

THE INFLUENCE OF FLAKE CHEMICAL PROPERTIES AND ZINC BORATE ON GEL TIME OF PHENOLIC RESIN FOR ORIENTED STRANDBOARD¹

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(Received July 2000)

ABSTRACT

The pH and buffer capacity of dry wood flakes from eleven southern species and their effects on gel time of phenol formaldehyde (PF) oriented strandboard resin with and without zinc borate addition were investigated in this study. It was shown that the pH of the hardwood flakes was acidic, with white oak being the most acidic (pH = 4.60) and elm nearly neutral (pH = 6.93). Southern pine flakes had a relatively low pH value (pH = 4.98), compared to the hardwood flakes. The alkali buffer capacity was larger than the corresponding acid buffer capacity for most species tested. The total buffer capacity varied from 0.09 for cypress to 0.358 for hackberry. Measured resin gel times with and without wood presence decreased as the amount of zinc borate increased. The reduced gel time was partially recovered by using polyethylene glycol (PEG) in combination with the borate. The effectiveness of PEG varied with wood species and the level of borate used. The gel time had no direct correlation to flake pH and buffer capacity of the wood species used.

Keywords: Buffer capacity, gel time, pH, phenolic resin, oriented strandboard, termite, zinc borate.

INTRODUCTION

Formosan subterranean termites (FSTs) have rapidly expanded their geographic domain in the southern United States and Hawaii. FSTs are thought to cause over \$1 billion structural damage per year in the country (Ring 1999). It is the most destructive insect in Louisiana. An epidemic destruction by FSTs causes about \$300 million per year in

historic and residential buildings in the greater New Orleans metropolitan area alone (Shupe and Dunn 2000). The ultimate solution to termite destruction is to use wood species resistant to termites or to use termite-repellent chemicals for wood-based products in residential construction. It has been shown that both chromated copper arsenate (CCA) and borate treatments prevent termite attack on wood members. Structural lumber and plywood can be successfully treated after their manufacture (e.g., treatments with CCA). Oriented strandboard (OSB), however, cannot be pressure-

¹ This paper (No:00-22-0685) is published with the approval of the Director of the Louisiana Agricultural Experiment Station, LSU Agricultural Center.

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treated with water borne preservatives once it is manufactured because of its large swelling characteristics.

Work has been done to incorporate borate chemicals such as zinc borate (ZB) into OSB furnish during the blending process (Laks et al. 1988, 1991; Sean et al. 1999). Panels with good termite-resistant properties have been successfully developed using zinc borate at various application rates (e.g., 1% based on dry wood weight). Borate, however, has an adverse effect on mechanical and physical properties of OSB, especially those bonded with phenol-formaldehyde (PF) resin (Laks et al. 1991). This has presumably been attributed to the interference of borate with the resin cure process during hot-pressing (Pfaff 1993; Sean et al. 1999). As a result, a higher resin-loading rate is usually needed to achieve acceptable board strength and durability at high levels of borate addition. Potential durability issues of borate-modified OSB have generated controversy in the structural application of this product. This signals the need for studying bonding characteristics and durability of OSB as influenced by flake chemical properties (e.g., pH and buffer capacity) and levels of borate application.

The importance of wood chemical properties and surface activation by oxidizing agents on wood-adhesive bonding has been well studied (Gardner and Elder 1988; Johns and Niazi 1980; Maloney 1977; Stamm 1961). The pH of wood may change the pH of the adhesive at the interface and modify the cure of resin during hot-pressing. As a result, a too high or too low pH of wood has been reported to be troublesome for achieving good adhesive bonds in wood-based products (Bryant 1968; Campbell and Bryant 1941; Chen 1970). The pH of wood is generally related to its extractive content, which varies from species to species. Although extractives represent only a small portion of wood, they include a wide range of chemical compounds from volatile terpenes, organic solvent-soluble fatty acids and waxes, water-soluble carbohydrates, and proteins (Cotton and Wilkinson 1988; Fengel

and Wegner 1984). These substances hinder adhesive wetting and thus interfere with the cure process of the adhesives by reducing or prolonging the cure time (Johns and Niazi 1980; Jordan and Wellons 1977). Removal of these extractives represents an effective means to reduce their effects on wood-adhesive bonding. It was reported that the pH of wood from temperate zones is in the weak-to-moderate acidic range of 3.3–6.4 (Gray 1958; Ingruber 1958), while the pH of tropical woods ranges from weak acidic to weak alkaline of 3.7–8.2 (Chen 1970). Johns and Niazi (1980) reported that both hardwoods and softwoods are in the pH range of 4.0–5.9, with an inconsistent difference between sapwood and heartwood. The process of hot-water soaking used to prepare wood for flaking and drying the flakes to a suitable moisture content for OSB production is likely to change the pH and buffer capacities of wood because of the removal of water-soluble extractives.

