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Comparison of the Adhesive Performances of Soy Meal, Water Washed Meal Fractions, and Protein Isolates

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Abstract

Adhesive bonding of wood plays an increasing role in the forest products industry and is a key factor for efficiently utilizing timber and other lignocellulosic resources. In this work, we obtained five soy meal products through commercial sources or in-house preparations. The protein content was 49.6%, 56.9%, 66.2%, 86.3%, and 91.9% for untreated defatted soy meal, pH 8.5 water washed meal, neutral water washed meal, commercial protein isolate, and in-house prepared protein isolate. The adhesive performances measured by the maximal dry and soaked shear strength of the bonded maple veneers at break were not exactly in the same order of the protein content, indicating that other components (e.g. carbohydrates, metals) might also have played certain roles in the adhesive ability of these products. Data at two press temperatures (i. e. 100, and 130 ^{o}C) with or without the addition of tung oil revealed that water washed soy meals behaved more like untreated meal than soy protein isolates. This observation is different from a recent report on the effect of water washing on cottonseed meal products. Thus, further elucidation of the mechanisms or causes of the differing effects of water washing would shed light on the adhesive mechanisms of the two types of oilseed meal materials, thus optimizing use of these materials and their fractions for wood bonding.

Keywords: protein isolate, soy meal, water resistance, wood adhesive

1. Introduction

Adhesive bonding of wood plays an increasing role in the forest products industry and is a key factor for efficiently utilizing timber and other lignocellulosic resources. As synthetic wood adhesives are mostly derived from depleting petrochemical resources and have caused increasing environmental concern, agricultural product and byproduct derived adhesives have attracted much attention in recent decades (Akaranta et al., 1996; Khan et al., 2004; Li et al., 2004; Wang et al., 2014). Among them, soy protein isolate (SPI) is the most studied for this purpose (Frihart et al., 2013; Liu & Li, 2007; Qi et al., 2012). SPI is obtained from defatted soy meal or flour, but is about 10 times more expensive than soy meal (Frihart et al., 2013). For this reason, soy meal and soy flour have also been tested as candidates for wood adhesive use (Frihart and Lorenz, 2013; Gao et al., 2011; Lin et al., 2012; Xu et al., 2014). For better and economical use of different soy products, Lorenz et al. (2015) compared the wood adhesive performance of a variety of commercial soy flours, protein concentrates and isolates. They reported that the carbohydrate interference is only part of the adhesive difference between commercial soy flour and purified soy proteins (isolate and concentrate). The protein denaturation in SPI could be an even larger factor. These observations indicate that the level (or purity) of protein in these products is not the only parameter in determining adhesive properties. As matter of fact, we (He et al., 2014c) also found, in seeking the enhanced utilization of defatted cottonseed meal, that higher protein content in cottonseed meal fractions did not necessarily lead to higher adhesive properties.

Working on the hypothesis that the removal of water soluble components would improve the adhesive properties of the defatted meal, especially water resistance, we (He et al., 2014a; 2014b; 2014c) developed a unique water washing procedure to produce water washed cottonseed meal and demonstrated that, as a wood adhesive, the insoluble cottonseed meal fraction (i. e. water washed meal) was good as cottonseed protein isolate. Further characterization of these products indicated that both water soluble proteins and carbohydrates were removed during water washing, the water insoluble fraction was less hydrophilic than the water soluble fraction (He et al., 2014d; 2015). Water washing is a less laborious and more environment-friendly process than protein isolation,

which involves multiple processing steps and caustic reagents. However, it is unknown if the water washing approach could improve the adhesive performances of other seed meals. Thus, in this work, we applied the same water washing process to produce water washed soy meal, and compared its adhesive performance with both unwashed soy meal and SPI. The purpose of this work was to 1) evaluate if the economic water washing way can be applied to improve the adhesive performances of soy meal products and 2) increase our general knowledge of the adhesive mechanisms of oilseed meal products by comparison of the data of the soy meal products with their cottonseed counterparts.

