

Development of Formaldehyde-Based Wood Adhesives with Co-Reacted Phenol/Soybean Flour

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Abstract

Ten resins, each with two replicates, were formulated from soy flour hydrolyzates and prepared with two hydrolyzation variables: phenol-to-soy flour ratios (w/w) of 4/4, 3/4, 2/4, 1/4, and 0/4 and sodium hydroxide-to-soy flour ratios (w/w) of 0.39 and 0.78. In addition, two commercially available phenol formaldehyde (PF) resins were used in the study as controls. The most interesting result in the study of resin properties was gel time. When soy flour was hydrolyzed in the presence of phenol, the gel times were consistently longer with low caustic hydrolysis than that of high caustic hydrolysis. When soy flour was hydrolyzed without phenol, however, the gel times were longer with high caustic hydrolysis than that of low caustic hydrolysis. The internal bond (IB) strengths of oriented strandboard panels made with co-reacted soy hydrolyzate resins were highly comparable with boards made with the two phenolic control resins. When soy flour is hydrolyzed under high caustic conditions in the presence of phenol, acceptable co-reacted resins resulted; while under low caustic hydrolysis conditions with low phenol or no phenol at all, the best IB strengths were achieved. The overall high performance of the co-reacted resin prepared with phenol-to-soy flour ratio of 1/4 under low caustic hydrolysis was the most interesting. It not only yielded the highest IB, attained one of the best dimensional stability results among all the co-reacted resins, and ranked high in modulus of elasticity. It also is significant that it was able to hydrolyze soy flour at low caustic content while maintaining a workable viscosity.

With a 30-percent phenol substitution with soy flour hydrolyzate, a co-reacted soy hydrolyzate resin system as developed in the study could result in more than 20-percent saving in material cost as compared to a conventional 2/1 ratio (F/P) phenolic resin system used for bonding structural flakeboards.

Introduction

Adhesives based on soybean protein were important in the early development of the Douglas-fir plywood industry. Soybean adhesives produced a strong bond which, although not waterproof, was water resistant. At the peak in 1942, the amount of soybean glue used for gluing plywood, together with a small amount of casein glue, represented 85 percent of the total softwood plywood glue production (1). However, the development of highly durable, exterior-grade synthetic adhesives from petrochemicals resulted in a long-term decline in the production of soybean adhesives. Cold-press soybean adhesives fell from about 28 percent of the market in 1954 to almost zero in 1970; while hot-press soybean adhesives (containing dried blood) rose to a peak of 45 percent in 1956, then fell to near zero in 1973 (3). Nevertheless, intermittent declines in petroleum supplies and increases in petroleum prices, together with the worldwide interest in the development of environmentally friendly adhesives from renewable resources, have stimulated a renewed interest in soybean adhesives. Efforts have been made to find new ways to overcome limitations inherent in the use of soy flour when gluing wood. A recent study

(5) has shown that a concentrated soy protein-based resin was capable of finger jointing green lumber at room temperature when used with **phenol-resorcinol-formaldehyde (PRF)** resin in a modified finger-jointing operation. Altering the structure of the soy protein to reduce costs, Vajayendran (7,8) and Clay (2) have shown that a mixed hydrolyzed soy flour/PRF resin system can be applied to the two sides of lumber at room temperature in finger-joint operations. More recently, soybean-based adhesive resins were formulated by crosslinking hydrolyzed soy flour with 30 percent (w/w) of phenol formaldehyde (PF) prepolymer for bonding medium density fiberboard (MDF) and flakeboard that met the product performance requirements (6).

It is generally recognized that for soy protein to become a useful base for adhesives, native soy protein needs to be hydrolyzed to break the internal bonds and uncoil the polar protein molecules. This exposes the functional groups of the protein complex and increases its water solubility and surface area. The soy protein hydrolyzate can then be readily mixed as a co-resin with conventional PRF or PF resins to facilitate the chemical reaction between functional groups as well as molecular entanglement of soy protein and PRF or PF resins to form a highly crosslinked thermoset matrix. Almost all previously mentioned studies have followed this general approach.