Resin gel time is considered a relative measure of the rate of resin cure. It is usually characterized by a sudden increase in the viscosity from liquid resin to a solid gel, as measured by a suitable gel-time apparatus. It was found that the gel time of urea-formaldehyde (UF) resin was directly correlated to wood pH and inversely correlated with acid-buffer capacity for both hardwood and softwood aqueous extracts (Johns and Niazi 1980). The adverse effect of borate in OSB has been attributed to the interference of borate with the resin flowing and curing process during hot-pressing (Laks et al. 1991; Sean et al. 1999). It is believed that boron ions react with the functional methylol groups on resin molecules, which causes the resin to precure prior to consolidation. As a result, severe pressing conditions have to be used for the resin to be able to cure in a normal manner (Hsu and Pfaff 1992). Sean et al. (1999), in evaluating the effect of borate treatment on the physical and mechanical behavior of OSB, used polyethylene glycol (PEG) as a flow agent to improve resin flow and curing during hot-pressing. They reported that the adverse effect of borate on ad-

hesive flow could be minimized by the addition of organic flow agents containing hydroxyl (-OH) groups such as PEG. Additional information regarding the gel process of PF OSB resin may shed more light on the resin-curing process in the presence of zinc borate and wood with different chemical properties.

Currently, very few data on chemical properties of OSB flakes are available. Also, the effects of flake pH and buffer capacity on resin gel time in the presence of chemical additives such as zinc borate are still unknown. The study reported here represents the first part of a comprehensive study on developing chemically modified OSB with desired strength, durability, and termite resistance using southern wood species. The objective of this work was to study effects of wood flake chemical properties and addition of zinc borate on gel time of PF resin used in the manufacture of OSB.

MATERIALS AND METHODS

Material selection and sample preparation

Lumber selection.—Green boards (2.44 m long by 2.54 cm thick by random width) from each of the following eleven species were obtained from Roy O. Martin Lumber Company in Louisiana. These species included ash (*Fraxinus* spp.), cottonwood (*Populus* spp.), cypress (*Taxodium distichum* (L.) Rich.), elm (*Ulmus americana* L.), hackberry (*Celtis occidentalis* L.), locust (*R. pseudoacacia* L.), pecan (*Carya* spp.), red oak (*Quercus* spp.), white oak (*Quercus alba* L.), willow (*Salix* spp.), and southern pine (*Pinus taeda* L.). Among the species, plantation loblolly pine is a primary species for the manufacture of southern pine OSB. The nine hardwood species and cypress are among the most common species used to manufacture mixed hardwood OSB. The specific gravity among all wood species ranges from 0.39 to 0.69 (USDA Forest Service 1986).

Wood flake preparation.—The boards were cross-cut along the length of the board into 152.4-mm-long blocks. These blocks were

soaked in water prior to making flakes. The soaked blocks were flaked using a laboratory disc flaker to produce 76.2-mm-long flakes (0.635 mm thick). The wide surfaces of the flakes were parallel to either longitudinal-tangential or longitudinal-radial plane, depending on whether the boards were flat or quarter-sawn. The flakes were dried to 2–3% moisture content using a steam-heated cabinet dryer at a temperature of 95°C. The dry flakes were screened to eliminate fines and stored in polyethylene bags until needed. Approximately one kilogram of dry flakes from each species was randomly collected for the measurements of pH, buffer capacity, and gel time described in this study. The rest of the flakes were used for preparing OSB.

Wood meal preparation.—A sufficient amount of dry flakes from each of the eleven species was selected. There was no separation between heartwood and sapwood flakes for any of the species. They were Wiley-milled to pass through a coarse screen (20 mesh per 25.4 mm). The produced wood meal was stored in polyethylene bags until needed. The moisture content of the wood meal at the time of testing was 6% for all species.

Resin and chemical additives.—Phenol formaldehyde OSB face resin with a 55% non volatile content was obtained from Neste Resins Corporation in Winnfield, Louisiana. The pH of the resin was 9.8, and specific gravity was between 1.1 and 1.3. The resin was kept in a freezer prior to the measurements. Several hours before actual tests, a sufficient amount of resin was removed from the freezer. The resin was allowed to thaw and was then placed in a water bath at 20°C to maintain a constant resin temperature. Zinc borate ($2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 3.5\text{H}_2\text{O}$) was obtained from U.S. Borax Company in Valencia, California. The specific density of the borate was 2.79. PEG, purchased commercially, has a general formula of $\text{H}(\text{OCH}_2\text{CH}_2)_n\text{OH}$. The average degree of polymerization of the PEG is between 68 and 84.

