Bond Performance and Structural Characterization of Polysaccharide Wood Adhesive Made from Konjac Glucomannan/Chitosan/Polyvinyl Alcohol

Rong Gu,^a Baoning Mu,^b and Yanpu Yang^a

The bond performance and bonding mechanism were evaluated for a Konjac glucomannan (KGM), Chitosan (CS), and polyvinyl alcohol (PVOH) blended wood adhesive. An optimized experimental strategy was used to investigate the effects of the formula parameters of adhesives on the bonding strength of plywood using a Box-Behnken design and response surface methodology (RSM). The microstructure of the blended adhesives was analyzed by Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). An optimum bonding strength (3.42 ± 0.31 MPa) was achieved with concentrations of KGM, CS, and 10% PVOH of 2.3%, 2.3%, and 5.0%, respectively. There was strong hydrogen bonding between the KGM, CS, and 10% PVOH adhesives and the interface. SEM observations indicated that the blended adhesive exhibited a net-like structure that increased the overall bonding strength. These results provided the scientific basis for the continual development of environmentally friendly wood adhesives and the improvement of processing conditions.

Keywords: Wood adhesive; Polysaccharide; Bonding strength; Response surface methodology; Konjac glucomannan; Chitosan

Contact information: a: School of Construction Machinery, Chang'an University, Xi'an, 710064, China; b: Shaanxi Youth Vocational College, Xi'an, 710068, China; *Corresponding author: ronica@chd.edu.cn

INTRODUCTION

The requirement for adhesives in the wood industry increases every year in China. Approximately two thirds of manufactured adhesives are formaldehyde-based, and the raw materials are non-renewable, petrochemical products (Gong 2011). In 2004, the World Health Organization (WHO) reported that formaldehyde may cause cancer in humans (World Health Organization 2004). In addition, the use of formaldehyde-based adhesives may pollute the environment. Therefore, the development of non-toxic adhesives made from renewable biomass is important to the wood composite materials industry. Biomass materials may be one of the most suitable raw materials for generating wood adhesives. The extraction of polysaccharides from plants is of particular interest because of their wide availability and low cost (Feng and Ye 2005).

Konjac glucomannan (KGM) is a water-soluble, non-ionic polysaccharide that can be extracted from *Amorphophallus* sp. tubers. It shows excellent properties for filmforming, gel formation, thickening, blending (Xu and Wang 2002; Zhang *et al.* 2005), viscosity, and biological adhesive bonding strength. Chitin is another renewable natural polymer material that is widely available. Natural cationic polysaccharide chitosan (CS) is obtained from chitin. Chitosan exhibits excellence in biodegradability, film-forming ability, biocompatibility, and antibacterial and antitumor properties (Wang *et al.* 2009; Patel *et al.* 2013). Chitosan is widely used in the medical, food, chemical, and environmental protection industries. Polyvinyl alcohol (PVOH) is a white, powdery, water-soluble polymer resin obtained by the hydrolysis of PVOH. It has applications as an adhesive and an adhesive additive (Li and Xie 2004; Baker *et al.* 2012).

In this study, KGM, CS, and PVOH were prepared and combined into a blended adhesive. To obtain the optimal preparation conditions for good bonding strength, a Box-Behnken design (BBD) was used to optimize the preparation conditions, including the final concentrations of KGM, CS, and 10% PVOH (Li *et al.* 2009; Kumar 2016). An analysis of the molecular interactions between the blended adhesive and the microstructure of the blended adhesive interface was performed using Fourier transform infrared spectroscopy (FTIR). Finally, scanning electron microscopy (SEM) of the adhesive film structure was performed to determine the bonding mechanism of the new adhesive material.

EXPERIMENTAL

Materials

Konjac glucomannan (100 mesh) was provided by the Chongqing Limao Agricultural Products Development Co., Ltd. (Chongqing, China), with a glucomannan content exceeding 92% and a molecular weight of 256,000. Chitosan (80 mesh) was obtained from the Xianju Tengwang Chitosan Factory (Zhejiang, China), with a deacetylation degree above 95% and a molecular weight of 213,000. Polyvinyl alcohol (analytically pure, 99.8% to 100% degree) was purchased from the Tianjin Kemiou Chemical Reagent Co., Ltd. (Tianjin, China) and a molecular weight of 14,000. Glacial acetic acid (HOAc, analytical pure) was purchased from the Xi'an Chemical Reagent Factory (Shannxi, China). All of the above reagents were used without further purification.

