ADHESIVE STRENGTH OF PILOT-SCALE WASHED COTTONSEED MEAL IN COMPARISON WITH A SYNTHETIC ADHESIVE FOR NON-STRUCTURAL APPLICATION

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Abstract

Water washed cottonseed meal (WCSM) has been shown the potential to be used as renewable and environment-friendly adhesives in wood products industry. Recently, WCSM was produced from defatted meal in a pilot scale. In this study, we initially compare the adhesive strength of the pilot-produced WCSM with that of a synthetic adhesive Vinavil 2259 L. Under the testing conditions, WCSM possessed very high heat resistance according to European standard EN14257 (WATT 91), adhesive strength after 1 hour at 80°C). However, the dry strength (EN204/205) and water resistance (specimen were put in water for 4 days and then pulled in the dynamometer) of WCSM were lower than those of the synthetic adhesive. Further work will be focused on the increase of solid content of the final adhesive slurries and the improvement of their water resistance.

Introduction

Great progress has been made on developing bio-based wood adhesives from renewable natural resources over last couple of decades (He, 2017). Defatted cottonseed meal and its protein isolate both showed wood bonding capability (Cheng et al., 2013; He and Cheng, 2017). However, the water resistance of cottonseed meal-based adhesives is poor, and the protein isolate is expensive in preparation. Thus, He et al. (He et al., 2014a) proposed water washing as a cost-effective method to improve the adhesive properties and water resistance of cottonseed meal. The product, water-washed cottonseed meal (WCSM), showed the adhesive performance comparable to cottonseed protein isolate (He et al., 2016a; 2014b). To promote WCSM as an industrial wood adhesive for non-structural application, in this work, we tested the WCSM's adhesive performance in reference to the relevant industrial standards. The results were further compared with an industrial synthetic adhesive. The information presented in this work would be helpful in formulating industrial-applicable WCSM-based wood adhesives.

Materials and Methods

Materials

WCSM was prepared from mill-produced cottonseed meal in a pilot scale (He et al., 2016c), and its basic properties were lsited in Table 1. Beech (Fagus sylvatica L.) with a density of 695 ± 15 kg/m3 and regular grain as specified in EN 205:2016 was used for the preparation of the various specimens. Wood assemblies were prepared by gluing beech thin boards of 640 mm x 125 mm x 5 mm. Each thin board was divided in two parts and, after spreading the adhesive on both sides, they were overlapped and put under the press to prepare the assembly.

Table 1. Selected components of pilot-produced washed cottonseed meal (WCSM). ADL- acid detergent lignin.

Major component	Moisture	Protein	Oil	Crude Fiber	ADL	Cellulose	Hemicellulose
%	8.6	46.3	1.0	16.0	9.4	17.6	8.4
Macro mineral	Ash	P	Ca	K	Mg	Na	S
%	5.2	1.22	0.27	1.04	0.67	0.08	0.46
Trace mineral	Fe	Zn	Cu	Mn	Ni	Al	B
ppm	166	100	15	50	0.5	130	20

Wood bonding and testing

Pilot-produced WCSM was used as it was without further treatments. WSCM (about 10%) was first mixed with water. WSCM slurry was applied for the bonding in the rates of 150 g m^{-2} and 250 g m^{-2}, respectively (dry weight). Wood assemblies were prepared by gluing beech thin boards of 640 mm x 125 mm x 5 mm. Each thin board was divided in two parts and, after spreading the adhesive on both sides, they were overlapped and put under the press (8 kg cm^{-2} or

0.8 MPa) for 30 min at 130 ºC to prepare the assembly (open time less than 1 minute, closed time 4 minutes, pressing time 2 hr, adhesive quantity 150 g/m2 by spreading the glue on each side of the assembly). (Chiozza and Pizzo, 2016; Chiozza et al., 2013).

For comparison, the synthetic adhesive, 2259 L, was used in bonding beech boards under the same conditions. Vinavil 2259 L is PVAc water based dispersion adhesive with the characteristics reported in Table 2. It is a water resistant adhesive belonging to the D3 class as defined by EN 204:2016.

Table 2. Typical analytical and technical characteristics of Vinavil 2259 L.

Analytical Characteristics	Method	Value
Polymer base	-	PVAc
Dispersing system	-	PVOH
Solid content	ISO 3251:2005	48 ± 1 %
Viscosity	ISO 2555:2002	11,500 ± 2,500 mPa·s
pH	ISO 976:1996	4.5 ± 0.5
Minimum Film Forming Temperature	ISO 2115:1996	+ 3°C
Durability class	EN 204:2016	D3

Results and Discussion

The results of the WCSM adhesive performance are listed in Table 3. The application rate seemed not a factor to influence the adhesive performance of WCSM as the two sets of data with different WSCM application rates were basically same. The dry shear strength of WCSM was 10.7 and 9.3 MPa, respectively, with the application rated of 150 g m^2 (WCSM 1) and 250 g m^2 (WCSM 2). The dry shear strength was much higher than the literature data (around 3.3 to 5.6 MPa) (He et al., 2016b). The higher adhesive strength observed in this work was apparently due to the thicker wood specimens and different bonding/testing conditions. After soaking for 4 days, the adhesive strength decreased to 0.5 and 0.3 MPa, respectively. The decreasing trend of adhesive strength by water soaking was observed before, however, the extent of the decrease observed in this study was greater than the water decrease reported in the literature (He et al., 2016a; 2016b). One possible cause was the longer (4 days) soaking time used in this work than the 2-days reported in (He et al., 2016a; 2016b). The adhesive strength after the bonded specimens exposed for 1 hour at 80°C was around 9.0 MPa, just about 10% decrease in average, compared to the dry adhesive strength. These data indicated that WSCM-based adhesives possessed good heat resistance. No heat resistance data of WCSM was reported before.

