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A New Soy Flour-Based Adhesive for Making Interior Type II Plywood

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Abstract In this study, we developed a formaldehyde-free adhesive from abundant, renewable, and inexpensive soy flour (SF). The main ingredients of this adhesive included SF, polyethylenimine (PEI), and maleic anhydride (MA). The optimum formulation of this adhesive and the optimum hot-press conditions for making plywood were investigated. A three-cycle soak test and a boiling water test (BWT) were employed for evaluating the strength and water-resistance of plywood bonded with this adhesive. Results showed that SF, PEI, MA and sodium hydroxide were all essential components for the adhesive and the SF/PEI/MA weight ratio of 7/1.0/0.32 resulted in the highest water-resistance. When the hot-press temperature was in the range of 140–170 °C, both water-resistance and shear strength of plywood bonded with the adhesive remained statistically the same, except that the dry shear strength of plywood at 170 °C was statistically lower than that at 160 °C. When the hot-press time ranged from 2 to 6 min, the plywood panels at 5 min had the highest boiling water test/wet (BWT/w) shear strength. The plywood panels made at 5 min had a higher dry shear strength than those made at 3 min. Plywood panels bonded with this SF/PEI/MA adhesive exceeded the requirements for interior applications.

Keywords Wood adhesives · Soy flour · Polyethyleneimine · Interior plywood · Water-resistance

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Introduction

In 2001, the worldwide wood adhesive consumption was 13.3 million tons and total sale value reached $6.1 billion [1]. Formaldehyde-based adhesive accounted for 99% of the total volume and 97% of the total sale value, which indicated that formaldehyde-based adhesives played a dominant role in the wood adhesive market [1]. However, formaldehyde-based adhesives are derived from non-renewable petrochemicals and natural gas. In addition, the emission of formaldehyde, especially from the breakdown of UF resins in wood composites, poses a great hazard to human health because formaldehyde is a human carcinogen [2]. Finite oil reserves, the expanding wood adhesive market and hazardous issues associated with formaldehyde-based adhesives generate an urgent need for the development of environmentally friendly alternative wood adhesives from renewable materials.

Soybean, originated from eastern Asia, is one of the most important crops in the US. Soybean oil and soybean meal are two major products of soybean. At present, soybean meal is mainly used as animal feed. In the US, most soybean meal is consumed by livestock and poultry. Only a small portion of soybean meal is currently used in non-food industrial applications such as surfactants, inks, fuels and lubricants. The worldwide production of soybean has been increasing in recent years. The US farmers produced about 3.19 billion bushels of soybean in 2006, which reached the highest record of soybean production in the US [3]. The traditional soybean market as food and animal feed has been saturated. New non-food industrial applications have to be developed for consuming the oversupplied soybean.

Wood adhesives are potentially a huge market for the oversupplied soybean. As a matter of fact, soy-based adhesive was widely used in the commercial production of

plywood in the 1930s–1960s [4]. Soy-based wood adhesives have many advantages such as low cost, easy handling, low pressing temperature [4]. However, they also have many inferior properties such as low strength and low water-resistance of the resulting wood composite panels, which led to their replacement by formaldehyde-based adhesives. However, soybean represents an ideal raw material for making wood adhesives because it is abundant, renewable, environmentally friendly, and readily available.

There has been renewed interest in the development of soy-based wood adhesives in recent years. Various new methods have been investigated for improving the strength and water-resistance of wood composite panels bonded with soy-based adhesives. It was demonstrated that the treatment of soybean protein with alkali and protease enzymes significantly improved the strength and water-resistance of plywood samples bonded with the modified soy proteins [5, 6]. The treatments of soy protein with urea, guanidine hydrochloride, and sodium dodecyl sulfate (SDS) were also found to improve the strength and water-resistance of the resulting plywood panels [7, 8]. Chemical modifications of soybean protein using mussel adhesive protein as a model have been demonstrated to be effective ways of converting soybean protein to a strong and water-resistant wood adhesive [9, 10]. A polyamidoamine-epichlorohydrin (PAE) resin has been found to be an excellent curing agent for soybean protein [11]. A patented technology based on soybean flour and the PAE resin has been successfully used in the commercial production of plywood and particleboard [11–13].