2. Materials and Methods

2.1 Soy Meal Products

The five soy products used in this study are listed in Table 1. Soy meal (SM) was obtained from Kentwood Co-op (Kentwood, LA, USA). SM was first ground by a cyclone sample mill (Model 3010-014, UDY Corporation, Fort Collins, CO, USA) to pass a 0.5-mm steel screen (He and Chapital, 2015). Water washed soy meal (SMw) was prepared from the ground SM after neutral (pH 7.0) water extraction to remove water soluble components in the meal as outlined in the procedure of He et al. (2014c). For this study, SM (25 g) was placed with distilled water (375 mL) in a blender (Model WF2211214, Waring Commercial, Torrington, CT, USA) and blended for 2 min at low speed, then 1 min at a high speed. The mixture was centrifuged at 3,980 x *g* for 30 min at 22 ^{o}C. The supernatant was discarded. The insoluble fraction was freeze-dried and named SMw.

Table 1. Abbreviations, treatments, and yields of soy meal products used in this study

Product	Abbreviation	Treatment	Yield (%)
Soy meal	SM	Purchased raw material, ground	100%
Water washed meal	SMw	Residue after neutral water washing/extraction	55%
Insoluble meal residue	SMr	Residue after alkaline water extraction (pH 8.5)	49%
Protein isolate	SPIe	Extraction (pH 8.5)/precipitation (pH 4.2)	16%
Commercial protein isolate	SPIc	Purchased, no treatment	Not applicable

Another batch of the ground soy meal was washed in the same manner, but the pH of the water-meal mixture was adjusted to pH 8.5 and blended as above. After centrifugation at 3,980 x *g* for 30 min at 22 ^{o}C, the precipitate and supernatant were separated. The precipitate was freeze-dried and identified as the insoluble soy meal residue (SMr). As a matter of fact, SPI reported in the literature was prepared from the soluble fraction of the pH 8.5 water washing/extraction (He et al., 2013; Huang and Sun, 2000). Thus, the supernatant after the centrifugation of the pH 8.5 water-meal mixture was acidified to pH 4.2 with 1 M HCl and stored at 4 ^{o}C for 12 h for protein to precipitate completely. The proteins isolate was obtained after centrifugation and freeze-drying. It was named SPIe. A commercially available SPI purchased from an on-line vendor (www.bulkfoods.com) was named SPIc.

All products were kept in a desiccator at room temperature (22 ^{o}C) until used. Selected chemical composition of SM, SMw and SPIe was reported previously (He et al., 2015) and the compositional analysis was done for SMr and SPIc in the same way (Table 2).

2.2 Preparation of Bonded Wood Specimens

Maple wood veneer (1.59 mm thick) was purchased from Certainly Wood, Inc. (East Aurora, NY, USA). Veneers were cut into strips 25.4 mm wide by 88.9 mm long, with the wood grain parallel to the long side, and stored in sealed plastic bags until ready for use.

Adhesive slurries were prepared by mixing a dessicated soy meal product with deionized water (3:25 w/w) for 2 h. If needed, tung oil (Real Milk Paint Co. Hohenwald, TN) was added and mixed for another 0.5 h (He et al., 2014b). Using a brush, each adhesive preparation was applied to one end of two wood veneer strips covering 25.4 mm (1.0") length. The wet adhesive was allowed to air-dry for 10-15 minutes or until tacky, then a second layer was applied on top of the first layer and air-dried again for the same time period. The tacky adhesive coated areas of the wood veneer strips were overlapped and bonded by hot-pressing (Carver Benchtop Heated Press, Model 3856, Carver Inc., Wabash, IN) at 100 ^{o}C (or specified temperature) for 20 min at a pressure of 2.8 MPa.

The bonded area between the two strips was 25.4 mm x 25.4 mm (1.0″ x 1.0″). The total amount of dry adhesive preparation applied was about 4.5 mg dry solid per cm^2 of bonding are of each wood strip. These bonded wood specimens were cooled and set aside for conditioning at normal room temperature (22-23 °C) and relative humidity (50-60%) for 4 days before further experiments were conducted unless stated otherwise (He and Chapital, 2015).

