Table 3) of 44.9 m²/kg for the 12% fire-retardant treatment. Reduction in ASEA for the borax/boric acid-treated maple lumber of a previous study was from 50.1 m²/kg for untreated specimens to 2.8 m²/kg for treated lumber specimens (White and Dietenberger 2004).

One screening method for fire-retardant treatments is to measure the mass loss rate and the final residual mass fraction (RMF). The method described in ASTM E 2102 is the cone calorimeter without the oxygen-consumption measurement of heat release. The regression model using averages for the other parameters showed a decrease in AMLR (Table 3) of 2.6 g/s-m² and an increase in RMF (Table 3) of 0.11 for the 12% fire-retardant treatment. In contrast, increase in residual mass fraction for the borax/boric acid-treated maple lumber of a previous study was from 0.18 for untreated specimens to 0.34 for treated lumber specimens (White and Dietenberger 2004).

The cone calorimeter has been used to provide estimates of the flame-spread index obtained in the tunnel test (ASTM E84) (White and Dietenberger 2004). Based on the model (Dietenberger and White 2001), a variable called beta (β) was also calculated (Table 3). Values for β are used to estimate ASTM E84 flame-spread indices. A β of 0.184 is used to differentiate between the Class C and Class B materials classified based on their ASTM E84 flame-spread index. All estimates for all the untreated and treated aspen flakeboard specimens were for Class C or higher (β greater than 0.184). Most untreated U.S. domestic wood products are Class C. Treatment did produce

reductions in the values for β (Table 3), which would correspond to reductions in the estimated flame-spread index.

TABLE 2

. HEAT RELEASE DATA.

RESULTS ARE MEANS AND (STANDARD DEVIATIONS). SEE TABLE 1 FOR DENSITY OF CORRESPONDING ROWS OF DATA

Adhesive Loading (%)	Fire Retardant Loading (%)	Peak Heat Release Rate (kW/m ²)	Average Heat Release Rates				Total Heat Released (MJ/m ²)
			60 s (kW/m ²)	180 s (kW/m ²)	300 s (kW/m ²)	Test Duration (kW/m ²)	
5	0	233 (6)	192 (3)	159 (3)	141 (4)	123 (5)	121 (6)
	6	216 (10)	165 (3)	125 (5)	109 (4)	99 (3)	84 (10)
		222 (20)	167 (6)	126 (2)	112 (2)	103 (2)	89 (9)
	9	206 (10)	156 (5)	119 (3)	105 (2)	97 (3)	86 (6)
		213 (14)	160 (5)	120 (5)	105 (5)	95 (2)	77 (5)
	12	201 (7)	150 (7)	115 (6)	101 (5)	96 (4)	87 (4)
		219 (5)	156 (6)	115 (6)	102 (6)	96 (3)	81 (3)
7	0	228 (4)	189 (5)	149 (4)	132 (4)	116 (6)	108 (7)
	6	224 (15)	165 (10)	125 (6)	110 (4)	103 (4)	87 (6)
		211 (12)	162 (4)	123 (4)	108 (4)	101 (3)	83 (5)
	9	210 (22)	154 (7)	115 (5)	101 (5)	95 (4)	84 (7)
		220 (19)	159 (7)	118 (3)	104 (2)	101 (2)	76 (6)
	12	213 (16)	150 (7)	110 (4)	97 (3)	91 (6)	83 (9)
		216 (6)	155 (5)	117 (5)	103 (5)	95 (5)	82 (6)

TABLE 3

. OTHER RESULTS FROM CONE CALORIMETER TESTS.

RESULTS ARE MEANS AND (STANDARD DEVIATIONS). SEE TABLE 1 FOR DENSITY OF CORRESPONDING ROWS OF DATA

Adhesive Loading (%)	Fire Retardant Loading (%)	Average Mass Loss Rate (g/s-m ²)	Average Effective Heat of Combustion (MJ/kg)	Average Smoke Extinction Area (m ² /kg)	Times for Ignition (s)	Residual Mass Fraction	Beta (β)
5	0	12.7 (0.05)	13.5 (0.4)	84.2 (19.4)	26.0 (1.8)	0.169 (0.016)	0.296 (0.014)
	6	10.8 (0.2)	11.5 (0.4)	40.3 (6.6)	25.1 (2.8)	0.242 (0.009)	0.249 (0.017)
		10.7 (0.3)	12.1 (0.3)	39.4 (5.5)	24.2 (2.8)	0.247 (0.009)	0.265 (0.031)

(continued)

TABLE 3
(CONTINUED)