Measurements of wood pH and buffer capacity

Flake pH and buffer capacity were determined using the method developed by Johns and Niazi (1980) to provide a comparable result between the two studies. In this method, a sample of 26.5 g wood meal was obtained for each measurement, which gave 25 g oven-dry wood weight based on the measured moisture content of wood meal (i.e., 6%). The wood meal was refluxed in 250 g of distilled water for 20 min to obtain liquid wood extract. The filtrate was then filtered through Whatman #1 filter paper with an aspirator vacuum. An Orion model 410A Benchtop pH meter was used to determine the pH value of the wood extract solution at room temperature (25°C). The meter was calibrated prior to each measurement. In determining the buffer capacities, 50 ml of the extract solution was titrated to a pH of 7 and 3 with 0.025 N NaOH and H₂SO₄ solutions, respectively. The pH of the solutions was recorded after adding each ml of acid and alkali solution. Acid, alkali, and total buffer capacities were calculated according to the following formula:

Acid Buffer Capacity

= Volume of 0.025 N NaOH solution required to change (increase) the pH of wood extract to 7 \times Normality of NaOH

Alkali Buffer Capacity

= Volume of 0.025 N H₂SO₄ solution required to change (decrease) the pH of wood extract to 3 \times Normality of H₂SO₄

Total Buffer Capacity

= Acid Buffer Capacity + Alkali Buffer Capacity (1)

The mean pH (based on four measurements) and buffer capacity (based on two measurements) values were reported for each wood species.

TABLE 1. Experimental design for gel time measurements.

Test group number	Experimental variables			
	Resin ^a	Zinc borate ^b	PEG ^c	Wood ^d
1	✓			
2	✓	✓		
3	✓	✓	✓	
4	✓			✓
5	✓	✓		✓
6	✓	✓	✓	✓

^a Resin: Ten grams of liquid PF resin (55% solid) conditioned at 20°C for all tests.

^b Zinc borate: Test groups 2 and 3 had 0.5, 1, 9, and 18% of solid resin weight. Test groups 5 and 6 had 5, 10, and 20% of oven-dry solid wood weight.

^c PEG: One PEG application rate at 40% of the corresponding zinc borate weight.

^d Wood: 0.5 grams of dry wood meal (test groups 4 and 5 had eleven single species and one mixture of all species, and test group 6 had only southern pine, red oak, and the mixture of all species).

Measurements of resin gel time

A Sunshine gel timer with water bath (100°C) was used to measure resin gel time. The measurements were conducted according to the schemes listed in Table 1. As shown, all tests were made with 10 g of the PF resin conditioned to a temperature of 20°C. Test groups 1, 2, and 3 dealt with the effect of borate and PEG on gel time of neat resin (without wood meal). The borate application rates were 0, 0.5, 1.0, 9.0, and 18% based on solid resin weight of 5.5 g. Test groups 3, 4, and 5 dealt with the effect of borate and PEG on gel time of PF resin in the presence of wood meal. A constant weight of 5.3 g of wood meal at 6% MC was used for all species. The zinc borate application rates were 0, 5, 10, and 20% based on dry wood weight of 5 g. Using an average surface area ratio between wood flake and wood meal (-9+20 Tyler mesh particles) of 3.5 (Gardener and Elder 1988), the corresponding target borate covering rate for wood flakes were 0, 1.5, 3.0, and 6%. The PEG application rate was 40% of the corresponding borate weight for all test runs. Two gel time measurements were conducted at each condition. The difference between the two measurements was on average less than 1.5%.

Each material was weighed to the required amount and mixed in a 13- \times 100-mm test