A novel adhesive based on soy protein isolate (SPI), maleic anhydride (MA), and polyethyleneimine (PEI) has been developed [14]. However, this adhesive is not practical for commercial application because SPI is too expensive to be used as a raw material for making wood adhesives. In addition, the long reaction time and elevated temperature in modifying SPI and the time-consuming process of drying modified SPI contribute to other drawbacks of the SPI-MA-PEI adhesive. In this study, we investigated if soy flour (SF), an abundant and inexpensive form of soybean protein product, can be used to replace SPI for bonding wood. We developed a practical preparation procedure for the SF-based adhesive.

Experimental Procedures

Materials

SF (7% moisture content) was provided by Cargill Incorporated (Minneapolis, MN, USA); a 50 wt% aqueous PEI solution (Mw = 750,000) and MA were purchased from Sigma-Aldrich (Milwaukee, WI, USA). Yellow-poplar veneer was a gift from Columbia Forest Products (Portland, OR, USA).

Preparation of SF-MA-PEI Adhesives

The following is a representative procedure for the preparation of SF-PEI-MA adhesives. A 50% PEI solution (228 g wet PEI, 114 g dry PEI), de-ionized water (1,517 mL), MA (36.5 g), and 1 N NaOH (285 mL) were sequentially added to a KitchenAid mixer and mixed for 5 min at room temperature. SF (859 g, wet weight) was added and further mixed for 10 min. The total solids content of the resulting adhesive was 36%.

Preparation of Three-ply Plywood Samples

The adhesive was applied to two sides of a yellow-poplar veneer (2 ft × 2 ft; moisture content, 12%) by a roller coater. The spread rate of the adhesive was 8 mg/cm^2 on a dry weight basis. The adhesive-coated veneer was stacked between two uncoated veneers with the grain directions of two adjacent veneers perpendicular to each other. The stacked veneers were put on a table at ambient environment for 5 min, cold-pressed at 100 psi and room temperature for 5 min, put on a table at ambient environment again for 5 min and hot-pressed at 150 psi with predetermined time and temperature. After hot-pressing, the panel was stored at ambient environment for at least 24 h before it was evaluated for its shear strength and water-resistance.

Water Resistance of the Plywood Samples

The water-resistance of interior plywood panels was determined with a three-cycle soak test in accord with the American National Standard for Hardwood and Decorative Plywood; Hardwood Plywood & Veneer Association; 2004 (ANSI/HPVA HP-1). Twenty plywood specimens (2 in. × 5 in.) cut from each plywood panel were soaked in water at 24 ± 3 °C for 4 h, and then dried at 49–52 °C for 19 h. All specimens were inspected to see whether they were delaminated after the first cycle and the third cycle if applicable. This soaking/drying cycle was repeated until three cycles were completed. According to the standard, a plywood panel meets water-resistance requirements for interior applications if 95% of the specimens, i.e., 19 out of the 20 specimens do not delaminate after the first soaking/drying cycle and 85% of specimens, i.e., 17 out of 20 specimens do not delaminate after the third soaking/drying cycle. The ANSI/HPVA HP-1 specifically provides the following definition of delamination: any continuous

opening between two layers has to be longer than 2 in. and deeper than 0.25 in. and wider than 0.003 in. A boiling water test (BWT) was performed in accordance with US Voluntary Product Standard PS 1-95 for Construction and Industrial Plywood (published by the US Department of Commerce through APA-The Engineered Wood Composites, Tacoma, WA, USA). At least nine specimens (3.25 in. × 1 in.) were boiled in water for 4 h, dried at 63 ± 3 °C for 20 h, boiled again for 4 h, cooled down with tap water and then evaluated for shear strength while the specimens were still wet. The shear strength determined by this way was called boiling water test/wet (BWT/w). The shear strength was measured with an Instron testing machine. The crosshead speed was 1.0 mm/min.