While combining polymers of different properties has been increasingly practiced by the plastic industry to obtain new and useful materials (4), it also hindered the development of highly reactive polymer blends. In this study, a new approach was used to hydrolyze low-cost soy flour in the presence of phenol. This hydrolyzate was then substituted for phenol in conventional phenolic resin synthesis. The objective of this new approach is to obtain a co-reacted resin system containing a soy flour hydrolyzate that is suitable for bonding exterior structural panels.

Experimental Methods

Alkaline Hydrolysis of Soy Flour

Soy flour was hydrolyzed in a Parr reactor equipped with a stirrer drive system. The variables used in the hydrolysis reaction were:

- five phenol-to-soy flour ratios (w/w): 4/4, 3/4, 2/4, 1/4, and 0/4, and
- two sodium hydroxide-to-soy flour ratios (w/w): 0.39 and 0.78.

Thus, 10 soy flour hydrolyzates were prepared.

To initiate the hydrolysis, the ingredients (i.e., soy flour, phenol, and sodium hydroxide) were weighed and placed in the reactor. The concentration of the ingredient mix was adjusted to 46 percent by the addition of

water. After pre-mixing for 5 minutes at room temperature, the heater was turned on and temperature controlled at 120°C. After continuous agitation for 60 minutes, the hydrolysis reaction was terminated by removing the reactor from the heater and cooling it to room temperature with running tap water. The hydrolyzate was then transferred into containers and stored at room temperature before use. Viscosity, pH, solid content, and alkaline content of the hydrolyzates were determined.

Resin Preparation

All phenol-based, co-reacted soy flour hydrolyzate resins were synthesized in the laboratory. A total of 10 resins, based on the 10 hydrolyzates, were formulated. The general resin formulation conditions were:

1. the molar ratio of formaldehyde-to-(phenol + soy flour hydrolyzate)-to-sodium hydroxide was controlled at 1.67/1/0.45,
2. the solid content of the reaction mixture was maintained at 46 percent by water addition,
3. the soy flour replaced 30 percent of phenol by weight, and
4. the molar weight of soy flour was assumed to be equal to that of phenol.

To prepare each resin all phenol, soy flour hydrolyzates, and water were placed in a resin reaction kettle. Formaldehyde was added in four equal parts at 5-min. intervals. To initiate the reaction, the mixture was heated and maintained at 80°C to promote the addition reaction for the formation of the methylol phenols. At 20 minutes reaction time, the reaction temperature was raised gradually to 95°C in 40-min. intervals, and this temperature was maintained for 30 minutes to promote the condensation reaction. Additional sodium hydroxide was introduced with four consecutive additions of 0.3 moles sodium hydroxide each at 40-min. intervals beginning at the reaction time of 90 minutes. The sodium hydroxide additions were also accompanied with a 10°C temperature decrease after each sodium hydroxide addition. All reactions were terminated by rapidly cooling the mixture to 25°C at 240 minutes. Gel time, pH, solid content, and viscosity were determined. The gel time was measured with a Sunshine gel timer at 100°C, and the viscosity was measured at room temperature (25°C).

Flakeboard Manufacture

All panels were made in the laboratory with mixed hardwood flakes obtained from a local flakeboard plant. Two flakeboards were fabricated for each resin adhesive. Thus, a total of 20 panels were made.

To prepare each panel, flakes were weighed and placed in a rotating drum-type blender. The resin blend,

Table 1.—Physical properties of resin adhesives.

Sample ID	P/S ratio ^a (w/w)	Solid content (%)	pH	Viscosity (cps)	Gel time (min.)
High caustic hydrolysis					
100 H	(4/4)	51.5	12.89	320	43.8
75 H	(3/4)	51.5	12.62	380	40.5
50 H	(2/4)	51.6	12.82	370	36.9
25 H	(1/4)	51.7	12.77	510	36.7
OH	(0/4)	52.2	12.72	410	43.2
Lowcaustichydrolysis					
100 H	(4/4)	51.1	12.64	710	56.3
75 H	(3/4)	51.2	12.61	420	55.6
50 H	(2/4)	51.5	12.68	470	48.2
25 H	(1/4)	51.7	12.81	410	49.2
OH	(0/4)	51.6	12.80	460	35.3
Phenol formaldehyde resin					
PF-A		55.0	10.2	600	17.2
PP-B		51.0	11.10	340	22.1

^a Phenol-to-soy flour ratio in hydrolysis reaction.

in amounts equal to 4.5 percent of the oven-dry weight of flakes, were then weighed and applied by air-atomizing nozzles. Average moisture content of the flakes after spraying was 11 percent.