Table 3. Adhesive strength of WCSM adhesives with the application rated of 150 g m^2 (WCSM 1) and 250 g m^2 (WCSM 2) under different testing conditions

Testing type	Testing conditions	Method applied	Adhesive strength (MPa)	
			WCSM 1	WCSM 2
Durability class D1	Dry adhesive strength	EN 204/205	10.7	9.3
Durability class D3	Adhesive strength after water soaking for 4 days	EN204/205	0.5	0.3
Heat resistance	Adhesive strength after 1 hour at 80°C	EN14257	8.9	9.1

The results of the adhesive performance of the comparative synthetic adhesive Vinavil 2259 L are listed in Table 4. The solid content of the three formulations of the adhesives was all >44.0%. The solid content is an important parameter as the standard usually is used for adhesives having a solid content of about 50%, and the lower is the solid the lower will be the specific performance (in general it is important to make a comparison of adhesives showing the quite the same polymer content). Compared to the WCSM-based adhesives, the synthetic adhesives showed better water resistance, but poorer heat resistance. Furthermore, the blend of WCSM and Vinavil 2259 L did not improve the heat resistance of the synthetic polymer by keeping the D3 class to the adhesive. However, the addition of Vinavil 2259 L improved the water resistance of the WCSM allowing the classification of the adhesive as D3.

Table 4. Effect of solid content and WCSM addition on the adhesive strength of Vinavil 2259 L measured with the same testing conditions in Table 3.

Testing type	Testing conditions	Method applied	100 pbw 2259 + 10 pbw water	100 pbw 2259 + 10 pbw WCSM	2259 L as it is
Solid content	110 °C for 30 min	ISO 3251:2005	44.4%	44.8%	48.0%
Durability class D1	Dry strength	EN 204/205	15.5	12.3	15.8
Durability class D3	Water resistance	EN204/205	3.5	3.8	4.8
Heat resistance	Heat resistance	EN14257	5.5	6.0	7,0

pbw =parts by weight

Table 5. Four types of wood adhesives for non-structural applications and their classification requirements per European Standards (EN204, 2001; EN205, 2003). NR=not required.

Adhesive type	Application field condition	Minimum mean tensile shear strength of 10 testing (MPa)				
		T1	T2	T3	T4	T5
D1	Inside	10	NR	NR	NR	NR
D2	Inside, rare contact with water	10	8	NR	NR	NR
D3	Protected outside	10	NR	2	8	NR
D4	Outside, exposed to the weather	10	NR	4	NR	$

T1: Test immediately after 7 days in standard atmosphere at 20 °C and 65% relative humidity.
T2: 7-day conditioning as T1, 3-h soaking in water at 20 ± 5 °C, samples tested in the dry state after re-conditioning for 7 days.
T3: 7-day conditioning as T1, 4-day soaking in water at 20 ± 5 °C, samples tested immediately in the wet state.
T4: 7-day conditioning as T1, 4-day soaking in water at 20 ± 5 °C, samples tested in the dry state after re-conditioning for 7 days.
T5: 7-day conditioning as T1, 3-h soaking in boiling water, 2-h soaking in water at 20 ± 5 °C, samples tested immediately in the wet state.

European standards classify wood adhesives for non-structural application into four types (Table 5). EN204 (2016) requires a minimum average strength of 10 MPa for class D1 wood adhesives. The WCSM slurry prepared under the experimental conditions marginally meets the requirement. For more widely used D3 adhesives, a minimum average strength of 2 MPa of water resistance is needed. The wet strength of the WCSM slurries was between 0.3-0.5 MPa so that significant improvements of the water resistance of WCSM for its application as D3 adhesives. The heat resistance value (former WATT91 value) of WCSM was around 9.0. For practical applications, a minimum value of 7 MPa is usually considered an acceptable value for a D3 wood adhesive (EN14257, 2006). Thus, the WCSM data, \ meet the heat resistance requirement of D3 adhesives.

Summary

This study demonstrated that WCSM may be used as the conventional D1 wood adhesives for nonstructural interior application. Non-structural D3 type adhesives is more widely used in protected outside. WCSM also partly meet the requirements of D3 with under value of water resistance.

Future work will be on improvement of WSCM adhesive performance for D3 classification. The addition of 2259 L increased the performances of WCSM by allowing it to reach a D3 glue, mainly after curing at 130°C. The effects of blending with other synthetic adhesives, addition of AlCl3 and/or other ingredient, and lowering WCSM's particle size, will be tested for such purpose.

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