Bond Quality of Soy-based Phenolic Adhesives in Southern Pine Plywood

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ABSTRACT: Increased demand for wood adhesives, environmental concerns, and the uncertainty of continuing availability of petrochemicals have led to recent attention on protein-based adhesives. This study was conducted to investigate the glue bond qualities of soy-based phenolic adhesive resins for southern pine plywood. Two types of soy-based resins were formulated and tested. The first was made by cross-linking soy flour with phenol–formaldehyde (pf) resins at neutral pH. The second type was obtained by cross-linking soy flour hydrolyzates with pf resin under alkaline conditions. Plywood bonded with the neutral phenolic soy resins containing 70% soy flour and 30% 1.6 g/cm^2 pf without the use of extenders and fillers compared favorably with the traditional southern pine pf glue mixes. Plywood bonded with alkaline phenolic soy resins, containing 40 or 50% 0.5 g/cm^2 PF with the addition of extender (19% corn-cob powder), provided better adhesive glue bond properties than traditional southern pine pf glue mixes. These results suggest that soy-based phenolic adhesive resins have potential for the production of exterior southern pine plywood.

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KEY WORDS: Exterior–grade adhesives, phenol–formaldehyde, plywood, soy-based phenolic adhesives, soy flour hydrolyzate.

Before the advent of modern synthetic adhesive resins, such as urea– and phenol–formaldehyde resins, soy glues were important adhesives for plywood production. Uncertainty about the future supply of petrochemicals and stringent regulations on toxic emissions have compelled wood industries to develop adhesive resins from renewable resources. Agricultural industries are also eager to invest in researching nonfood uses of agricultural products to expand their markets.

Carbohydrates and proteins are the most abundant renewable substances suitable for uses as wood adhesives. It is a significant challenge to develop adhesives from these natural substances at a reasonable cost that can compete with synthetic thermosetting adhesives and meet stringent performance requirements. A current concept to achieve this goal is to use these natural substances as copolymers with synthetic polymers to reduce the dependency on petroleum-derived chemicals. In this respect, proteins are more suitable than carbohydrates because amino, carboxyl, aliphatic and aromatic hydroxyl, and other functional groups in proteins provide various functionalities for chemical cross-linking.

The use of proteinaceous materials, such as blood, casein and soybean, as adhesive glues was common in the middle of 20th century. Golick and Dike (1) formulated exterior phenolic plywood glues containing up to 70% dried blood. Ash and Lambuth (2) prepared plywood glues containing a high content of blood solids blended with phenol–formaldehyde (PF) resins. Weakley and Mehltretter (3) developed moisture-resistant plywood adhesives by cross-linking casein with dialdehyde starch. The use of soy-based glues derived from the discoveries of Davidson was quite popular until 1960 (4). However, the soy-based glues were limited to mainly interior applications due to short pot life (i.e., the time for which the resin/hardener remains in a liquid state), poor biological stability, low solids, slow press times, and poor water resistance. Consequently, the soy-based glues were replaced by petroleum-based glues for exterior uses (5). However, since late 1990s, soybeans have been extensively studied for their use as an exterior-grade adhesive resin as a result of recent increases in petroleum prices, formaldehyde emission concerns, and phenol safety issues.

Recent successes in the development of durable soy-based resins have included improved adhesive strength in wood adhesives prepared with chemically (6–8) and enzymatically (6,9) modified soy proteins. In 1997, a cold-setting adhesive for finger-joint lumber was invented by Steele *et al*. (10), and the adhesives contained equal parts of soy protein isolates and phenol—resorcinol–formaldehyde (PRF) resin (10). The adhesive is being produced and sold by a chemical company in Oregon. Riebel *et al*. (11) described methods of preparing a soybean-based molding compound by cross-linking soy flour with 12% methylene diphenyl isocyanate. PF–cross-linked soy resins comprising 70% soy flour and 30% PF resin were developed by Kuo *et al*. (12) and Kuo and Stokke (13). A similar PF–cross-linked soy resin comprising 30% soy flour and 70% PF resin also has been developed by Hse *et al*. (14). However, most of the studies focused on identifying the relationship between adhesive strength and different methods for denaturing and hydrolyzing soy proteins. In addition, several studies were conducted to evaluate the potential of soy-based plywood glues (15–17), but the studies did not provide an extensive understanding of newly developed soy-based adhesive systems for plywood production. Therefore, the objective of this study was to fully explore the potential of soy-based phenolic resins developed at Iowa State University for the production of exterior plywood panels.