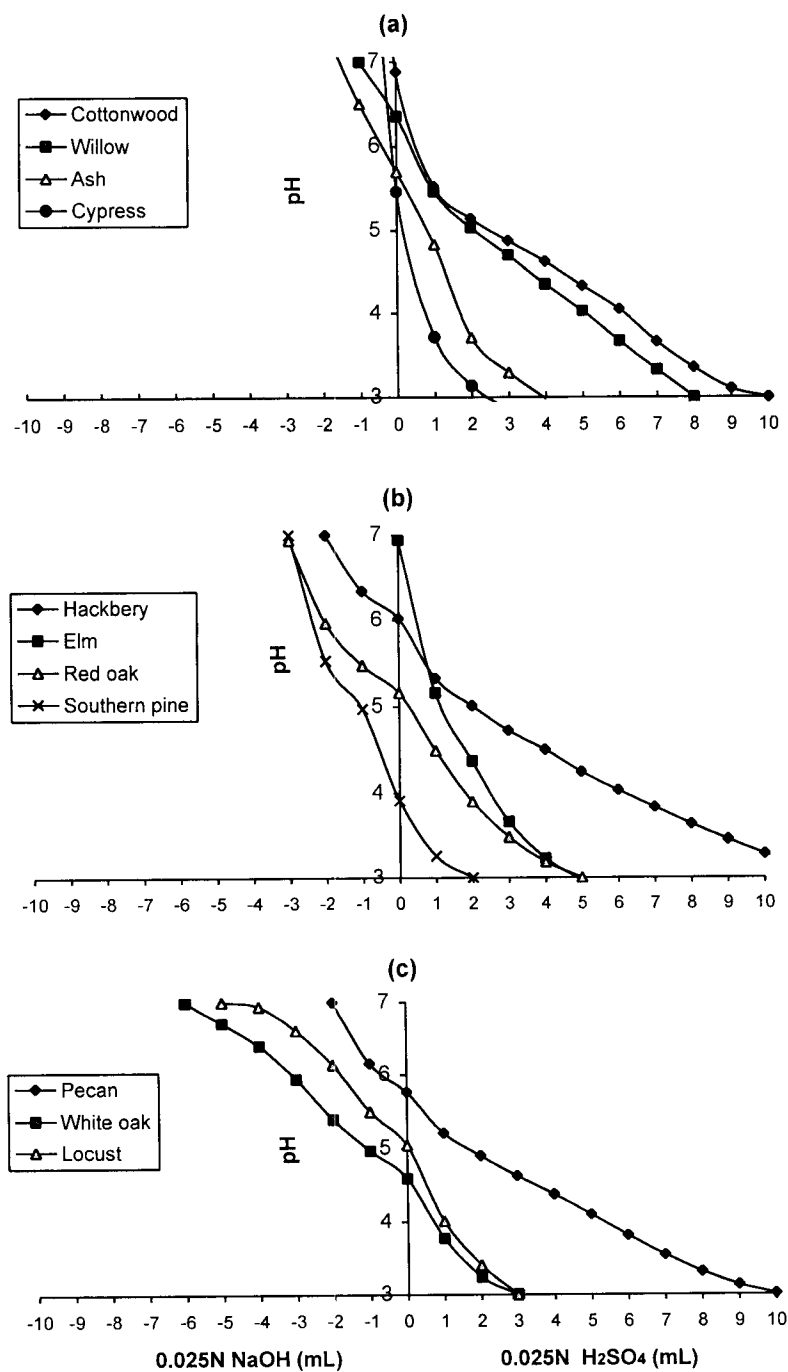


FIG. 1. Measured pH and buffer capacity of the eleven southern wood species. (a) low-, (b) medium-, and (c) high-density groups.

TABLE 2. Summary of test data on wood flake pH, buffer capacities, and gel time of phenolic OSB face resin.

Species	SG	pH ^a	Buffering capacity ^b			Gel time (minutes) at various zinc borate covering levels (based on dry wood weight) ^c			
			Acid	Alkali	Total	0%	5%	10%	20%
Ash (<i>Fraxinus</i> spp.)	0.49	5.68 (0.12)	0.038	0.125	0.163	22.40	23.18	22.51	20.40
Cottonwood (<i>Populus</i> spp.)	0.40	6.88 (0.14)	0.003	0.250	0.250	22.88	22.91	22.47	21.23
Cypress (<i>Taxodium distichum</i>)	0.46	5.46 (0.06)	0.015	0.075	0.090	22.43	23.30	22.60	20.65
Elm (<i>Ulmus americana</i> L.)	0.50	6.93 (0.05)	0.005	0.125	0.125	22.57	23.08	22.40	20.40
Hackberry (<i>Celtis occidentalis</i> L.)	0.53	6.00 (0.08)	0.058	0.300	0.358	23.07	23.42	22.52	20.55
Locust (<i>R. pseudoacacia</i> L.)	0.69	5.04 (0.03)	0.125	0.075	0.133	22.35	23.12	22.60	21.66
Pecan (<i>Carya</i> spp.)	0.61	5.73 (0.07)	0.041	0.250	0.291	24.08	22.08	22.21	20.58
Red oak (<i>Quercus</i> spp.) ^d	0.59	5.14 (0.03)	0.075	0.125	0.200	22.19	22.40	22.98	20.50
White oak (<i>Quercus alba</i> L.)	0.68	4.60 (0.01)	0.150	0.075	0.225	23.15	23.08	22.23	20.86
Willow (<i>Salix</i> spp.)	0.39	6.35 (0.06)	0.014	0.203	0.216	23.30	24.61	23.90	21.67
Southern pine (<i>Pinus taeda</i> L.) _d	0.58	4.98 (0.03)	0.043	0.071	0.118	23.30	24.22	23.07	20.55
Mixture (Eleven species) ^d			Not Available			22.96	22.83	22.15	20.46
						22.83	22.10	22.15	21.12
						23.06	22.10	21.33	19.65
							22.83	22.77	21.17

^a Average value of four measurements from two extract replications. Values in parentheses are standard deviations.^b and ^c Average value of two measurements from two extract replications.^d Data shown under 5, 10, and 20% borate covering rates was from tests made with 40% PEG of corresponding zinc borate weight.

tube with a mechanical mixer. After a glass test rod was inserted in the tube, the tube was quickly placed in the water bath at 100°C. The glass rod was connected by magnetic force to the meter's spinning head, which was connected to a timer inside the meter. The timer was started immediately after the test tube was placed in the water bath. The tube was allowed to remain in the bath until the meter indicated completion of each test.

RESULTS AND DISCUSSION

Wood pH and buffer capacity

Measured pH and buffer capacity data are presented in Fig. 1 (a: low-density species, b: medium-density species, and c: high-density species). Initial pH readings and buffer capacities (acid, base, and total) are summarized in Table 2. The pH data from the four replicates

were quite consistent as indicated by small standard deviations (Table 2). The buffer capacity data are plotted in Fig. 2 for the eleven species.