Poplar rotary-cut veneer with a thickness of 1.5 mm was sawn in the laboratory using a wood veneer lathe (8% to 9% moisture content) and later sectioned into 30×30 cm² samples.

Preparation of the Adhesive Blends and the Adhesive Films

The process was divided into three stages: preparation of the PVOH solution (10% mass fraction), preparation of the KGM/CS blended solution, and preparation of the KGM/CS/PVOH blended adhesive.

In the first stage, PVOH was weighed and swelled at room temperature for 5 to 10 min, dissolved in distilled water at 10% (w/w) concentration, and incubated at 90 °C for 20 to 30 min until completely dissolved. In the second stage, the glacial acetic acid solution (1% mass fraction), konjac glucomannan, and chitosan powders were weighed and dissolved in distilled water according to the formulation shown in Table 1. The blended solution was stirred vigorously for 30 min to 1 h at room temperature using a mechanical stirring machine.

In the third stage, the 10% PVOH solution was mixed with the blending adhesive and stirred vigorously for 1 to 2 h at room temperature with a mechanical stirring machine. The adhesive was stirred until a clear, homogeneous polymer solution was obtained. After 2 h of standing and deaerating, the blended adhesive was added to assembled three-layer veneers. PEER-REVIEWED ARTICLE

The prepared adhesives (KGM, CS, KGM/CS, KGM/CS/PVOH) were coated on a glass plate by flow casting and cured at 130 °C in an oven. The resulting adhesive films were peeled from the glass plates. The average thickness of the film was 0.53 ± 0.16 mm.

	Coded Levels			Viscosity	Solid Contents	Bonding Strength (MPa)	
Run No.	KGM (X1, %)	CS (X ₂ , %)	10% PVOH (X ₃ , %)	(Pa∙s)	(%)	Experimental	Predicted
1	1(2.5%)	-1(1.5%)	0(5%)	236	4.5	2.13	2.15
2	-1(1.5%)	0(2%)	1(6%)	123	4.1	2.05	2.07
3	0(2%)	0	0	214	4.5	3.18	3.16
4	1	0	-1(4%)	358	4.9	3.00	2.98
5	0	0	0	208	4.5	3.15	3.16
6	1	1(2.5%)	0	612	5.5	3.40	3.41
7	0	-1	1	101	4.1	2.00	2.01
8	0	0	0	225	4.5	3.20	3.16
9	0	1	-1	347	4.9	2.95	2.97
10	-1	-1	0	76	3.5	1.44	1.43
11	0	1	1	531	5.1	2.97	2.97
12	-1	1	0	184	4.5	2.26	2.24
13	-1	0	-1	81	3.9	2.00	1.98
14	0	0	0	196	4.5	3.14	3.16
15	1	0	1	427	5.1	3.00	2.99
16	0	0	0	217	4.5	3.12	3.16
17	0	-1	-1	83	3.9	1.90	1.90

Table 1. Box-Behnken Design and Response to Bonding Strength

All the above formulations were dissolved in the glacial acetic acid solution (1% mass fraction).

Preparation of the Three-Layer Plywood

The KGM/CS/PVOH adhesive was applied to a single side of the 30×30 cm² surfaces, the back veneers, and both sides of the core materials. The three pieces were assembled with the grain of the core perpendicular to the grain of the surface and back veneers. The total amount spread was 200 g/m².

The assembled, three-ply veneers were cold-pressed at 0.98 MPa and room temperature for 2 h (to eliminate the inner force of the veneer), and then the pressure was increased to 3 MPa for 1 min. Next, the samples were hot-pressed at 130 $^{\circ}$ C at 3.0 MPa for 15 min. Finally, the pressure was reduced in three stages: 2.0 MPa for 1 min, 1.0 MPa for 1 min, and then complete release.