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EXPERIMENTAL PROCEDURES

Preparations of soy solution and soy hydrolyzate. An aqueous solution of defatted soybean flour (Honey Meal 90; Harvest State Oilseed, Mankato, MN), containing about 54% protein, was prepared by dissolving 1.25% sodium bisulfite ($NaHSO_3$) in 250 mL water for each 100 g of moisture-free soy flour. The pH was then adjusted to neutral with 50% aqueous sodium hydroxide solution. The solution was heated and maintained at 50°C, followed by the addition of 0.5 mL cedarwood oil (Fisher Scientific Co., Fair Lawn, NJ) used as a defoamer. Subsequently, 100 g of dry soy flour was slowly added with vigorous stirring, resulting in a viscous but smooth and homogeneous solution. For soy hydrolyzates, soybean flours were hydrolyzed with 10% NaOH in water at 140°C for 2 h. The viscosities of soy hydrolyzate at pH 9.5, 36% solids, and 25°C were 10.9 g/cm^2 at 10 rpm and 2.7 g/cm^2 at 100 rpm, respectively.

Adhesive resins. Two types of soy-based phenolic resins, neutral phenolic soy (NPS) resin and alkaline phenolic soy (APS) resin, were formulated for the laboratory production of three-ply southern pine plywood. The resins were used to identify the relationship between pH of the resins and the curing property of the resins in a plywood panel. For the preparation of the NPS and APS resins, PF resin was prepared as follows; a mixture of 1 mole phenol, 2.4 moles formaldehyde, and 0.1 mole NaOH with a sufficient amount of water was heated at 65°C for 1.5 h. Then the mixture was heated to 95°C for 60 and 75 min to make 0.5 and 1.6 g/cm^2 PF resins, respectively. The characteristics of the PF resins were 55% solids and pH 9.5 at 25°C. The PF resins with different viscosities were formulated to examine the effect of PF viscosity on the adhesive strength of soy-based resins. To formulate APS and NPS resins, soy solution and soy hydrolyzates were heated to 50°C, followed by the slow addition of PF resins with vigorous stirring for 20 min. NPS resins were prepared by cross-linking soy solution with 0.5 and 1.6 g/cm^2 PF resins at a neutral pH. APS resins were formulated by cross-linking soy hydrolyzates with PF resins under alkaline conditions. The APS resin had a solid content of about 42%, a pH of 9.3 to 9.5, and a viscosity range of 10–15 g/cm^2 depending on the soy hydrolyzate/PF composition. The final formulations of the NPS and APS resins varied in different soy-to-PF weight ratios (70:30, 60:40, and 50:50) and in two PF viscosities (0.5 and 1.6 g/cm^2). A control PF resin with a resin solids level of 50% and a viscosity level of 2 g/cm^2 was obtained from Borden Chemical Company (La Grande, OR).

Glue mix formulations. Commercial wheat flour (WF) and corn-cob powder (CCP) were used as extenders and fillers to reduce the amount of the primary binder required per unit area and to improve the working properties, strength, and other qualities of the glue mix formulations. The composition of glue mixes prepared with NPS and APS resins is shown in Table 1. Owing to the high viscosity and low solids content of the NPS resin as described by Kuo *et al.* (12), NPS resin is suitable for plywood bonding, softwood and hardwood laminating, and paper overlay bonding. In particular, the NPS resin containing 1.6 g/cm^2 PF was very viscous, and thus the glue mixes were formulated without extenders and fillers. APS resin with a solids content of 45% was less viscous than NPS resin (13). The APS resin did not show any difficulties in application when subjected to a high shear environment such as a spraying process. Therefore, APS glue mixes were formulated with WF and/or CCP to obtain lower-cost, better-performing resins.