Adhesive Loading (%)	Fire Retardant Loading (%)	Average Mass Loss Rate (g/s-m ²)	Average Effective Heat of Combustion (MJ/kg)	Average Smoke Extinction Area (m ² /kg)	Times for Ignition (s)	Residual Mass Fraction	Beta (β)
6	9	10.4 (0.2)	11.6 (0.1)	48.0 (8.0)	23.4 (2.5)	0.266 (0.010)	0.234 (0.017)
		10.6 (0.3)	11.7 (0.2)	34.5 (21.0)	21.4 (2.8)	0.260 (0.016)	0.248 (0.017)
	12	10.2 (0.2)	11.9 (0.3)	40.3 (24.3)	26.9 (4.0)	0.284 (0.007)	0.224 (0.015)
		10.2 (0.5)	11.6 (0.4)	44.1 (11.8)	25.2 (1.5)	0.276 (0.025)	0.251 (0.010)
7	0	12.6 (0.03)	13.2 (0.3)	78.1 (15.8)	22.8 (1.4)	0.173 (0.025)	0.290 (0.006)
	6	10.9 (0.3)	12.1 (0.4)	45.9 (23.6)	27.3 (3.7)	0.254 (0.019)	0.257 (0.032)
		11.2 (0.4)	11.7 (0.1)	59.2 (13.7)	23.9 (2.3)	0.236 (0.024)	0.244 (0.019)
	9	10.2 (0.2)	11.7 (0.4)	49.4 (12.0)	24.2 (1.8)	0.276 (0.023)	0.242 (0.036)
		10.5 (0.04)	12.1 (0.2)	51.9 (12.1)	21.3 (4.9)	0.271 (0.022)	0.259 (0.020)
	12	10.0 (0.2)	11.4 (0.5)	17.7 (9.5)	26.9 (9.0)	0.270 (0.028)	0.237 (0.018)
		10.1 (0.5)	11.9 (0.4)	34.8 (27.2)	22.5 (2.4)	0.267 (0.016)	0.254 (0.015)

DISCUSSION

Whereas treated specimens performed better statistically than untreated control specimens in fire performance, improvements were considerably less than that expected for a viable fire-retardant-treated wood product. Differences in adhesive levels used in this study did not affect fire performance results.

Ayrilmis and others (2005) studied different fire-retardant treatments for aspen oriented strandboard. One treatment was DOT at levels of 2, 4, and 6% of dry wood mass. The adhesive was a phenol-formaldehyde resin at a 3.5% per dry wood weight-loading level. Whereas the fire test results for the untreated aspen boards in the Ayrilmis study were slightly better than the untreated results of Tables 2 and 3, the modest improvements obtained by the treatments were similar to that shown in Tables 2 and 3. Ayrilmis and others found that DOT treatment did not affect the times for sustained ignition. The PHRR results decreased slightly from 162 to 195 kW/m² results for untreated to 150 to 162 kW/m² for treated specimens. Their results for the residual mass fraction were similar to the results of Table 3. The β of Dietenberger's model (2001) decreased from 0.21 for the untreated to 0.15 to 0.17 for the treated

specimens. In tests done at FPL, the β was 0.22 for untreated southern pine plywood and -0.08 for the southern pine plywood treated with a commercial fire-retardant.

In terms of mechanical properties, Ayilimis and others (2005) found reductions in MOE (5.19 to 4.31 GPa), MOR (31.17 to 22.86 MPa), and internal bond strength (0.60 to 0.33 MPa) for the 6% treatment level compared with the untreated. Our regression models indicated that reductions for 6% fire-retardant treatments were 6.0 to 5.8 GPa for MOE, 41.1 to 36.7 MPa for MOR, and 1.12 to 0.92 MPa for internal bond strength.

Fire performance results generally improved with higher treatment levels but the differences in results for various treatment levels were not statistically significant in most cases. LeVan and Tran (1990) found that improvements in fire performance on fire tube and OSU rate of heat-release tests were reduced once treatment levels of the lumber and plywood specimens with a borax-boric acid mixture were higher than 7.5% add-on by weight (or 48 kg/m³ by weight or 6.4% aqueous solution). This flattening in improvements was also observed by Eickner and Schaffer (1967) in 8-ft. tunnel tests of plywood treated with borates.

In our study, DOT powder was added to the blend of flakes and adhesive during the manufacturing process. Possible ways to improve fire performance of the product include (1) a gradient in concentrations of fire-retardant chemical so more chemical is near the product's surfaces; (2) combination of DOT with other fire-retardant chemicals, and (3) treatment of wood fibers prior to manufacture.

CONCLUSIONS

All three parameters of density, adhesive, and fire-retardant treatment had a statistically significant effect on MOR, MOE, internal bond strength, and T-nut withdrawal strength. Adhesive level was a statistically significant predictor of the 180 s and 300 s averages for the rate of heat release (p-values less than 0.05). The density was a statistically significant predictor of the 60 s heat release rate average, total heat release, and ignition times at the 0.10 significance level. Except for ignition times, changes in the fire-retardant-treatment level did improve fire performance results. Except for the smoke extinction area, improvements in results from the cone calorimeter tests were poorer than expected for a viable fire-retardant-treated wood product. The literature suggests that improved results for fire performance can be obtained by combining DOT with other fire-retardant chemicals or treating wood flakes prior to panel manufacture.

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