The pH of wood extracts decreased (Fig. 1) with addition of H₂SO₄ and increased with addition of NaOH (i.e., buffering). To achieve a given pH level, the amount of H₂SO₄ and NaOH addition varied significantly from species to species. However, the general trend was the same for all species, similar to those reported by Johns and Niazi (1980). The initial pH values of the southern hardwood and cypress flakes studied were on the acidic side with white oak being the most acidic (pH = 4.60) and elm being nearly neutral (pH = 6.93). Southern pine flakes had a relatively low pH value (4.98), compared to most of the hardwoods. The alkali buffer capacity was

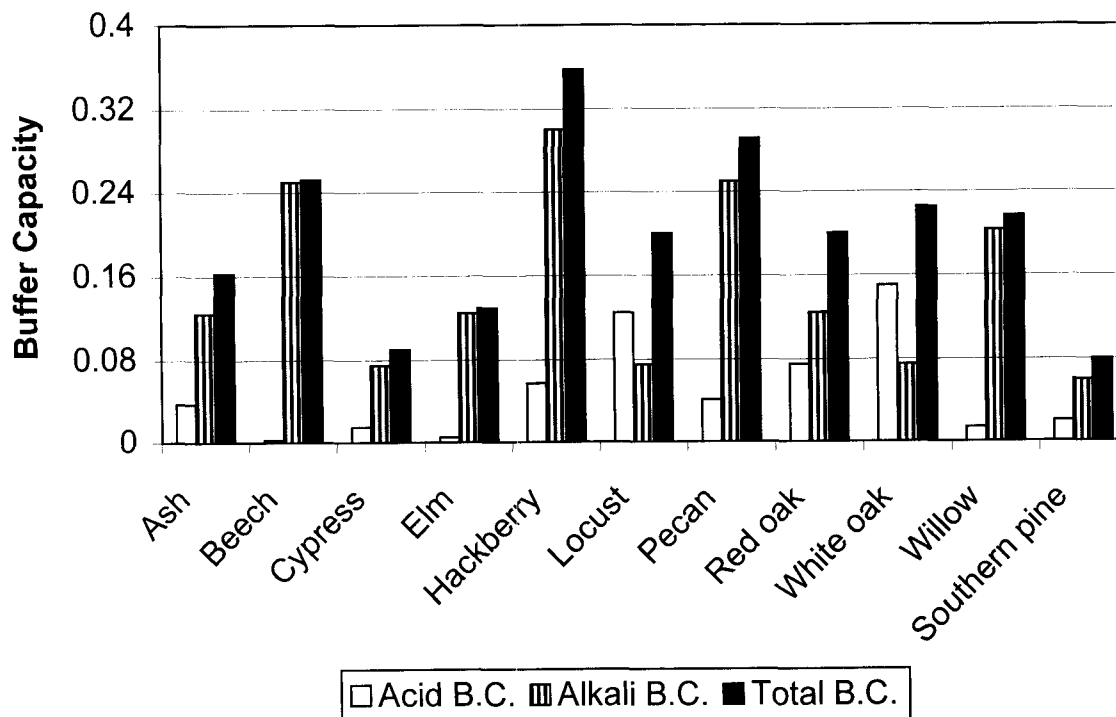


FIG. 2. A comparison of acid, base, and total buffer capacities from the eleven southern wood species.

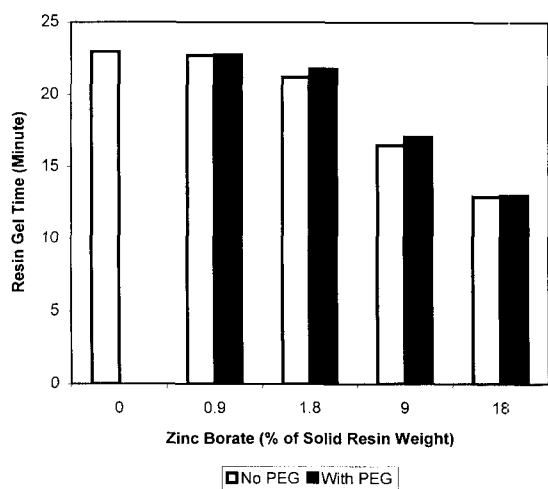


FIG. 3. Effect of zinc borate and polyethylene glycol on the gel time of neat phenol-formaldehyde face resin for oriented strandboard.

larger than the corresponding acid buffer capacity for most wood species tested. The total buffer capacity varied from 0.09 for cypress to 0.358 for hackberry. For the same species, both pH and buffering capacity values from this study were in a range similar to the data reported by Johns and Niazi (1980). However, it is not possible to statistically compare the values from the two studies because of differences in the wood sources and type (solid wood versus wood flakes).