After blending, the randomly oriented flakes were carefully felted into a 17.5 by 20-in. box to form the mat. The mat was transferred immediately to a 20- by 20-in. single-opening hot press with the platen temperature regulated at 188°C. Sufficient pressure (about 550 psi) was applied so that the platens closed to 0.5-m stops in approximately 45 seconds. Press time was 4 minutes, 15 seconds after closure. All panels were hot-stacked in a wood box overnight immediately after removing from the hot press.

Sampling and Testing

The flakeboards, removed from the hot-stack box, were trimmed to 14- by 16.5-in. panels. After trimming, each panel was cut to yield three static-bending specimens (2-by 14-in.), two-dimensional stability test specimens (2- by 14-in.), and twelve specimens (2- by 14-in.) for tensile strength perpendicular to the face (IB).

Bending and IB specimens were evaluated according to ASTM D1037-72. For durability evaluations, an oven-dry to vacuum pressure soak test (ODVPS) was employed with the following constraints:

1. dried at 100°C oven for 24 hours,
2. placed in a pressure cylinder and flooded with tap water,
3. vacuumed in 27 ± 2 in. of mercury for 1 hour, and
4. put under 90 ± 10 psi for 2 hours.

The procedure was developed by the American Plywood Association and designated as **APA Test Method P-1** for linear expansion (LE) evaluation. Linear expansion and thickness swell (TS) values are based on the change from the oven-dry condition to the end of the ODVPS cycle.

Results and Discussion

Resin Properties

Average physical properties of co-reacted soy hydrolyzate/PF resin adhesives are summarized in Table 1.

On average, the resin properties, with exception of gel time, showed little difference among the 10 resins prepared in the study. Solid content ranged from 41.4 percent to 44.6 percent, and pH ranged from 12.61 to 12.89. The results suggest that the soy flour hydrolyzates prepared in presence of various amounts of phenol at two caustic content levels had no significant effect on resin properties when the same proportion of formaldehyde, phenol, soy flour hydrolyzate, and sodium hydroxide content were maintained at the final co-reacted soy resins. In comparison with the property of the two PF resins included in the study as controls, the co-reacted soy resins had lower solid content, but higher resin pH.

Average resin viscosity ranged from 320 to 610 cps that were felt in line with the targeted viscosity of 300 to 450 cps in resin synthesis. In general, the viscosity was comparable with that of the PF resins.

The most interesting result in the study of resin properties was gel time. When soy flour was hydrolyzed in the presence of phenol, the gel times were consistently longer with low caustic hydrolysis than with high

Table Z.—Average physical and mechanical properties of flakeboard made with soy hydrolyzate/phenolic resin adhesives.

ResinID	P/S ratio ^a (w/w)	Density (pcf)	IB ----- (psi) -----	MOR	MOE (10 ³ psi)	LE ----- (%) -----	TS
Highcaustichydrolysis							
100 H	(4/4)	48.9	102.0	4,350	589	0.621	40.7
75 H	(3/4)	49.4	104.8	4,280	607	0.631	35.3
50 H	(2/4)	47.6	106.3	3,870	523	0.585	36.3
25 H	(1/4)	47.9	105.8	4,200	549	0.593	34.4
OH	(0/4)	47.0	92.8	4,035	582	0.633	43.2
Lowcaustichydrolysis							
100 H	(4/4)	47.9	94.8	4,395	618	0.602	40.0
75 H	(3/4)	48.2	101.5	4,515	607	0.586	36.0
50 H	(2/4)	48.2	95.8	4,630	590	0.604	36.2
25 H	(1/4)	48.8	110.3	4,015	609	0.535	34.4
OH	(0/4)	48.9	105.0	4,445	618	0.595	36.1
Phenolformaldehyderesin							
PF-A		47.3	85.0	3,515	601	0.531	30.1
PF-B		49.0	104.6	4,405	586	0.540	30.0

^aPhenol-to-soy flour ratio in hydrolysis reaction.

caustic hydrolysis. When soy flour was hydrolyzed without phenol, however, the gel times were longer with high caustic hydrolysis than that of low caustic hydrolysis (Fig. 1). It was noted that the gel time of co-reacted soy resins ranging from 35.3 to 56.3 minutes were substantially longer than the gel time range of 17.2 to 22.1 minutes of PF resins (Table 1).