Table 2. Selected chemical properties of soy meal products (% of dry matter based)

	Protein	AA [1] (p/n)	Arabinose	Rhamnose	Galactose	Glucose	Xylose	Mannose	P	K	Na	Ca	Mg
	%		%	%	%	%	%	%	%	%	%	%	%
SM [2]	49.6	1.63	2.51	1.07	7.78	8.06	0.89	1.61	0.70	2.40	0.01	0.47	0.32
SMw	66.2	1.52	4.33	1.74	7.65	4.12	1.43	1.88	0.56	0.88	0.01	0.54	0.19
SMr	56.9	1.55	4.38	1.77	8.41	4.81	1.37	1.75	0.70	1.35	0.22	0.59	0.24
SPIe	91.9	1.48	0.36	0.17	0.70	0.67	0.04	0.56	0.96	0.28	0.05	0.05	0.05
SPIc	86.3	1.34	0.04	0.01	0.04	0.02	0.01	0.00	0.87	0.19	1.14	0.19	0.21

[1] Ratio of polar amino acids/non polar amino acids.

[2] Data of SM, SMw and SPIe taken from He et al. (2015).

2.3 Water Resistance Test

The test conditions were based on one of the water exposures in ASTM D1151-00 (ASTM, 2013). Specifically, after conditioning the bonded wood specimens were immersed in a water bath at 63 ºC for 4 h, then dried at room temperature (22-23 °C) overnight (18-20 h). The bonded specimens were returned to the 63°C bath for 4 h, removed and set aside for conditioning at room temperature and relative humidity (50-60%) for 2 days before shear strength measurement (Cheng et al., 2013; He et al., 2014a). These specimens are identified as the soaked specimens

2.4 Lap Shear Strength Measurements

The lap-shear strength of both dry and water soaked wood specimens were measured with a Zwick Materials Tester (Zwick GmbH & Co., Ulm, Germany) fitted with 32 mm x 40 mm fishscale gridded wedge grips, and operated with a crosshead speed of 1 mm min^{-1}. The shear strength at break (MPa) was reported as the adhesive strength of the tested soy meal products.

2.5 Statistical Analysis

At least five bonded wood specimens were tested for each treatment. The data analysis package in Microsoft Excel 2007 was used for statistical analysis. The Descriptive Statistics Tool Data was used to calculate averages and standard deviations (SD). Single-factor analysis of variance (ANOVA) was used to evaluate the significance levels of the effects of treatments on adhesive properties.

3. Results and Discussion

3.1 Chemical Composition of the Soy Meal Products

The yield of SMw, SMr and SPIe was 55%, 49%, and 16% of SM, respectively (Table 1). The protein content of commercial SM was 49.6% of dry matter with the ratio of 1.63 for polar amino acids and nonpolar amino acids (Table 2). All the soy meal products showed larger protein content and lower ratios of polar and nonpolar amino acids than SM itself. The lower protein content of SMr than SMw was apparently due to the fact that more protein solubilized at pH 8.5 than in neutral water during the washing. Recently, Jang and Li (Jang and Li, 2015) separated soy flour into soluble and insoluble fractions using water adjusted to pH 8.0. They named the insoluble fraction as insoluble carbohydrates (IC). They reported that IC contained about 20% proteins so that the remaining 80% were assumed to be carbohydrates. However, the protein content in either SMw or SMr in this work was higher than that in IC. The difference might be due to the difference in operation and/or raw materials. Interestingly, the results of the SMw and SMr are more similar to those of their corresponding counterparts of cottonseed meal (He et al., 2014c; 2015), implying that the operational difference could be the major factor.

Among the six carbohydrates measured, the content of arabinose, rhamnose and xylose increased by 54-74% in the two insoluble SMw and SMr fractions, compared to the corresponding carbohydrate content in SM. On the other hand, the glucose content in SMw and SMr were 49% and 40% lower than in SM, respectively. The

content of galactose and mannose did not change much in SMw and SMr. The carbohydrate contents were low in SPIe and SPIc as they were assumed to be purified protein fractions.