Plywood preparation and testing. Southern pine (*Pinus* spp.) veneers (30 × 30 × 0.3 cm thick), which were produced by rotary lathe, were obtained from Southern Research Station, Forest Products Laboratory, USDA (Pineville, LA). Veneers with a constant thickness and density were used to minimize experimental errors. The veneers were adjusted to 4% moisture content before the glue mix was applied. The glue mix was applied with a brush at a rate of 410 to 420 kg/1,000 m^2 of double glue lines. The three-ply assemblies were subjected to 30- and 60-min closed assembly times, meaning the time interval between glue and pressure (or heat or both) application (18) at room temperature. Then the assembly was pressed with a 14 kg/cm^2 pressure for 8 min. All assemblies were pressed at 175°C with the exception of the assemblies applied by APS resin glue mixes with 19% CCP alone. The assemblies were pressed at 200°C to examine the effect of press temperature on the curing properties of the APS glue mixes. Each glue mix and pressing treatment was replicated three times.

Plywood panels were conditioned in the laboratory (65% RH and 25°C for 24 h) before cutting each panel into 30 shear

TABLE 1
Compositions of Soy-Based Phenolic Resin Glue Mixes for Southern Pine Plywood[a]

	Soy-to-PF weight ratio	Extender level
Neutral phenolic soy resin		
50 cP	70:30, 60:40, 50:50	No extender
		7% WF and 10% CCP
		14% WF and 19% CCP
160 cP	70:30, 60:40, 50:50	No extender
Alkaline phenolic soy resin		
50 cP	70:30, 60:40, 50:50	14% WF and 19% CCP
50 cP	70:30, 60:40, 50:50	19% CCP
Control PF resin		
200 cP (Borden PF resin)	0:100	14% WF and 19% CCP

[a]cP, centipoise (mPa·s); PF, phenol–formaldehyde; WF, wheat flour; CCP, corn-cob powder.

specimens. Ten random test specimens from each panel were tested in dry condition. Another 10 random specimens were tested after vacuum-pressure soaking (19). The shear test was done with a universal test machine (MTI 5K; Measurements Technology Inc., Roswell, GA) with a 1 in./min load rate, and the glue bond quality of the plywood panel was determined by dry and wet percent wood failure, which means the rupturing of wood fibers in shear strength tests on bonded plywood specimens (18), according to the adhesive bond requirements for an exterior plywood panel in the U.S. Product Standard 1-95 (19).

Experimental design and statistical analyses. This experiment was analyzed as a 3×2 factorial design. Analysis was carried out with the Statistical Analysis System (20) programming package. A 95% confidence level was used in all statistical tests. Effects of each variable were examined by the General Linear Model procedure. Significant effects with a $P < 0.05$ were further characterized by Tukey's significant difference test between means.

RESULTS AND DISCUSSION

Adhesive glue bond properties of southern pine plywood bonded with NPS resin. Table 2 shows that plywood bonded with NPS resins had a broad range of dry percent wood failure (DWF), ranging from 45 to 82%. The corresponding range for wet percent wood failure (WWF) was from 16 to 86%. According to U.S. Product Standard PS 1-95, the glue bond quality of NPS and APS resins was determined by DWF and WWF (19).