Resin Performance

Average physical and mechanical properties of flakeboards are summarized in Table 2. Effects of hydrolysis reaction conditions on bond performance of co-reacted soy hydrolyzates resin were evaluated by analysis of variance at the 0.05 level of probability. The performance comparisons between co-reacted soy hydrolyzate resins and phenolic resins were evaluated using the least significant difference.

Mechanical Strength

Average internal bond (IB) ranged from 93 to 110 psi, MOR ranged from 3,870 to 4,630 psi, and MOE ranged from 523,000 to 618,000 psi for the 10 co-reacted soy resins. The analysis of variance (ANOVA) for IB, MOR, and MOE is summarized in Table 3. MOR and MOE differed significantly between low- and high-caustic-to-soy flour (C/S) ratios, but did not differ statistically among various phenol-to-soy flour (P/S) ratios. The effects of either C/S ratio or P/S ratio on IB were not significant.

The significant effects of C/S ratios on MOR and MOE are summarized in Table 4. Low-caustic-soy hydrolyzates resulted in panels with significantly higher

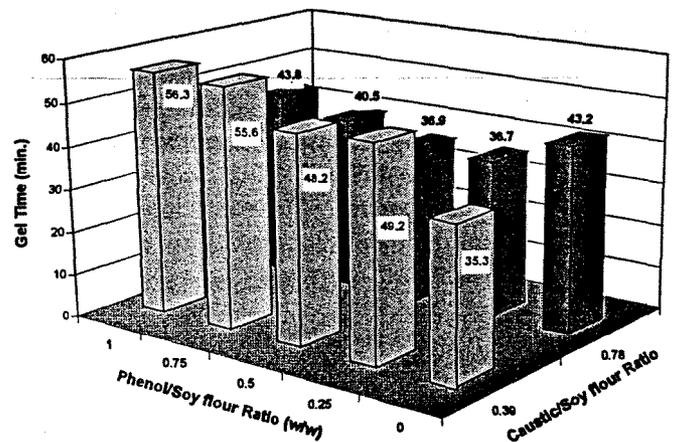


Figure 1.—Effect of phenol/soy flour and caustic soy flour ratio in the soy flour hydrolyzate on gel time.

average MOR and MOE as compared to those made with high-caustic-soy hydrolyzates.

In comparing the mechanical properties of co-reacted soy hydrolyzate resins and the phenolic resins used as controls, the results of the least significant difference analysis are summarized in Table 5. The performances of IB, MOR, and MOE results are discussed as follows:

IB Strength.—All co-reacted soy hydrolyzate resins yielded IB significantly higher than PF-A while most of the co-reacted resins had no significant differences in IB as compared with PF-B, indicating that the IB of co-reacted resins was highly comparable with the two phe-

Table 5.—Analysis of the mechanical strength properties of flakeboard panels made with various co-reacted phenol/soy flour hydrolyzate adhesives.

IB			MOR			MOE		
T grouping ^a	Mean	Resin ^b	T grouping ^a	Mean	Resin ^b	T grouping ^a	Mean	Resin ^b
A	110.3	25L	A	4,630	SOL	A	618,500	OL
A B	106.3	50H	A B	4,515	75L	A	618,000	100L
A B	105.8	25H	A B C	4,445	OL	A B	609,500	25L
A B	105.0	OL	A B C	4,405	PF-B	A B	607,000	75H
A B	104.8	75H	A B C	4,395	100L	A B	606,500	75L
A B	104.6	PF-B	A B C D	4,350	100H	A B	600,500	PF-A
A B	102.0	100H	A B C D	4,280	75H	A B	590,000	50L
A B	101.5	75L	A B C D	4,200	25H	A B	589,000	1 00H
C B	95.8	50L	B C D	4,035	0 H	A B	585,500	PF-B
C B	94.8	100L	C D	4,015	25L	C B	582,000	OH
C B	92.8	OH	E D	3,870	50H	C D	549,000	25H
	85.0	PF-A		3,515	PF-A	D	522,500	50H