The content of phosphorus (P) was 0.7% in SM. Higher contents (0.96% and 0.87%) were observed in SPIe and SPIc as some P in phytate was associated with protein (Han, 1988). The contents of K, Na, Ca, and Mg in the four soy meal products were generally at the same levels of SM as these metal contents were low in soy meal and the reagents used in the preparative processes could also introduce minor amounts of these elements into the products.

3.2 Effect of Pressing Time on Adhesive Strength

The effect of pressing time on adhesive strength was tested using maple specimens bonded with SM and SPIc (Figure 1). As expected, the adhesive strength of SM was about 1.5-2.0 MPa lower than SPIc. The adhesive strength of SM gradually increased from 1.5 MPa (1 min press time) to 3.2 MPa (15 min). Further extension of the press time did not further increase SM's adhesive strength. SPIc reached to the stable adhesive strength at a shorter press time (5 min). From 5-20 min, the adhesive strength of SPIc basically fluctuated around 4.0 MPa. Thus, we adopted the press time of 15 min for general adhesive performance testing of all five soy meal products. Zhong et al. (2002) reported the sharp increase in shear strength of fiberboard glued with 1.0 M guanidine HCl-modified SPI within the first 5 min of the 15-min press time tested. After that time, the shear strength decreased, but not statistically significant. They also measured the effect of assembly time that was defined as the time the brushed fiberboard rested at room temperature prior to assembling. They found the optimal assembly time between 10-15 min for the maximum shear strength. We did not measure the effect of assembly time, but kept the assembly time the same for all products tested.

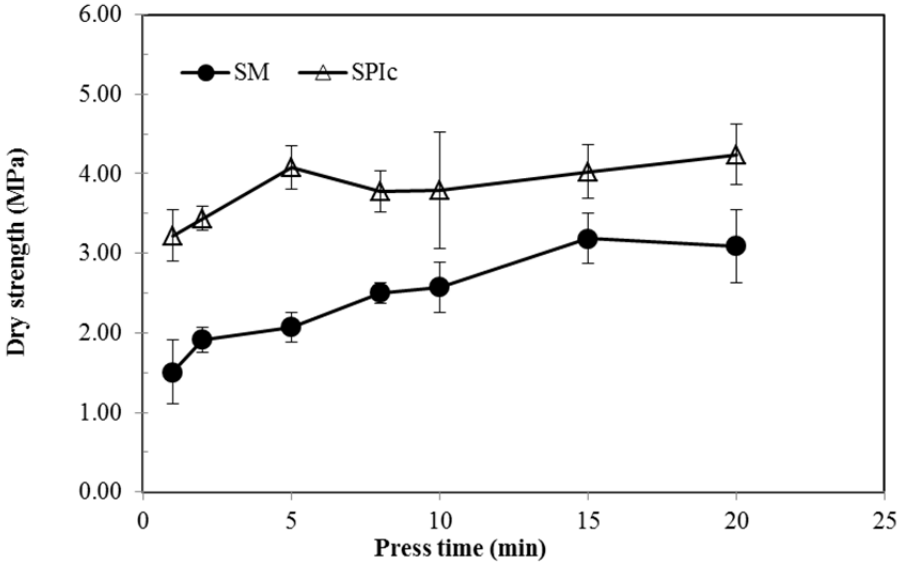

Figure 1. Effect of press time on the dry shear strength of maple veneer specimens bonded with commercial soy meal (SM) and protein isolate (SPIc) adhesives. Values are averages ± standard deviations (n=6)