(i) Effects of PF dosage on glue bond quality of NPS resin. For the unextended soy/0.5 g/cm^2 PF resin, increasing the PF level in NPS resins improved DWF and WWF ($P = 0.01$). Increasing the PF level from 30 to 40% did not improve DWF but did increase WWF. When the PF level increased from 40 to 50%, no significant differences in DWF and WWF were found. WWF showed more increase when the PF level in NPS resins was increased from 30 to 40% than from 40 to 50% (Fig. 1). For the unextended soy/1.6 g/cm^2 PF resins, the resins produced nearly 80% DWF (Table 2). The PF level in the resins ($P = 0.50$) and assembly time ($P = 0.60$) had no significant effects on DWF. When the PF level increased from 30 to 40%, WWF did not increase. However, further increase in the PF level from 40 to 50% significantly improved WWF (Fig. 1). These results may be due to the difference in degree of resin curing. In this study, PF resin was added to improve the degree of cross-linking in NPS resins. Consequently, higher PF levels in unextended NPS resins increased the cross-linking in cured glue lines. Assembly time did not affect DWF ($P = 0.63$) and WWF ($P = 0.11$) of plywood bonded with unextended soy/0.5 g/cm^2 PF resins. From these results, the unextended soy/1.6 g/cm^2 PF resins formulated with 50% PF with a 60-min assembly time provided the best plywood glue bond quality and com-

TABLE 2
Adhesive Glue Bond Properties[a] of Southern Pine Plywood[b] Bonded with Neutral Phenolic Soy Resins and Pressed at 175°C

PF type (cP)	Soy/PF (w/w)	Total solids (%)	PF solids (%)	Extenders in solid	Assembly time (min)	DWF[b] (%)	DSS[b] (psi)	WWF (%)	WSS (psi)
Control		44.1	29.5	14% WF + 19% CCP	30	92	177	63	106
50	70:30	37.4	11.2		30	50	164	47	88
					60	57	158	42	62
	60:40	40.4	16.1		30	60	182	67	123
					60	61	201	52	105
	50:50	41.4	20.7		30	70	200	67	127
					60	68	227	67	144
50	70:30	38.6	9.6	7% WF + 10% CCP	30	57	175	30	86
					60	45	190	20	68
	60:40	40.8	13.5	7% WF + 10% CCP	30	74	216	50	116
					60	61	204	24	82
	50:50	42.2	17.5	7% WF + 10% CCP	30	68	218	48	116
					60	61	239	45	123
	70:30	40.5	8.1	14% WF + 19% CCP	30	62	179	43	105
					60	45	134	16	45
	60:40	43.1	11.6	14% WF + 19% CCP	30	82	235	60	122
					60	76	216	49	109
	50:50	41.9	14.1	14% WF + 19% CCP	30	64	211	50	118
					60	58	155	43	87
160	70:30	41.2	12.4		30	79	261	79	149
					60	77	232	74	118
	60:40	42.3	16.9		30	82	229	80	152
					60	81	228	79	146
	50:50	39.9	20.0		30	79	260	84	172
					60	77	233	86	149

[a]Corn-cob powder percentage based on total solids; DWF, dry wood failure; DSS, dry shear strength; WWF, wet wood failure; WSS, wet shear strength. For other abbreviations see Table 1.
[b]Each value is the average of 30 tests, 1 test per specimen, 10 specimens per panel for three plywood panels.