Statistical Analysis of Strength Data

Strength data were analyzed with a two-sample *t*-test using S-PLUS® statistical software (Edition version 7.0, Insightful Corp., Seattle, WA, USA). All comparisons were based on a 95% confidence level.

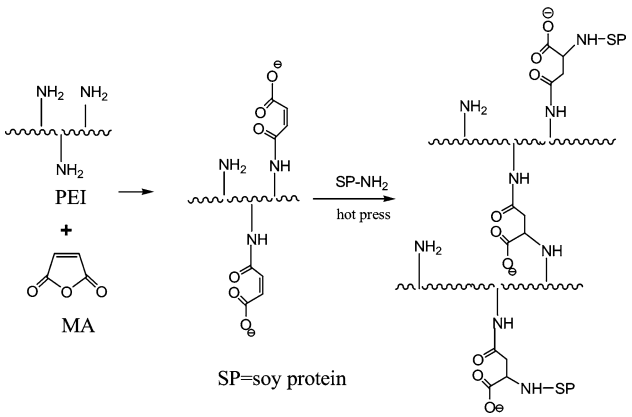
Results and Discussion

In the previous study on SPI-MA-PEI adhesives [14], SPI was first modified with MA at 50 °C for 120 min while maintaining a pH value of the reaction mixture at 10, and then mixed with PEI to form the adhesives. We replaced SPI with SF and tried the same preparation procedure described in the literature. We soon found that it was too tedious to make a sufficient quantity of MA-modified SF for making 2′ × 2′ plywood panels because the modification of SPI with MA was carried out in a dilute solution. We then explored different methods of modifying SF with MA. After numerous trials, we found that mixing PEI, MA and NaOH for 5 min before adding SF to the mixture was the best procedure for making the adhesive in terms of simplicity of preparation and the strength and water-resistance of the resulting plywood panels.

In the previous publication, it was demonstrated that MA first reacted with hydroxyl groups and amino groups of SPI to form ester-linked maleyl groups and amide-linked maleyl groups on SPI. The reactions between those maleyl groups with amino groups of PEI formed highly cross-linked adhesive networks during the hot-press, thus curing the adhesives [14]. In this study, the preparation procedure for the SF-MA-PEI adhesives was different from the literature procedure for making SPI-MA-PEI adhesives. The curing mechanisms for the SF-MA-PEI adhesives are expected to be different from those for the SPI-MA-PEI adhesives. We proposed that MA first reacted with PEI to form amide-linked maleyl groups that further reacted with amino groups in SF and PEI during a hot-press of making plywood panels (Scheme 1). SF may contain some water-soluble carbohydrates that would reduce the water resistance of adhesive bonds. The PEI-MA adduct might coat or bundle water-soluble carbohydrates, thus minimizing their negative effects on the water resistance. Hydroxyl groups of the carbohydrates might also react with the PEI-MA adduct via Michael addition although hydroxyl groups are weaker nucleophiles than amino groups, thus further reducing the negative effects of water-soluble carbohydrates on the water resistance.

Effect of the adhesive components on the water-resistance of plywood bonded with the adhesives is shown in Table 1. All specimens from all five panels delaminated after the first soaking/drying cycle in the absence of PEI and NaOH, i.e., when the adhesive only consisted of SF and MA. Three panels passed the three-cycle soak test and two panels failed when MA and NaOH were absent, i.e., when the adhesive only consisted of SF and PEI. Therefore, the SF-PEI adhesive was better than the SF-MA adhesive by comparing their soak test results (Table 1). However, this SF-PEI adhesive generated inconsistent results. Four panels passed the three-cycle soak test and only one panel failed when NaOH was absent, i.e., when the adhesive consisted of SF, MA and PEI. This SF-MA-PEI adhesive appeared to be slightly better than the SF-PEI adhesive. When SF, PEI, MA and sodium hydroxide were all present, all panels passed the three-cycle soak test and almost no specimens delaminated.