3.3 Adhesive Strength of Soy Meal Products

Using a press temperature of 100 ^{o}C, all five products showed good dry adhesive strength ranging from 2.4 and 4.2 MPa (Table 3). The dry strength of SMw and SPIc glued at 100 ^{o}C was 2.40 ± 0.50 MPa and 4.16 ± 0.56 MPa, respectively. Those values were not statistically significantly different (p>0.05) from those (3.09 ± 0.46 MPa and 4.24 ± 0.38 MPa, respectively) in Figure1 obtained under the same bonding conditions with different batch of adhesive slurries prepared at another time. However, the difference between the meal products is obvious as the dry strength increased in the order of SM< SMr< SMw< SPIe< SPIc. After the water resistance test, the adhesive strength of SM, SMw, and SMr decreased 48, 53 and 72%, respectively; whereas the strength of SPIe and SPIc increased slightly. The order of the soaked shear strength was SMr <SM <SMw <SPIe <SPIc. Raising the press temperature to 130 ^{o}C produced little change in the dry adhesive strength of SM, SMw, SPIe and SPIc, but dramatically decreased the adhesive strength of SMr. On the other hand, the higher press temperature greatly improved the water resistance adhesive strength values of SM and SMw. However, unlike their counterparts of cottonseed products (He et al., 2014a; He et al., 2014c), the water resistance adhesive

strength values of SMw remained more similar to SM than to SPIe and SPIc strength values.

Table 3. Dry and soaked shear strength of maple veneer specimens bonded at 100 and 130 °C using soy meal products, and change of soaked shear strength compared to the corresponding dry strength. Values are averages ± standard deviations (n=5)

	Dry strength (MPa)	Soaked strength (MPa)	Change (%)
	----------100 °C----------		
SM	2.40 ± 0.50	1.25 ± 0.19	-47.9 *** [1]
SMw	3.22 ± 0.72	1.52 ± 0.39	-52.8 ***
SMr	2.87 ± 0.73	0.81 ± 0.51	-71.8 ***
SPIe	3.51 ± 0.95	3.73 ± 0.62	6.3 ns
SPIc	4.16 ± 0.56	4.24 ± 0.37	1.9 ns
	----------130 °C----------		
SM	2.85 ± 0.51	2.48 ± 0.29	-13.0 ns
SMw	3.25 ± 0.80	2.20 ± 0.29	-32.3 *
SMr	0.76 ± 0.15	0.57 ± 0.34	-25.0 ns
SPIe	3.73 ± 0.62	3.67 ± 1.28	1.6 ns
SPIc	4.50 ± 0.42	4.95 ± 0.34	10.0 ns

[1] Symbol *, ***, and ns represent the significant difference between dry and soaked strength of the adhesive at P=0.05, 0.001, and no significant difference at P=0.05, respectively

These adhesive strength results indicated that the residue fraction using pH 8.5 water extraction (SMr) was a poor choice for wood adhesive applications. Indeed, this observation was reasonable as, the SPIe fraction, which was precipitated out from the soluble fraction of SMr, showed good adhesive performance. Jang and Li (2015) also showed that their insoluble fraction of soy flour at pH 8.0 extraction had poor adhesive performance when compared to soy flour.

The poor adhesive performance in water resistance tests using SMw was in contrast to our assumption that water washing would improve the soaked strength of soy meal per our previous observation on cottonseed meal (He et al., 2014c). On the other hand, the result is similar to that of Lorentz et al. (2015). They reported that a soy protein concentrate did produce better wet adhesive strength than soy flour, in contrast to their assumption that removal of the soluble carbohydrates and low-molecular weight protein during preparation of the concentrate should have improved the wet adhesive bond strength. Thus, Lorenz et al. (2015) proposed that both insoluble and soluble carbohydrates could prevent good physical contact between the proteins for better adhesive performance of soy meal-based products. The hypothesis could partly explain the similar adhesive data of SM and SMw (Table 2).