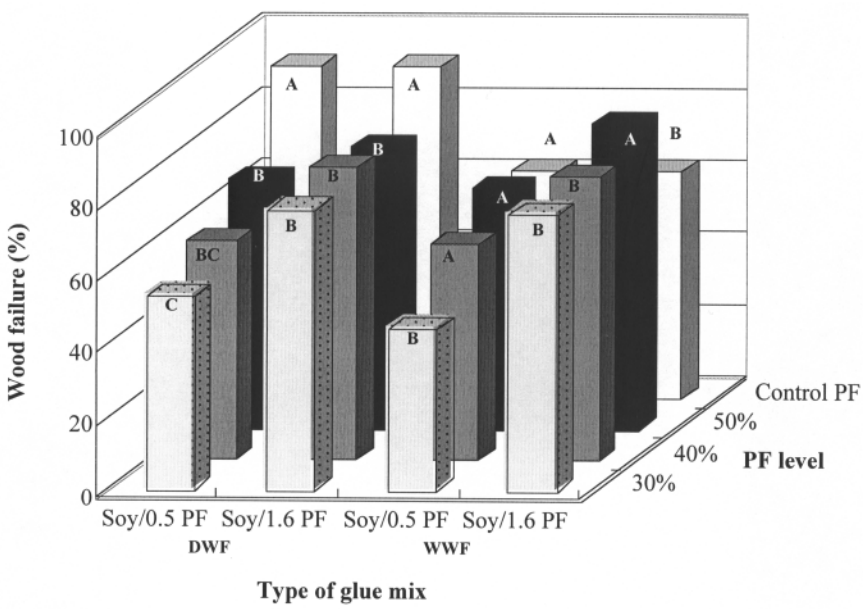

FIG. 1. Effect of phenol–formaldehyde (PF) level on dry wood failure (DWF) and wet wood failure (WWF) of southern pine plywood panels with neutral phenolic soy (NPS) resins formulated without any extenders. Bars within a column having the same letter above them do not differ significantly at $P = 0.05$ (Tukey's significant difference test).

pared favorably with the plywood bonded with the control PF glue mix (Table 2).

(ii) Effects of extender on glue bond quality of NPS resin. The addition of extender in soy/0.5 g/cm^2 PF resins increased DWF (Fig. 2). The improvement of DWF that the addition of an extender provides is due to the resultant uniformity of adhesive ingredients, filling of holes, fewer irregularities of veneer surfaces, and the decrease in porosity of the surface for consistent bonding (18). The use of extender in the soy/0.5 g/cm^2 PF resins had a negative effect on WWF. Inclusion of extender in the resins caused a drastic viscosity increase, making glue spreading very difficult. Therefore, NPS resins do not need an

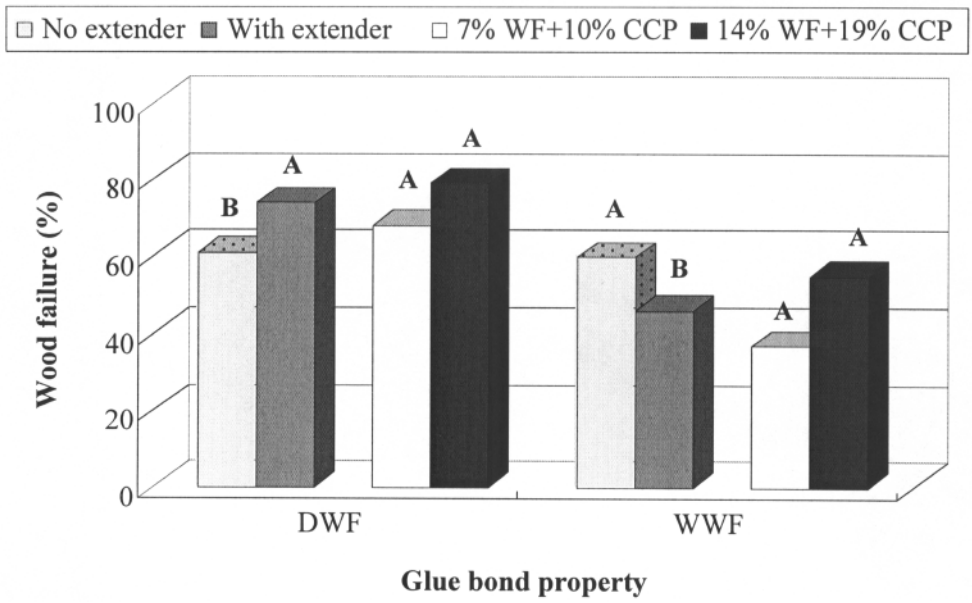

FIG. 2. Effect of extender level on DWF and WWF of southern pine plywood panels with NPS resins. WF, wheat flour; CCP, corn-cob powder; for other abbreviations see Figure 1. Bars within a grouping having the same letter above them do not differ significantly at $P = 0.05$ (Tukey's significant difference test).