Glue Bond Strength Tests

The prepared plywood was cut into standard tensile strength test specimens ($10 \times 2.5 \text{ cm}^2$) according to the GB/T 9846.7 (2004). Determination of bonding strength was based on the GB/T 17657 (2013). The dry bonding strength of the plywood was measured with a SANS testing machine (MTS Systems Corp., Shenzhen, China) under a loading rate of 5 mm/min.

Experimental Design

The concentrations of KGM ($X_1 = 1.5\%$, 2.0%, and 2.5%), CS ($X_2 = 1.5\%$, 2.0%, and 2.5%), and 10% PVOH ($X_3 = 4.0\%$, 5.0%, and 6.0%) were varied to determine the effect on the bonding strength of the wood. The BBD was applied to study the effects of the initial concentrations of KGM, CS, and PVOH on the bonding strength of the adhesive. Design-Expert software (Trial Version 8.0.6, Stat-Ease, Minneapolis, MN) was used to analyze the data. The selection range of each variable is shown in Table 1. The BBD consisted of 17 experiments; 12 experiments were organized in a factorial design and an additional 5 were replicated at the central point of the designed model to estimate the pure error sum of squares.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

All samples tested using a SANS apparatus (MTS Systems Corp., Shenzhen, China) were selected according to a physical index of failure rate that frequently is required to be 0%. The wood bonded interface of the chosen samples were cut with a microtome, therefore were scanned using FTIR. Wood veneer samples were cut with a microtome from poplar veneer without coating and scanned using FTIR. All samples and adhesive films were vacuum dried at 80 °C for 24 h, treated with KBr, and tested at room temperature from 400 to 4000 cm⁻¹ using FTIR (TENSOR 27, Bruker, Karlsruhe, Germany).

Scanning Electron Microscopy (SEM) Observation

All samples tested using a SANS apparatus were selected according to a physical index of failure rate that frequently is required to be 0%. the cross-section of the chosen samples, therefore were observed based on a SEM image.

The test specimens were dried at 100 °C and spray-coated in gold. The microstructure of test specimens was observed by SEM (JSM-6360LV, JEOL, Tokyo, Japan). The working conditions included an accelerating voltage of 30 kV, 25 KV, 5×10^{-9} mA beam, and a working distance of 15 mm.

RESULTS AND DISCUSSION

Analysis of the Adhesive Strength Property of KGM/CS/PVOH Blended Adhesive

To determine the effects of the three adhesives on the bonding strength, the BBD design was chosen with the corresponding results of the response surface methodology (RSM) experiments (Table 1). An analysis of variance regression analysis was performed to evaluate the effect of adhesive type on the bonding strength (Table 2). The model was based on the following quadratic polynomial equation:

$$Y = -19.57625 + 6.514X_{1} + 7.59150X_{2} + 2.64125X_{3} + 0.45X_{1}X_{2} - 0.025X_{1}X_{3}$$

- 0.04X_{2}X_{3} - 1.586X_{1}^{2} - 1.816X_{2}^{2} - 0.249X_{3}^{2} (1)

where Y is the bonding strength of the blended adhesive, and X_1 , X_2 , and X_3 are the concentrations of KGM, CS, and 10% PVOH, respectively.

As shown in Table 2, the *P*-value of the model was significant (P < 0.001), and the lack-of-fit value was 0.4829, indicating that the model fit the data well. The coefficient of

determination (predicted R^2), is called a fitness degree (*i.e.*, the ratio of explained variation to total variation). A small value indicated poor relevance of the dependent variables in the model. An R^2 value of 0.9988 indicated that 99.88% of the variation between measures could be explained by model. The predicted R^2 of 0.9908 is in reasonable agreement with the adjusted R^2 value of 0.9973, indicating that there was only 2.64% of the total variation that could not be explained by the model. Therefore, this regression model described the true behavior of the system.