Whereas the adhesive performance of both protein isolates (SPIe and SPIc) were better than soy meal, the dry and soaked strength of commercial SPIc was greater than SPIe (Table 3). Previously, Lorenz et al. (Lorenz et al., 2015) reported better adhesive performance of a commercial soy protein isolate than their lab-prepared protein isolates. As a matter of fact, Lorenz et al. (2015) tested seven commercial soy protein isolates, and found that all of them provided good wet adhesive strength ranging from 1.9 to 2.5 MPa despite viscosities ranging from 10 to 58,000 cps and pH values of 5.4 to 7.6 in 15 percent dispersions. Thus, Lorenz et al. (2015) guessed that the homogenization with heat and water converted the commercial SPI into a more functional protein, leading to greater adhesive strength, and concluding that purifying the protein by removing the carbohydrates and other components in flour to obtain a >95% protein content did make for a better adhesive. The procedure of lab-made SPIe in our study was the same as that of Lorenz et al. (2015). Our confirmative observation implied more research is needed for the adhesive mechanism involving the difference between the commercial protein isolate and the more purified lab counterpart. In reviewing the metal composition data (Table 2), higher Ca and Mg contents in SPIc may have contributed to better adhesive performance using SPIc than SPIe (Jang and Li, 2015; Liu et al., 2010; Scilingo and Anon, 2004).

3.4 Effect of Tung Oil on Adhesive Strength of Soy Meal Products

In general, the effect of adding 0.1% tung oil to the adhesive slurries either decreased or increased the adhesive strength of the five products (Table 4) when compared to the adhesive strength data of soy products without tung oil (Table 3). The only exception was SMr at the press temperature of 130 °C, where the addition of tung oil

increased the dry and soaked adhesive strength of SMr by142% and 75%, respectively. Even though, the adhesive performance of SMr with the press temperature at 130 °C was still the poorest among the 10 sets of data. Those data showed the effect of tung oil addition for soy products was not greater than for cottonseed meal products (He et al., 2014b). Thus, the results of this research with or without tung oil addition demonstrated that there were some differences in adhesive performance between soy meal-based and cottonseed meal-based products. Cheng et al. (2013) showed that cottonseed protein exhibits superior adhesive strength and water resistance relative to soy protein. In a more recent study, Cheng et al. (2016) blended soy and cottonseed protein with hemicellulose, cellulose, or starch and measured the adhesive performance. Their data showed that the cottonseed protein blends exhibited stronger adhesive strength and hot water resistance than the soy protein blends. They assumed that this difference is attributed to better adhesive performance of cottonseed protein since the level of arginine was higher in cottonseed protein (about 11-12%) than in soy protein (about 7-8%, per literature). However, there was not much difference in arginine content in the soy and cottonseed protein isolate products used in our studies (He et al., 2015). Therefore, for the five soy meal-based products reported here, some other factors may also have contributed to the different behavior between soy and cottonseed products. For example, Chen et al. (2013) and Lorenz et al. (2015) reported that there are many possible impacts from carbohydrate compounds on the adhesive performance of soy meal products. Therefore, we assume that the different carbohydrate profiles in our soy and cottonseed products (He et al., 2015) might be a significant factor in the adhesive performance between the two types of oilseed products.

Table 4. Effect of tung oil (0.1%) on the dry and soaked shear strength of maple veneer specimens bonded at 100 and 130 °C, and change of soaked shear strength compared to the corresponding dry strength. Values are averages ± standard deviations (n=5)

	Dry strength (MPa)	Soaked strength (MPa)	Change (%)
	----------100 °C----------		
SM	2.29± 0.35	1.31± 0.31	-42.8*** [1]
SMw	3.05 ± 0.35	1.74 ± 0.14	-43.0 ***
SMr	2.74 ± 0.51	0.94 ± 0.24	-65.7 ***
SPIe	3.47 ± 0.80	3.48 ± 0.80	0.3 ns
SPIc	4.03 ± 0.58	4.05 ± 0.14	0.5 ns
	----------130 °C----------		
SM	2.35 ± 0.31	2.37 ± 0.23	0.9 ns
SMw	2.99 ± 0.80	2.30 ± 0.22	-23.1 ns
SMr	1.84± 0.52	1.00 ± 0.40	-45.6 *
SPIe	4.03 ± 0.49	5.04 ± 0.64	25.1 *
SPIc	4.14 ± 0.76	5.30 ± 0.49	28.0 *

[1] Symbol *, ***, and ns represent the significant difference between dry and soaked strength of the adhesive at *P*=0.05, 0.001, and no significant difference at *P*=0.05, respectively.