From the proposed reaction mechanisms in Scheme 1, SF, PEI and MA are all essential components for the final adhesive networks, which is consistent with our results that all these components had to be present for the resulting plywood panels to have superior water resistance. It was poorly understood why NaOH also was an essential component. We speculate that NaOH facilitated all those



Scheme 1 Proposed curing reactions of SF/PEI/MA adhesives

Table 1 Effect of the adhesive components on the water-resistance of plywood bonded with the adhesive

Components				The number of specimens failed in the three-cycle soak test/total specimens		Pass (P) or fail (F) of the three-cycle soak test
SF	PEI	MA	NaOH	First cycle	Third cycle	
Y	N	Y	N	20/20	–	F
				20/20	–	F
				20/20	–	F
				20/20	–	F
				20/20	–	F
Y	Y	N	N	6/20	–	F
				0/20	0/20	P
				0/20	1/20	P
				7/20	–	F
				0/20	0/20	P
Y	Y	Y	N	0/20	1/20	P
				0/20	1/20	P
				0/20	1/20	P
				0/20	0/20	P
				0/20	4/20	F
Y	Y	Y	Y	0/20	0/20	P
				0/20	0/20	P
				0/20	1/20	P
				0/20	0/20	P
				0/20	0/20	P

SF/PEI/MA weight ratio, 7/1/0.4; hot-press conditions: 150 psi, 160 °C and 5 min; Y represents that the component was present in the adhesive and N represents that the component was not present in the adhesive. The sign "–" means that no test was taken. Each row in the table represents one independently made panel

reactions shown in Scheme 1 and helped unfold soy protein in SF so that amino groups buried inside the compact soy protein structure were made available for the curing reactions. It is also possible that NaOH swelled the wood, thus improving the penetration and adhesion of the SF-MA-PEI adhesives.

Effect of SF/PEI weight ratio on the water-resistance of plywood bonded with the adhesives is shown in Table 2. At the 5/1 SF/PEI weight ratio, four panels passed the three-cycle soak test. However, one panel failed because 2 of 20 specimens failed after the first soaking/drying cycle. Strictly speaking, plywood panels bonded with the adhesive at the 5/1 SF/PEI ratio failed to meet the water resistance requirements for interior application. However, only 3 of 20 specimens failed after the third soaking/drying cycle, which implied that this panel passed the third cycle test. It is still possible that the adhesive at this SF/PEI ratio can be used for production of interior plywood when a large number of plywood panels are made and evaluated. At the 7/1 SF/PEI weight ratio, all panels passed the three-cycle soak test and all test specimens showed little opening or crack on the gluelines. At the 9/1 SF/PEI weight ratio, two panels passed the three-cycle soak test and three failed. At the 11/1 SF/PEI weight ratio, three panels passed the three-cycle soak test, but two failed. These results indicated that plywood panels had inconsistent water resistance at the 9/1 and 11/1 SF/PEI weight ratios. All panels failed to pass the three-cycle soak test when the SF/PEI weight ratio was further increased to 13/1 (Table 2).

According to the proposed reactions (Scheme 1), the amount of PEI has to be high enough (the results in Table 2 reveal that the SF/PEI weight ratio has to be lower than 9/1). Otherwise, the reactive sites (the amide-linked maleyl groups) for crosslinking may not be sufficient for forming water-resistant adhesive networks. The amino groups in PEI can also form salts with carboxylic acid group in soy protein and the salts may loss water to form amide linkages during the hot-press. These may explain why the water-resistance of the resulting plywood panels failed to pass the three-cycle soak test when the SF/PEI weight ratio was equal to and higher than 9/1.