PF resin gel time

Measured gel time for neat PF resin averaged 22.93 min (Fig. 3). The data agreed well with test results made by the resin manufacturer with similar equipment. It should be pointed out that the gel time varies in general with the amount of resin and heating methods used. For example, a water bath at 100°C tends to give a slightly lower gel time because of the boiling effect of water, compared with a

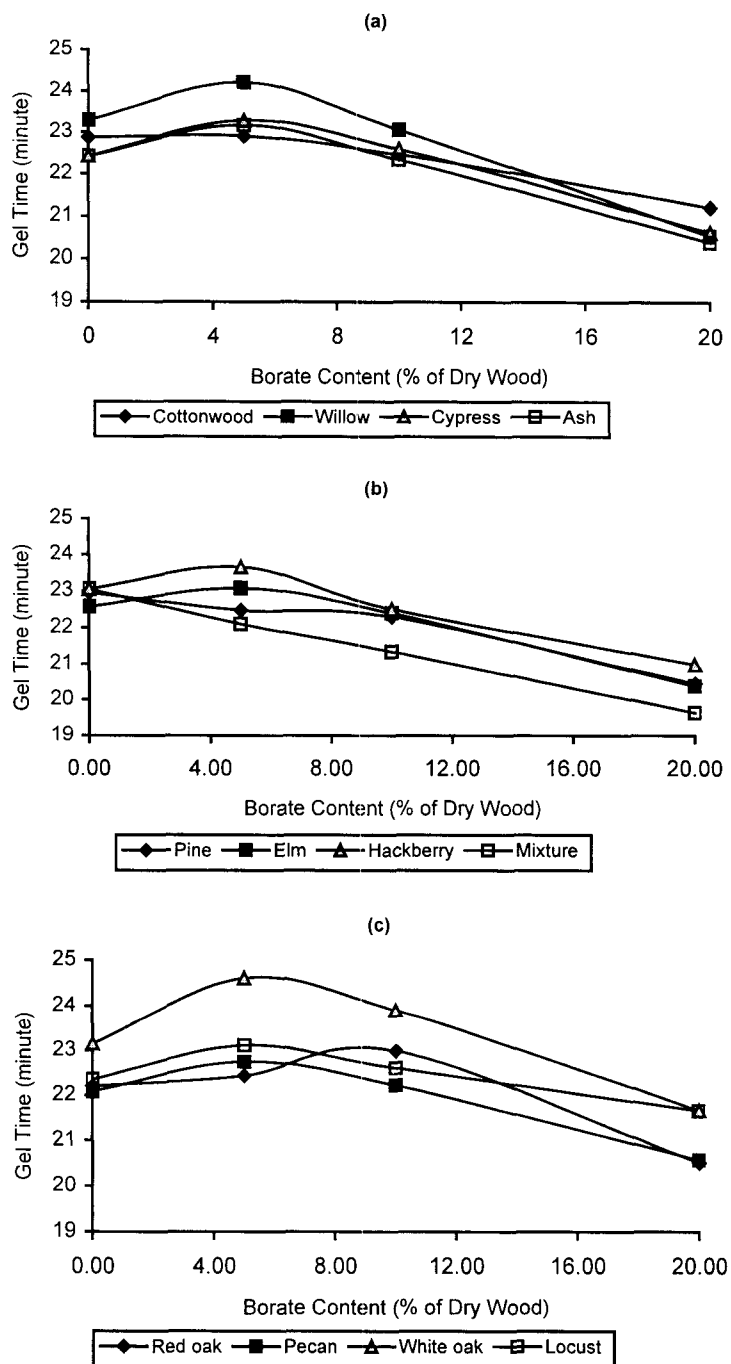


FIG. 4. Measured PF resin gel time as a function of zinc borate content for various wood species and their mixture. (a) low-, (b) medium-, and (c) high-density species groups.

silicon oil bath at the same temperature. Thus, the reported resin gel time should be considered as a relative measure among the treatments. Addition of zinc borate led to a reduced gel time as shown in the graph. At the 0.9% application level, the reduction was slightly over 1% (gel time = 22.65 min). As the amount of zinc borate increased, the gel time decreased significantly. At the 18% application level (based on solid resin weight), the gel time was reduced by over 43% (gel time = 12.9 min). This obviously indicates an interaction between PF resin and zinc borate. It is believed that boron and oxygen from methanol (CH_2OH) of the PF resin formed the coordinate bonding by donating the lone pair of electrons from oxygen to boron. Such an interaction is known to cause the resin to gel before it is able to develop an effective bond (Sean et al. 1999).

Addition of PEG helped recover some of the lost gel time at a given borate application level (Fig. 3). At the 40% PEG application rate, gel time was increased by 0.2, 2.7, 3.7, and 0.7% at the 0.9, 1.8, 9, and 18% borate levels (based on solid resin weight), respectively. Thus, the effect of PEG appeared to be more pronounced at the intermediate borate application levels. It is believed that the OH functional group of PEG disturbed the linkage between boron and the oxygen ion of CH_2OH in PF resin, making a weak linkage with borate.