Source	Sum of Squares	Degrees of freedom	Mean Square	F-value	<i>P</i> -value
Model	5.94	9	0.66	650.22	< 0.0001
X1	1.79	1	1.79	1759.66	< 0.0001
X ₂	2.11	1	2.11	2080.31	< 0.0001
X ₃ - 10%PVOH	3.613E-003	1	3.613E-003	3.56	0.1012
X1X2	0.051	1	0.051	49.88	0.0002
X ₁ X ₃	6.250E-004	1	6.250E-004	0.62	0.4584
X ₂ X ₃	1.600E-003	1	1.600E-003	1.58	0.2496
X1 ²	0.66	1	0.66	652.16	< 0.0001
X ₂ ²	0.87	1	0.87	855.03	< 0.0001
X ₃ ²	0.26	1	0.26	257.20	< 0.0001
Residual	7.105E-003	7	1.015E-003		
Lack of Fit	3.025E-003	3	1.008E-003	0.99	0.4829
Pure Error	4.080E-003	4	1.020E-003		
Cor Total	5.95	16		1.9	

Table 2. Analysis of Variance for the Regression Model of Bonding Strength

P < 0.01 highly significant; 0.01 < P < 0.05 significant; P > 0.05 not significant

The adequacy of the models was further justified by the analysis of variance (ANOVA) results. The ANOVA for the quadratic model of bonding strength is shown in Table 2. The F-value (650.22) and *P*-values (< 0.0001) implied that the model was significant and that there was only a 0.01% chance that the data could occur because of noise. The significance of the model was also analyzed by a lack-of-fit test. As shown in Table 2, the F-value and *P*-value of the lack-of-fit test were 0.99 and 0.4829, respectively, which implied that the model was not significant and exhibited a 48.29% chance that the data could have occurred because of noise. In this case, the concentrations of KGM (X₁), CS(X₂), their interaction (X₂X₃), and the three quadratic terms (X₁², X₂², and X₃²) significantly (*P*-value) affected the bonding strength, but the concentration of 10% PVOH (X₃), X₁X₃, and X₂X₃ did not significantly (*P*-value) affect the bonding strength. This indicates that the most significant interactions were dependent on the concentrations of KGM (X₁) and CS (X₂). Therefore, X₁ and X₂were the most important factors, followed by their interaction (X₁X₂).

To further analyze the effect of the three adhesives on the bonding strength of the blends, a three dimensional (3D) response surface and two dimensional (2D) contour plot representing the graphical response of the regression equations was conducted. The relationship between the parameters and the response variable was illustrated in a 3D representation of the response surfaces, and 2D contour plots were generated by the model for bonding strength of the blended adhesive (Fig. 1).

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Fig. 1. Response surface (left) and contour plots (right) for the bonding strength of blended adhesive: a) KGM and CS concentrations; b) KGM and 10% PVOH concentrations; c) CS and 10% PVOH concentrations

Combined with the results in Table 2, X_1 and X_2 showed a significant (*P*-value) effect on the bonding strength, whereas X_3 was not significant (*P*-value). As shown in Fig. 1, the bonding strength increased significantly (*P*-value) as the concentration of KGM and

CS increased from 1.5% to 2.0%. When the concentrations of KGM and CS were increased from 2.0% to 2.5%, the bonding strength increased rapidly. The bonding strength improved initially and then decreased as the concentration of 10% PVOH increased. Compared with the concentrations of KGM and CS, 10% PVOH only slightly affected the bonding strength of the adhesive. The interactions among 10% PVOH, KGM, and CS did not significantly (*P*-value) impact the bond strength; however, the interaction between KGM and CS was significant (*P*-value).

The range in the optimal preparation conditions for blended adhesives was obtained using Eq. 1, which was derived from the surface response experiments using Design Expert software. The predicted optimal condition for the blended adhesive was with 2.32% KGM, 2.28% CS, and 5.02% of 10% PVOH. Under this condition, the model predicts a maximum bond strength of 3.48 MPa. In consideration of practical operating conditions and to simplify the protocol, the concentrations were rounded to the nearest 10^{ths} place, resulting in a final condition of 2.3% KGM, 2.3% CS, and 5% of 10% PVOH. Under these conditions, an average strength value of 3.42 ± 0.31 MPa was obtained, which was close to the model's predicted value. This confirms that the model adequately reflects the expected optimization and that Eq. 1 is satisfactory and accurate.