Effect of the MA dosage on the water-resistance of plywood bonded with the adhesives is shown in Table 3. At the 40 wt% MA, both plywood panels passed the three-cycle soak test, but 4 out of 12 specimens delaminated after a BWT. At the 32 wt% MA, both panels passed the three-cycle soak test and no specimens delaminated after a BWT. At the 24 wt% MA, both panels passed the three-cycle soak test, but one out of nine specimens delaminated after a BWT. At the 16 wt% MA or 8 wt% MA, one panel passed the three-cycle soak test and one failed. However, a high percentage of specimens delaminated at these two dosages

Table 2 Effect of SF/PEI weight ratio on the water-resistance of plywood bonded with the adhesives

Weight ratio (SF/PEI)	The number of specimens failed in the three-cycle soak test/total specimens		Pass (P) or fail (F) of the three-cycle soak test
	First cycle	Third cycle	
5/1	2/20	3/20	F
	0/20	0/20	P
	1/20	1/20	P
	0/20	0/20	P
	0/20	0/20	P
7/1	0/20	0/20	P
	0/20	0/20	P
	0/20	1/20	P
	0/20	0/20	P
	0/20	0/20	P
9/1	0/20	0/20	P
	2/20	5/20	F
	0/20	0/20	P
	0/20	5/20	F
	6/20	–	F
11/1	1/20	4/20	F
	0/20	0/20	P
	0/20	0/20	P
	0/20	2/20	P
	3/20	–	F
13/1	2/20	5/20	F
	0/20	5/20	F
	0/20	4/20	F
	4/20	–	F
	0/20	4/20	F

PEI/MA weight ratio, 1/0.4; 1 N NaOH solution, 285 mL, hot-press conditions: 150 psi, 160 °C and 5 min. The sign "–" means that no test was taken. Each row in the table represents one independently made panel

after a BWT. In summary, all plywood panels passed the three-cycle soak test at the 24–40 wt% MA, and all test specimens had little opening or crack on the gluelines, which means that the adhesive at this range of MA usage can be used to make interior plywood. Plywood panels bonded with the adhesive at the 32 wt% MA had the highest water resistance. Reduction of the MA usage to 16 or 8 wt% significantly decreased the water-resistance of

Table 3 Effect of the MA dosage on the water-resistance of plywood bonded with the adhesives

Wt % MA on PEI (dry basis)	The number of specimens failed in the three-cycle soak test/total specimens		Pass (P) or fail (F) of the three-cycle soak test	The number of specimens delaminated after a BWT/total specimens
	First cycle	Third cycle		
40	0/20	0/20	P	4/12
	0/20	0/20	P	4/12
32	0/20	1/20	P	0/9
	0/20	0/20	P	0/9
24	0/20	0/20	P	1/9
	0/20	0/20	P	1/9
16	2/20	5/20	F	–
	0/20	0/20	P	8/9
8	8/20	–	F	–
	0/20	0/20	P	3/9

SF/PEI weight ratio, 7/1; 1 N NaOH solution, 285 mL; hot-press conditions: 150 psi, 160 °C and 5 min. The sign "–" means that no test was taken. Each row in the table represents one independently made panel

Table 4 Effect of hot-press temperature on the water-resistance of plywood bonded with the adhesives

Hot-press temperature (°C)	The number of specimens failed in the three-cycle soak test/total specimens		Pass (P) or fail (F) of the three-cycle soak test	The number of specimens delaminated after a BWT/total specimens
	First cycle	Third cycle		
110	14/20	–	F	–
	12/20	–	F	–
120	19/20	–	F	–
	0/20	0/20	P	–
130	1/20	1/20	P	–
	3/20	–	F	–
140	1/20	3/20	P	0/9
	0/20	0/20	P	0/9
150	0/20	0/20	P	0/9
	0/20	0/20	P	0/9
160	0/20	1/20	P	0/9
	0/20	0/20	P	0/9
170	0/20	1/20	P	0/9
	0/20	0/20	P	0/9

SF/PEI/MA weight ratio, 7/1/0.32; 1 N NaOH solution; 285 mL; hot-press conditions: 150 psi, 160 °C and 5 min. The sign "–" means that no test was taken. Each row in the table represents one independently made panel

the resulting plywood panels. At the 8–16 wt% MA, the resulting plywood panels could not meet the water resistance requirement for interior applications.