The relationship between resin gel time and borate application rate for various wood species is shown in Fig. 4 (a: low-density species, b: medium-density species, and c: high-density species). Actual gel time data at various borate application rates are listed in Table 2. At the 0% borate application level (control), the gel time varied from 22.40 to 24.08 min among the species. The mean gel time for all species (22.67 min) was not significantly different from that of neat resin (22.93 min), indicating little effect of wood species alone. At the 5% borate loading rate, measured gel time for most species increased. Further increases in borate content led to decrease in resin gel

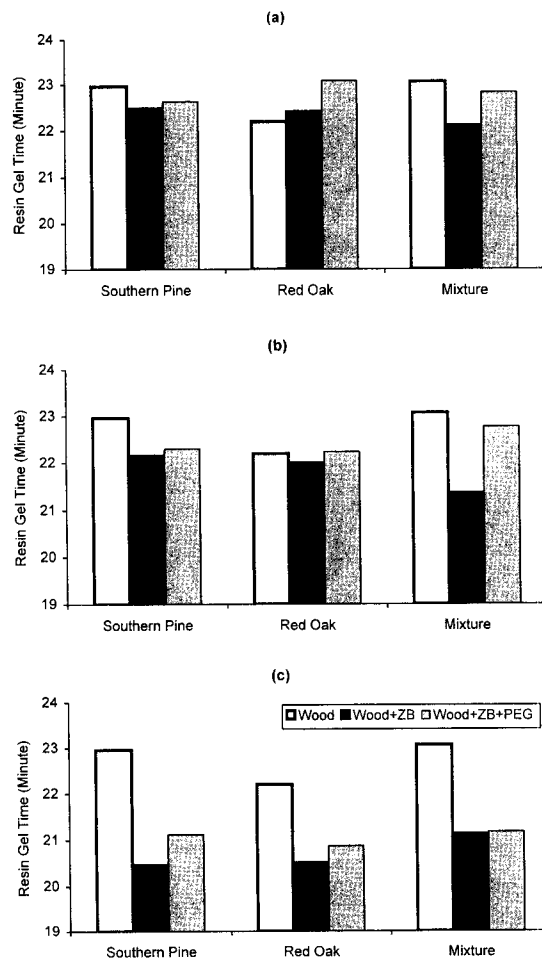


FIG. 5. A comparison of the PF resin gel time as influenced by zinc borate, polyethylene glycol, and wood (southern pine, red oak, and mixture of the eleven wood species). Borate content was 5% (a), 10% (b), and 20% (c) based on 0.5 g of oven-dry wood meal. Polyethylene glycol content was 40% based on the borate weight.

time. At the 20% loading rate (based on dry wood weight), the average gel time was reduced by about 8%.

Plots demonstrating a combined effect of wood (southern pine, red oak, and a mixture of the eleven species), zinc borate, and PEG on resin gel time are shown in Fig. 5 (a: 5% ZB, b: 10% ZB, and c: 20% ZB). The effect of borate and PEG on the gel time varied from species to species. At the 5% ZB application rate, the gel time was reduced by 2%, -0.1%