Fig. 2. Fourier transform infrared spectrum of a) KGM, CS, and PVOH and b) KGM/CS and KGM/CS/PVOH

Infrared Spectrum Analysis

The infrared spectra of KGM, CS, PVOH, and their blends are shown in Fig. 2. A comparison of the infrared spectra of the KGM films and the KGM/CS blended adhesives revealed the following changes: the mannose characteristic absorption peaks at 878.3 cm⁻¹

and 807.6 cm⁻¹ were still present in the KGM/CS blended adhesive, indicating that the primary structure of the KGM remained intact after bending KGM and CS (Li *et al.* 2007); the carbonyl characteristic peak of KGM at 1727.9 cm⁻¹ disappeared with the KGM/CS blend and some of the acetyl groups were removed; and the =CH₂ absorption peak of KGM at 1394.6 cm⁻¹ changed with the KGM/CS blend. Compared with the two single polymers, the absorption peaks of the KGM/CS blend at 3200 cm⁻¹ to 3700 cm⁻¹ and 2800 cm⁻¹ to 3000 cm⁻¹ showed different degrees of displacement. This indicated that after blending the stretching vibration of -OH, -NH, and -CH underwent considerable changes.

In comparison with the spectra of the CS films, the KGM/CS blended adhesives indicated several changes. The deformation vibration absorption peak of -NH₂ located at 1598 cm⁻¹ in pure CS completely disappeared in the blended film, and a new absorption peak at 1485 cm⁻¹ appeared that can be attributed to the bending oscillation of -NH₂, indicating that the CS and the KGM in the blended film formed a polyelectrolyte complex due to the strong electrostatic interaction of anion and cation. The absorption peak of O-H and N-H near 3418.5 cm⁻¹ gradually widened, indicating that CS and KGM hydrogen bonded in the blended film, and the crystalline area of CS at 1028 cm⁻¹ was significantly (*P*-value) modified by the blended film (Li *et al.* 2012a), such that the absorption peak at 1028 cm⁻¹ became inseparable from the shoulder peak. This further supported a strong interaction between CS and KGM in the blended film and indicated a modification in the original crystal structure of CS.

A comparison of the spectra containing the three adhesives and KGM\CS\PVOH blended adhesive revealed that the absorption peaks of KGM at 1645.9 cm⁻¹, 878.3 cm⁻¹, 807.6 cm⁻¹, and 1727.9 cm⁻¹ in the blended adhesive were all shifted. The absorption peak of CS in the vicinity of 1598 cm⁻¹ shifted to approximately 1563 cm⁻¹ for the blended adhesive. The deformation vibration peak of -CH₂ of PVOH (1425 cm⁻¹) increased after blending. In addition, a new absorption peak appeared at 1663 cm⁻¹ for the blended adhesives. The blended adhesive showed an absorption peak near 1624 cm⁻¹ and the large characteristic peak at 3316 cm⁻¹ became a sharp peak. The -OH and -NH groups were reduced, and the hydrophilicity was weakened. These facts supported hydrogen bonding between the molecules of the KGM, CS, and 10% PVOH blends.

Fourier transform infrared spectral analysis confirmed the interaction between the KGM, CS, and 10% PVOH. There was an interaction between the amino and hydroxyl groups of KGM and CS, and hydrogen bonding occurred between the hydroxyl groups. PVOH, KGM, and CS contain a large amount of hydroxyl groups, with the latter two containing amino groups (Chen *et al.* 2015a, b; Prawitwong *et al.* 2007). There was evidence of hydrogen bonding between KGM and PVOH, CS and PVOH, and KGM and CS. This bonding resulted in a three-dimensional network structure.

Lignin and cellulose are the main chemical components of wood. The bonding of wood is primarily influenced by the strength of the adhesive, the interaction between wood and adhesive molecules, and the polarity of the wood (Liu *et al.* 2008). Bonding requires that the self-strength of the adhesive is equal to or greater than the self-strength of the wood.