MA is susceptible for hydrolysis to form maleic acid and is very water-soluble. A high amount of maleic acid in the final adhesive would definitely reduce the water-resistance of the adhesives. This is why the plywood panels at 40 wt% MA had more specimens delaminated after a BWT than those at 32 wt% MA (Table 3). However, the amount of MA has to be sufficiently high (the results on Table 3 reveal that the weight percentage of MA based on PEI has to be higher than 16%). Otherwise, the amount of amide-linked maleyl groups is not sufficient to form highly crosslinked water-resistant adhesive networks. This explanation is consistent with the results that some plywood panels even could not pass the three-cycle soak test when the amount of MA was equal to and lower than 16 wt%.

Effect of hot-press temperature on the water-resistance and shear strength of plywood panels is shown in Table 4 and Fig. 1, respectively. At 110 °C, both panels failed after the first soaking/drying cycle. At 120 and 130 °C, the resulting plywood panels had inconsistent water resistance: one panel passed the three-cycle soak test and one failed. When the hot-press temperature was at 140–170 °C, all panels passed the three-cycle soak test and no specimens delaminated after a BWT. Therefore, the hot-press temperature has to be higher than 130 °C for the resulting plywood panels to meet the water resistance requirements for interior applications.

According to the ANSI/HPVA HP-1 standard, there are no shear strength requirements for interior type II plywood, i.e., the most commonly used decorative plywood for making furniture, hardwood floor, and kitchen cabinet. We measured dry shear strength and BWT/w shear strength to investigate whether a different hot-press temperature in the range of 140–170 °C would make subtle differences on the strength properties of the resulting plywood panels. When the hot-press temperature was increased from 140 to 160 °C, the dry shear strength did not significantly change (Fig. 1). When the hot-press temperature was further increased from 160 to 170 °C, the dry shear strength significantly decreased ($p < 0.0005$) (Fig. 1). The BWT/w shear strength did not significantly change when the

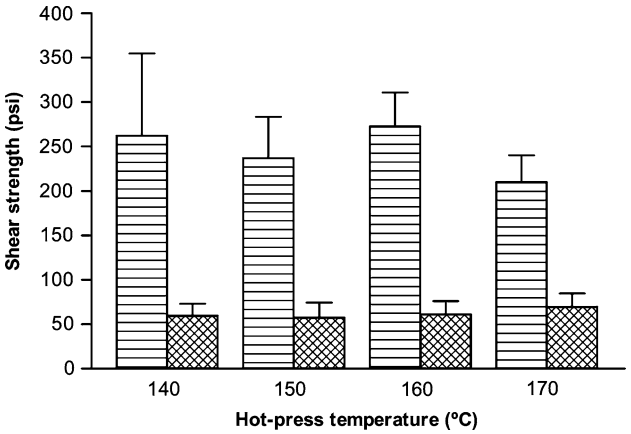


Fig. 1 Effect of hot-press temperature on dry (*horizontally lined bars*) and BWT/w (*crosshatched bars*) shear strength of plywood specimens bonded with the adhesives (SF/PEI/MA weight ratio, 7/1/0.32; 1 N NaOH solution: 285 mL hot-press conditions: 150 psi, 5 min. Data are the means of 18 replicates, and the *error bar* represents one standard deviation)