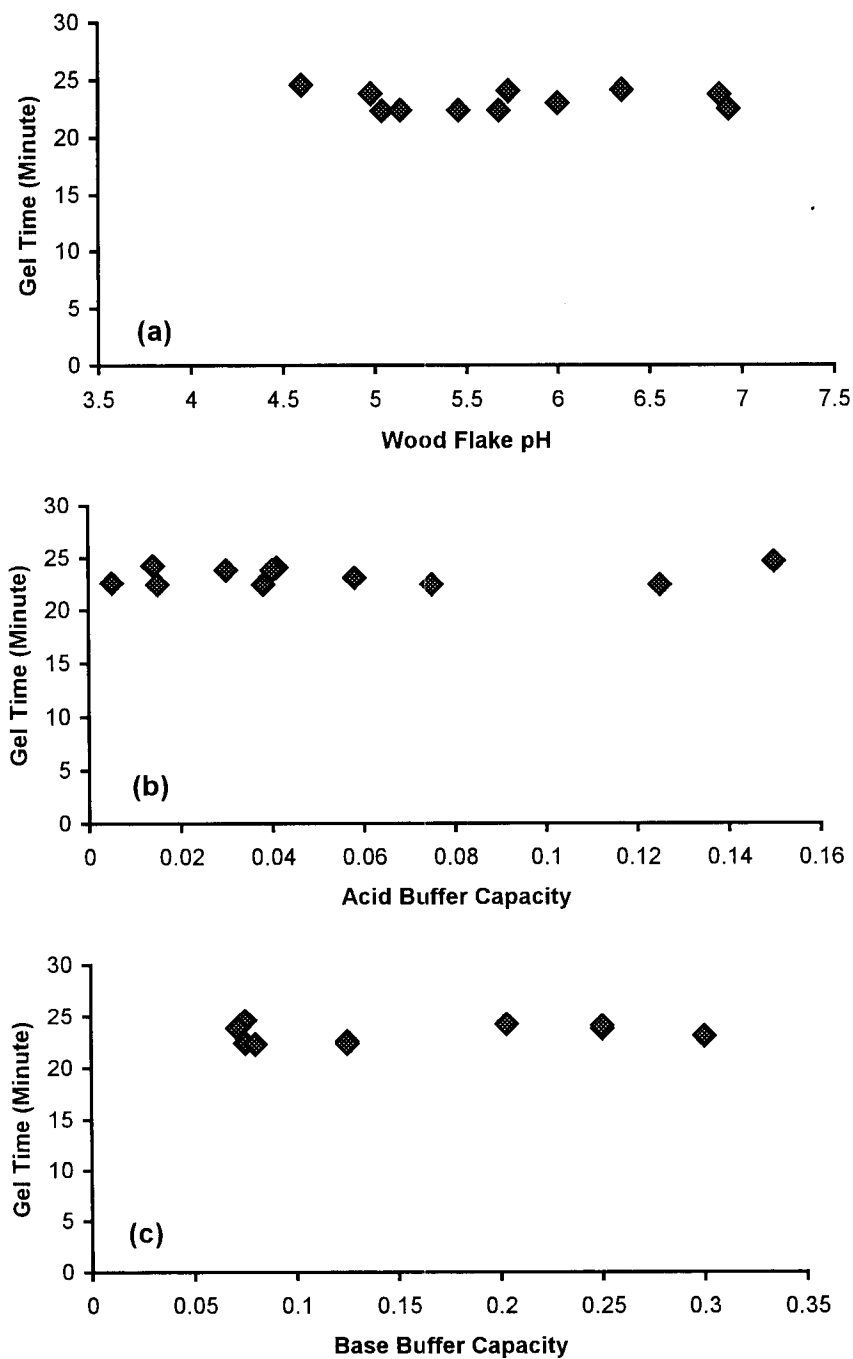


FIG. 6. Scatter-plots of resin gel time as a function of wood flake pH (a), acid buffer capacity (b), and base buffer capacity (c).

(an increase), and 4.2% for southern pine, red oak, and the mixture, respectively. Adding PEG to the resin-wood-borate mixture led to an increase in the gel time for all three species groups. At the 10% ZB application level, the gel time was reduced by 3.5% for southern pine, 1% for red oak, and 7.5% for the mixture. Adding 40% PEG at this borate application level led to a significant recovery of the reduced gel time for the mixture. At the 20% ZB application rate, there was an average of 9% reduction in the gel time for all three species groups. The use of PEG led to some recovery in the gel time, but the extent was smaller compared to the two smaller borate application levels, especially for the mixture. This indicates that PEG can help only to a certain extent in offsetting the negative effect of borate on the resin gel process. Once the borate content reaches a certain level, PEG may not provide significant improvement in the gel time. Sean et al. (1999) used PEG to improve the flow properties of phenol-formaldehyde resin in the presence of zinc borate for OSB manufacturing. They found an improvement in resin flow with the addition of the flowing agent. The curing time of PF resin increased markedly with an increased amount of PEG presence, as compared to the controls.

Correlation between flake chemical properties and resin gel time

There was no significant correlation between the gel time of PF resin and flake pH, acid or base buffer capacity (Fig. 6). Johns and Niazi (1980) found that the gel time of UF resin was directly correlated with acid buffer capacity for both softwood and hardwood extracts. They showed that the gel time of UF resin decreased with a decrease in wood pH and with an increase in acid buffer capacity. The difference may be because of the type of materials (solid wood versus wood flakes) and resin type (UF versus PF) used in the two studies.

CONCLUSIONS

The chemical properties of wood flakes from eleven southern species and gel time of

PF OSB face resin under the influence of wood, zinc borate, and PEG were investigated as part of a study with an ultimate goal of developing chemically modified OSB for residential construction. It was found that the pH of the southern hardwood flakes was acidic, in which white oak was the most acidic (pH = 4.60) and elm was nearly neutral (pH = 6.93). Flakes from southern pine had a relatively low pH value of 4.98, compared to the southern hardwood flakes. For most species tested, the alkali buffer capacity was larger than the corresponding acid buffer capacity. The total buffer capacity varied from 0.09 for cypress to 0.358 for hackberry. Wood species alone had little effect on the gel time of the resin. The measured gel time decreased as the amount of zinc borate increased. The reduced gel time was partially recovered by using PEG in combination with the borate. The effectiveness of PEG varied with wood species and the level of borate used. The gel time of PF resin-wood mixture had no direct correlation with the pH of wood and buffer capacity for the species studied. Work is on-going to develop borate-modified OSB using southern wood species and to investigate strength, long-term durability, and termite resistance of the product.

ACKNOWLEDGMENTS

The authors wish to acknowledge the financial and material support from the National Science Foundation (CMS-0080248), Neste Resins Corp., US Borax Corp., and Roy Martin Lumber Company.

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