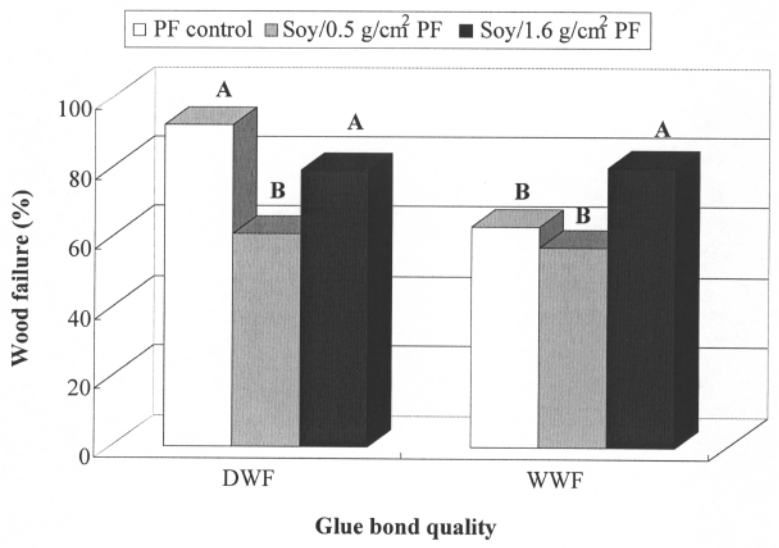

FIG. 3. Effect of PF type on DWF and WWF of southern pine plywood panels with NPS resins. For abbreviations see Figures 1 and 2. Bars within a grouping having the same letter above them do not differ significantly at $P = 0.05$ (Tukey's significant difference test).

extender, because soy flour may act in the resins both as extender and as filler. In addition, the foaming property of soy flour may contribute to the increased performance of soy resins for plywood bonding (16,17). There were no significant differences between the two levels of extender content for DWF and WWF (Fig. 2). In extended soy/0.5 g/cm^2 PF resins, WWF decreased significantly when the assembly time was lengthened from 30 to 60 min. The results indicate that glue line dehydration and pre-cure of the resin, especially at long assembly times, might cause a decrease in WWF.

(iii) Effects of PF type on glue bond quality of NPS resin. Soy/1.6 g/cm^2 PF resin demonstrated higher glue bond properties in both dry and wet tests than did soy/0.5 g/cm^2 PF resins (Fig. 3). The difference in glue bond properties might be due to different resin penetrations into veneer. The difference in resin penetration was observed by microscopy (Fig. 4). For instance, soy/0.5 g/cm^2 PF resin is more penetrated than soy/1.6 g/cm^2 PF into cells. The molecular size of the soy/1.6 g/cm^2 PF resins may be larger than that of soy/0.5 g/cm^2 PF resin. The molecular size therefore may prevent resins from overpenetration into