3.5 Green Strength

Green strength indicates the time to bond the wood veneers together before the adhesive develops ultimate bond properties when fully cured (Kong et al., 2011; Phetphaisit et al., 2013). In practice, a series of shear strength measurements with different curing times (i.e., time course) are performed. Those strength values before reaching the stable bonding strength are considered the green strength and the time with the stable adhesive strength is considered to be the time needed for the adhesive fully cured. Desai et al. (2003) measured the green strength (1-20 days) of biopolyol-based polyurethane adhesives for wood bonding. Their data showed polyols with high hydroxyl values cure rapidly to give high initial and final adhesive bond strength in wood joints. By contrast, polyols with low hydroxyl values take longer to cure. For evaluation of green strength of the soy meal based adhesives, both dry and soaked shear strengths of maple veneer specimens bonded with SMw/0.1%TO adhesive were measured with a cure time of one to 20 days (Figure 2). The dry shear strength increased to a maximum value of 3.44 MPa on Day 2 and the soaked shear strength reached a maximum value of 2.60 MPa on Day 4. After achieving the maximum values, the adhesive shear strength for both tests remained the same, or decreased slightly during the remaining testing times. The results confirmed our previous observation (He et al., 2014b) for washed cottonseed meal that four-days of conditioning is sufficient to assess the proper functioning of the oilseed meal/tung oil adhesive for both dry and soaked conditions.

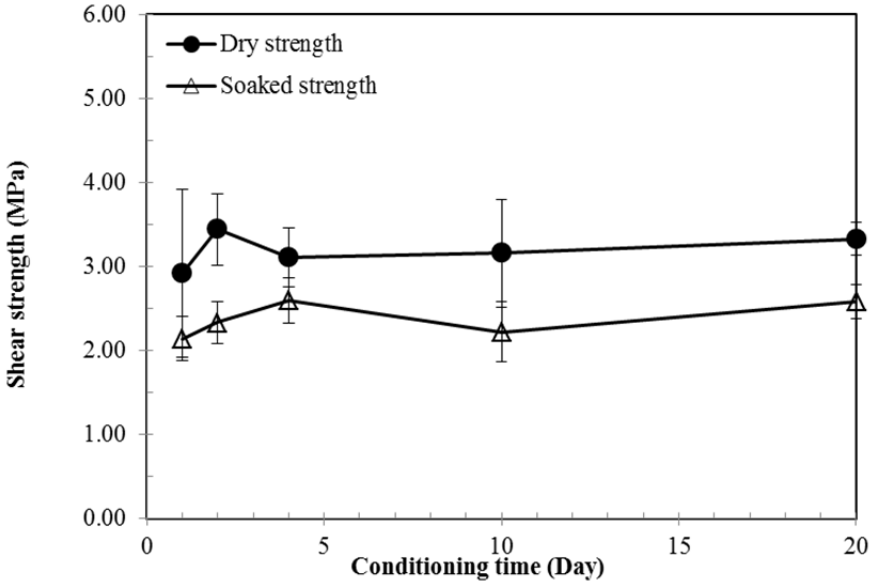

Figure 2. Dry and soaked shear strength of maple veneer specimens bonded with SMw/0.1%TO adhesive at 100 ◦C for 15 min as a function of conditioning time. Values are averages±standard deviations (n=5)

4. Conclusion

Five soy meal products were prepared and tested for their adhesive performance on bonding maple wood veneers. The protein content of these products was SM< SMr <SMw <SPIc <SPIe. Using the same amount of solid for each of the adhesive slurries, the dry shear strength of the bonded maple veneers was SM< SMr< SMw< SPIe< SPIc. Water soaking the bonded specimens reduced the adhesive strength of SM, SMw, and SMr, whereas the two protein isolates, SPIe and SPIc, remained the same. The water washed soy meal product SMw behaved more like untreated defatted soy meal SM than protein isolates, SPIe and SPIc. The effect of tung oil on the adhesive performance of the five soy meal products was minimal. These observations using soy meal products differed from our previous observations for cottonseed meal products. Further investigation is needed to determine the mechanisms and causes of the difference in adhesive performance between the two types of oilseed meal products.

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