^a A indicates the best property among 12 resins adhesives. The same letter in the column indicates no significant difference at the 5% level.
^b The number represents phenol to soy flour ratio in percent. L = co-reacted resin made with low caustic hydrolyzate; H = co-reacted resin made with high caustic hydrolyzate; PF-A and PF-B = commercial phenol formaldehyde resin A and B, respectively.

one co-reacted soy resin even yielded slightly lower LE, when compared to PF-B. However, all co-reacted resins were also shown to have higher TS than those of PF resins-used as controls. Thus, unlike its strength properties, the co-reacted resins seemed to perform less than expected in dimensional stability. It is likely that the high caustic content needed in hydrolyzing soy flour to maintain workable resin viscosity or the inherent limitation of soy protein toward water resistance might have contributed to the low dimensional stability.

The most interesting result in the study was the overall high performance of co-reacted resin 25L. It yielded the highest IB, attained one of the best dimensional stability among all the co-reacted resins, and ranked high in MOE strength. Although it was low in bending strength, improvement of MOR is anticipated when the hydrolysis process is optimized. A study based on this low caustic hydrolysis together with process modification in resin synthesis is in progress.

All co-reacted soy resins were synthesized with 30 percent substitution of phenol with the soy hydrolyzates. Considering the renewable aspects of the soy flour as a substitute for phenol and the low cost of soy flour (e.g., \$0.12/lb.) as compared to phenol (e.g., \$0.40/lb.), the most expensive constituent of conventional PF resin adhesives, economic gains by using the co-reactive soy resin system could be substantial. With a 30 percent phenol substitution with soy flour hydrolyzate, a co-reacted soy hydrolyzate-resin system as developed in this study could result in more than a 20 percent saving in material cost as compared to a conventional 2/1 ratio

Table 6.—Analysis of dimensional stability properties of flakeboard made with various co-reacted phenol/soy flour hydrolyzate adhesives.

LE			TS		
T grouping	Mean	Resin ^b	T grouping ^a	Mean	Resin ^b
C	0.633	OH	B	43.2	OH
B C	0.631	75H	A 0	40.7	100H
A B C	0.621	100H	A B	40.0	100L
A B C	0.604	SOL	A 0	36.3	50H
A-B C	0.602	100L	A B	36.2	50L
A B C	0.595	OL	A B	36.1	OL
A B C	0.593	25H	A B	36.0	75L
A B C	0.586	75L	A B	35.3	75H
A B C	0.585	50H	A B	34.5	25L
A B C	0.540	PF-B	A B	34.4	25H
A B	0.535	25L	A	30.1	PF-A
A	0.531	PF-A	A	30.0	PF-B

^a A indicates the best property among 12 resins adhesives. The same letter in the column indicates no significant difference at the 5% level.
^b The number represents phenol to soy flour ratio in percent. L = co-reacted resin made with low caustic hydrolyzate; H = co-reacted resin made with high caustic hydrolyzate; PF-A and PF-B = commercial phenol formaldehyde resin A and B, respectively.

(F/P) phenolic resin system used for bonding structural flakeboards.

Conclusion

A co-reacted phenol/soy flour hydrolyzate resin system has been developed for bonding exterior grade flakeboards. With a 30 percent soy flour hydrolyzate substitution for phenol, flakeboard panels made with

Table 3.—Statistical analysis of mechanical strength data.

Source	DF	F-value	P-value
IB			
C/S ratio	1	0.05	0.8337
P/S ratio	4	1.59	0.2505
C/S*P/S ratio	4	2.10	0.1552
Bending MOR			
C/S ratio	1	5.42	0.0422
P/S ratio	4	0.92	0.4890
C/S *P/S ratio	4	2.19	0.1438
Bending MOE			
C/S ratio	1	15.91	0.0026
P/S ratio	4	0.90	0.4970
C/S*P/S ratio	4	2.64	0.0974

nolic resins used as control. It is interesting to note that the IBs of panels bonded with co-reacted resins containing soy hydrolyzates that were made in the presence of phenol at high caustic (i.e., 100H, 75H, 50H, and 25H) were significantly higher than those bonded with co-reacted resins where the hydrolyzate was made without the presence of phenol (i.e., OH). On the contrary, the IB's of panels made with resins containing low caustic hydrolyzates which were made without the presence of phenol (i.e., OL) or with low phenol content (i.e., 25L) were higher than those made with hydrolyzates containing high phenol content (i.e., 100L, 75L, and 50L). These results seemed to suggest that