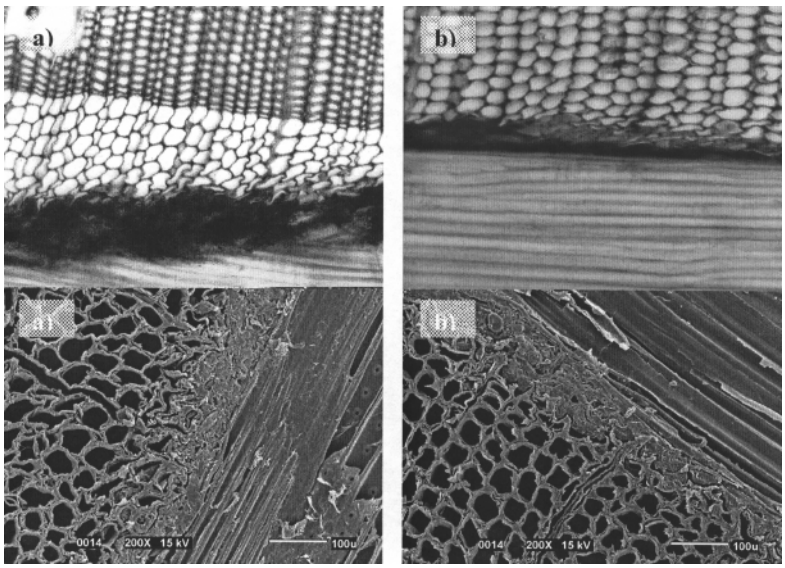

FIG. 4. Light microscopy (upper) and scanning electron microscopy (lower) images of plywood panels bonded with NPS resins; (a) soy/0.5 g/cm^2 PF resin was more penetrated than (b) soy/1.6 g/cm^2 PF into cells. For abbreviations see Figure 1.

TABLE 3
Adhesive Glue Bond Properties[a,b] of Southern Pine Plywood Bonded with Alkaline Phenolic Soy Resins

SH/PF (w/w)	Total solids (%)	PF solids (%)	Extenders in solid	pH	Press temp. (°C)	Assembly time (min)	DWF (%)	DSS (psi)	WWF (%)	WSS (psi)
Control	44.1	29.5	14% WF + 19% CCP	9.4	175	30	92	177	63	106
70:30	47.6	9.6	14% WF + 19% CCP	8.6	175	30	83	257	19	86
					175	60	70	212	38	99
60:40	48.9	13.1	14% WF + 19% CCP	8.7	175	30	82	227	33	111
					175	60	93	231	57	121
50:50	48.5	16.3	14% WF + 19% CCP	8.9	175	30	82	249	38	121
					175	60	81	228	63	140
70:30	52.4	11.8	19% CCP	10.3	200	60	87	97	45	60
60:40	52.4	15.8	19% CCP	10.2	200	60	97	148	79	109
50:50	52.3	19.7	19% CCP	10.3	200	60	100	131	79	130

[a]SH/PF, alkaline soy hydrolyzate/50 cP PF resins; for other abbreviations see Tables 1 and 2.
[b]Each value is the average of 30 tests, 1 test per specimen, 10 specimens per panel for three plywood panels.

veneer during the resin curing. The proper resin penetration might appear to be the main reason for high glue bond properties of the resin. In addition, soy/1.6 g/cm^2 PF resin is more viscous than soy/0.5 g/cm^2 PF resin, and thus the highly viscous resin might reach a gel point faster than soy/0.5 g/cm^2 PF resin. Wet glue bond properties of plywood bonded with the soy/1.6 g/cm^2 PF resin, regardless of PF level and assembly time, were superior to those of plywood bonded with the PF control glue mix. The soy/1.6 g/cm^2 PF resins also showed appropriate consistency as plywood glues.

Although soy/1.6 g/cm^2 PF resins did not meet the WWF level of 85% in U.S. Product Standard PS 1-95 (19), the results of this study showed that the NPS resin has potential use in the manufacture of exterior-grade southern pine plywood. However, for industrial applications of the NPS resin, more work will be required to find the optimal viscosity of PF resin in NPS resins. If such resins are developed successfully, the use of the NPS resin might contribute to reduce overall PF resin content for the manufacture of southern pine plywood. The light-color glue line of the NPS resins might provide a potential for decorative wood applications.

Adhesive glue bond properties of southern pine plywood bonded APS resin. Table 3 shows the adhesive glue bond properties of plywood bonded with APS glue mixes, which incorporated soy flour hydrolyzates with 0.5 g/cm^2 PF resin.