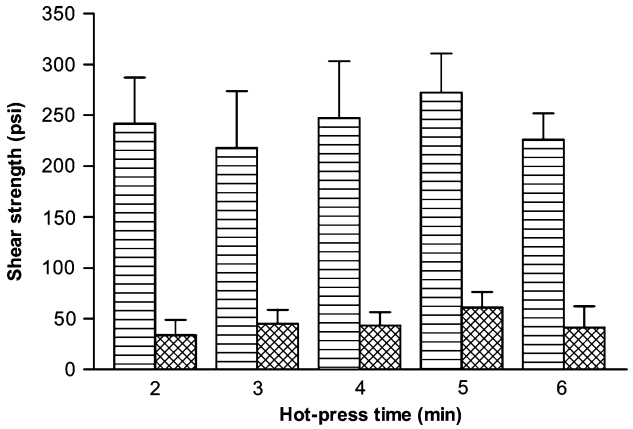


Fig. 2 Effect of hot-press time on dry (*horizontally lined bars*) and BWT/w (*crosshatched bars*) shear strength of plywood specimens bonded with the adhesives (SF/PEI/MA weight ratio, 7/1/0.32; 1 N NaOH solution, 285 mL; hot-press conditions: 160 °C, 150 psi. Data are the means of at least 15 replicates, and the *error bar* represents one standard deviation)

hot-press temperature was increased from 140 to 170 °C (Fig. 1).

Effect of hot-press time on the water-resistance and shear strength of the resulting plywood is shown in Table 5 and Fig. 2, respectively. At 1 min of hot-press time, both panels failed after the first soaking/drying cycle. At 2 min, both panels passed the three-cycle soak test, but three of nine specimens from one panel delaminated after a BWT. At 3 min, both panels passed the three-cycle soak test, and only one of nine specimens from one panel delaminated after a BWT. At 4–6 min, not only all panels passed the three-cycle soak test, but also no specimen delaminated after a BWT. Therefore, when the hot-press time was longer than 2 min, the resulting plywood panels met the water resistance requirements for interior applications. The overall water resistance increased when the hot-press time increased.

When the hot-press time was in the range of 2–6 min, only the dry shear strength of the specimens at 5 min was significantly higher than that at 3 min ($p = 0.0051$) and other comparisons of dry shear strengths did not show any significant change (Fig. 2). The BWT/w shear strength of the specimens at 5 min was statistically higher than those at 2 min ($p < 0.0005$), 3 min ($p = 0.0037$), 4 min ($p = 0.0035$) and 6 min ($p = 0.01645$).

The optimum hot-press conditions (hot-press time and hot-press temperature) for SF-MA-PEI adhesives are highly dependent upon the number of plywood layers and the thickness of the plywood panels. For the three-ply panels in this study, it appeared that the adhesives were not fully cured at a hot-press temperature of lower than 130 °C for 5 min. At 160 °C hot-press temperature, the hot-press time had to be longer than 2 min for the three-ply panels.

In this SF/PEI/MA adhesive, PEI is still too expensive to allow the immediate commercial application of this adhesive for making interior plywood. In addition, PEI is currently made from petrochemicals. Development of a polyamine from renewable materials for replacing PEI is currently ongoing in our lab. Despite the negative aspects of PEI, results from this study show that a SF/MA/polyamine adhesive has the potential of being commercially used for making interior plywood. Such an incremental advancement of soy-based adhesive technology enriches our knowledge base and may eventually lead to development of a commercially viable, formaldehyde-free SF-based adhesive.

Table 5 Effect of hot-press time on the water-resistance of plywood bonded with the adhesives

Hot-press time (min)	The number of specimens failed in the three-cycle soak test/total specimens		Pass (P) or fail (F) of the three-cycle soak test	The number of specimens delaminated after a BWT/total specimens
	First cycle	Third cycle		
1	7/20	–	F	–
	3/20	–	F	–
2	0/20	0/20	P	0/9
	0/20	2/20	P	3/9
3	0/20	0/20	P	0/9
	0/20	0/20	P	1/9
4	0/20	0/20	P	0/9
	0/20	0/20	P	0/9
5	0/20	1/20	P	0/9
	0/20	0/20	P	0/9
6	0/20	0/20	P	0/9
	0/20	0/20	P	0/9

SF/PEI/MA weight ratio, 7/1/0.32; 1 N NaOH solution, 285 mL; hot-press conditions: 150 psi, 160 °C. The sign "–" means that no test was taken. Each row in the table represents one independently made panel

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