1. acceptable co-reacted resins are obtained by the use of high-caustic-hydrolyzed soy flour in the presence of phenol and
2. the best IB strength is achieved by the use of low-caustic-hydrolyzed soy flour in the presence of either low phenol content or no phenol at all.

Because IB is generally considered to be closely related to the general bond performance of resin adhesive, the significantly higher IB of co-reacted resin 25L among all of the resins in the study is most interesting. Not only was its IB 5.2 percent and 22.9 percent higher than that of PF-B and PF-A, respectively, its ability to hydrolyze soy flour at low caustic content while maintaining a workable viscosity provides the needed flexibility desired for further refining the hydrolyzation reaction.

Bending Strength (MOR).—The bending strengths of co-reacted soy resins were shown to be highly comparable to those of PF-A but not with PF-B. Table 5 shows that all co-reacted soy resins formulated from high caustic soy flour hydrolysis resulted in significantly lower MOR, while low caustic soy flour hydrolysis with

Table 4.—Effect of caustic to soy flour ratio in soy flour hydrolyzed on MOE and MOR of flakeboards.

Caustic/soy flour ratio	MOR	MOE*10 ³
	----- (psi) -----	
0.78	4,147	570
0.39	4,400	619

the exception of 25L yielded higher MOR, indicating the importance of caustic content level in soy flour hydrolysis as related to bending strength. During the solids content determination of the hydrolyzates, it was observed that the hydrolyzate prepared with high caustic content absorbed moisture from the air after removing it from the oven which slowly turned it into a gummy substance, while the hydrolyzate from low caustic content hydrolysis turned to a moist, soft substance. These effects of moisture on the hydrolyzate could have weakened the curing properties of the co-reacted resin and, in turn, affected the bending strength properties. It seems likely that dynamic mechanical analysis of cured co-reacted soy resin samples could lead to an understanding of the moisture effect on the co-reacted soy resin system. A study on the effects of caustic content levels on dynamic viscoelastic properties of co-reacted soy resin film is in progress.

Stiffness (MOE).—Table..! shows that MOE of the co-reacted soy resins were comparable with the phenolic resins used as controls. As with MOR, the co-reacted soy resins made from hydrolyzates prepared at low caustic content resulted in higher MOE; while high caustic hydrolyzates, with the exception of 75H, yielded lower MOE. It is postulated that the effect of caustic content level on moisture absorption by the soy hydrolyzate could reduce MOE as did MOR.

Dimensional Stability

Average linear expansion (LE) of the co-reacted soy hydrolyzate resins ranged from 0.535 to 0.633 percent and thickness swell (TS) ranged from 34.4 to 43.2 percent. All co-reacted soy resins showed higher LE and TS than the PF resins used as controls. The only exception was co-reacted soy resin 25L with its LE value falling in between PF-A and PF-B.

The analysis of the dimensional stability using the least significant difference is summarized in Table 5. All co-reacted soy resins yielded significantly higher LE than that of PF-A even though the PF-A performed poorly in bond strength. The LE increased 0.75 percent for co-reacted resin 25L to as much as 19.2 percent for resin OH than that of PF-A. Although the LEs of co-reacted resins seemed to be more comparable, only two co-reacted resins resulted in significantly higher LE and

the new co-reacted soy resin exceeded the **IB** strength of conventional PF resin and apparently resulted in savings of more than 20 percent in material cost. With **ad**-ditional studies of the dynamic viscoelastic properties of the co-reacted resin systems to evaluate the interactions of caustic content level and moisture absorption, the possibility for developing a co-reacted soy resin with improved dimensional stability is anticipated. Co-reacted resins based on use of a renewable raw material should play an important part in the development of new lower cost structural panel adhesives.

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