(i) Effects of PF dosage on glue bond quality of APS resin. In the glue mixes of APS resin formulated with 14% WF and 19% CCP, DWF was not affected by PF level in APS resin (p = 0.11), but WWF increased with increasing PF level (Fig. 5). These results indicate that APS glue mixes with a higher PF

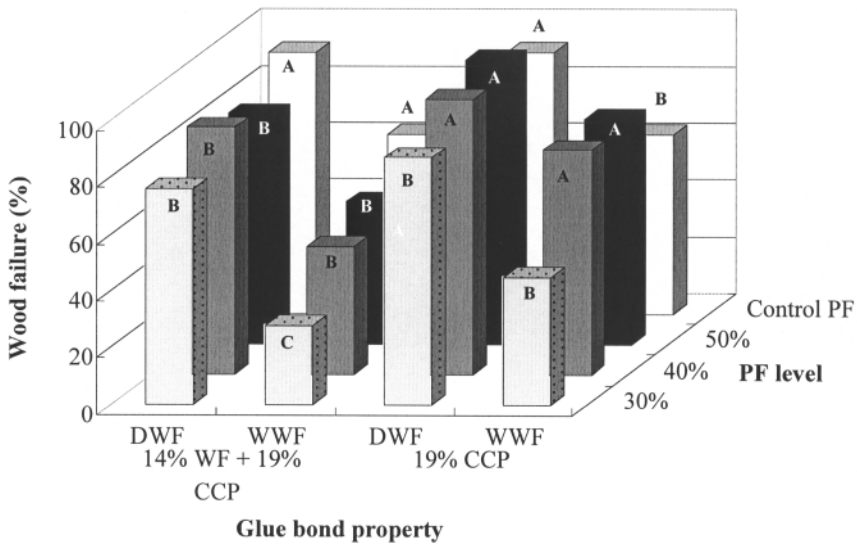

FIG. 5. Effects of PF level and extender level on DWF and WWF of southern pine plywood panels with alkaline phenolic soy resins. For abbreviations see Figure 1. Bars within a column having the same letter above them do not differ significantly at P = 0.05 (Tukey's significant difference test).

level might have a more cross-linked network in cured glue lines than one with a lower PF level. In addition to the more cross-linked network, the molecular size of the more cross-linked resins might be suitable to prevent the resins from overpenetration into veneers. WWF also improved by lengthening of assembly time ($p = 0.01$). For instance, during the 60-min assembly, water in glue lines is absorbed into veneers or evaporated, resulting in increased viscosity of glue mixes. Consequently, the glue mixes might prevent resin from overpenetration into the wood surface.

(ii) Effects of extender on glue bond quality of APS resin. Plywood panels were prepared with other APS glue mixes formulated with just 19% CCP. Increasing PF level in the APS resins had significant effects on DWF and WWF (Fig. 5). At 40 and 50% PF levels, DWF values of the plywood panels were over 95%, but the DWF was decreased to 87% when the PF level decreased to 30%. With PF levels of 40 and 50%, the plywood glue bonds were very durable, having 79% WWF values. But at 30% PF level in the APS resins, there was a drastic reduction in the WWF below 50%. These results might be attributed to more cross-linked cured glue lines due to the combination of high press temperature and PF level in the APS resins. Besides greater cross-linking, the exclusion of WF in the APS glue mixes showed the improvement of WWF. WF, used as filler in the glue mix of plywood panels, typically contains approximately 70% carbohydrate, and the carbohydrate adversely affects the wet adhesive properties of an adhesive for the production of plywood panels (18). Consequently, the APS glue mixes formulated without WF may lead to the reduction in detrimental effect of WF on WWF.

APS resin glue mixes—with 40 or 50% PF level and 19% CCP as an extender, and pressed at 200°C press temperature—produced WWF superior to those of a control PF glue mix (Fig. 5). Furthermore, the WWF approached the requirement of 85% for construction and industrial plywood (19). At 60-min assembly, plywood bonded with APS glue mixes containing 14% WF and 19% CCP and a 40 or 50% PF level had comparable glue bond properties with those bonded with the control PF glue mix. These results suggest that APS resins have a potential for the production of exterior-grade southern pine plywood.

The use of NPS and APS resins might provide cost savings and durable adhesive properties as well as a more environment-friendly substitute for phenol–formaldehyde adhesives. However, the optimal viscosity, curing mechanism, press temperature, and extender types are not yet fully characterized.

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