Figure 3 shows the FTIR spectrum of poplar veneer and the adhesive interface. The poplar veneer exhibited a hydroxyl O-H absorption peak at 3425 cm⁻¹ and an absorption peak of methyl and methylene C-H at 1737 cm⁻¹. The non-conjugated carbonyl group C=O and conjugated carbonyl group C=O showed expansion vibrations at 1737 cm⁻¹ and 1623

cm⁻¹, respectively (Lin *et al.* 2012b), C-O-H had stretch vibration at 1157 to 1054 cm⁻¹, and a cellulose C-H absorption peak at 895 cm⁻¹.



Fig. 3. Fourier transform infrared spectra of poplar veneer and the interfacial adhesion of poplar veneer

The following changes were evident when compared with the adhesive's interface. The peak intensity of the hydroxyl group O-H at 3425 cm⁻¹ was greatly reduced, indicating a strong interaction of the hydroxyl group between the wood and the adhesive. The peak intensities of the methyl group, methylene group, and the non-conjugated carbonyl group also significantly (*P*-value) decreased to 2932 cm⁻¹ and 1737 cm⁻¹. The absorption peak of the conjugated carbonyl C=O group at 1623 cm⁻¹ disappeared. These active groups demonstrated the strong interaction between the adhesives and wood interface. The absorption peak of C-O-H at 1157 to 1054 cm⁻¹ notably decreased and shifted. The change in the intensity of the absorption peak indicated a strong interaction between the wood interface and the adhesive.



Fig. 4. Scanning electron micrographs of the bonding interface of poplar plywood at a) 50X and b) 300X

Scanning Electron Microscope Analysis

The cross-section of the plywood was enlarged 50 times by SEM (Fig. 4a). The interlaced poplar veneers combined tightly, and the vessels in the middle portion of the poplar veneers and pits and scalariform perforation in the duct wall were clearly visible.

The morphology of the cell cavity, surrounded by the adhesive, was clearly visible between the vertical and horizontal veneers. The characteristics of the wood cell wall between the cross-section almost disappeared, consistent with the action of the adhesive. The junction of the adhesive appeared to spread evenly into the wood structure, filling the pits of the wood vessels and masking the characteristics of the cell wall. This resulted in an enhancement of the cell wall's strength.

The cross-section of the plywood was enlarged 300 times by SEM in Fig. 4b. The adhesive and wood between the interlaced poplar veneers exhibited good bonding capabilities. The good interfacial contact revealed in the SEM image suggests that there was physical attraction between the polymers, including van der Waals and hydrogen bonding forces. Although the unit value of these forces is small, the large molecular weight makes the sum considerably greater, affecting the strength of the bond. Wood is a porous material with many liquid channels equaling approximately 25% to 85% of the total volume of wood. Because of capillary tension, liquid adhesive should enter the channel and act as a "plastic nail" upon curing. The result is an improvement in the mechanical strength of wood between the interface layers and an increase in the binding properties between the adhesive and the wood.

Deep penetration of the wood cell walls, and the mutual penetration of the mesh polymer formed by adhesive curing, increases cell wall stability (Wimmer *et al.* 2013). The interfacial penetration of the adhesive improves the bonding performance of wood veneer; however, too much adhesive can reduce the formation of a glue layer and affect bond performance. Thus, the penetration depth of the adhesive can affect the finished product.



Fig. 5. Scanning electron micrographs of the KGM/CS/PVOH blending adhesives at a) 1000X and b) 3500X

The KGM/CS/PVOH blend adhesive films are shown in enlarged SEM micrographs in Figs. 5a and 5b, respectively. The ternary blends were uniform and smooth, indicating the proper ratio of KGM, CS, and 10% PVOH. The ternary blends produced strong hydrogen bonding between the surfaces of the films, forming a distinct network structure that improved the bonding strength (Li *et al.* 2009).

CONCLUSIONS

- 1. Optimum bonding strength $(3.42 \pm 0.31 \text{ MPa})$ was achieved with 2.3% konjac glucomannan (KGM), 2.3% chitosan (CS), and 5% of 10% PVOH.
- 2. Fourier transform infrared spectra analysis showed that there was strong hydrogen bonding among the KGM, CS, and 10% PVOH adhesives. The active group of the adhesive interacted with the hydroxyl groups on the cellulose and hemicellulose of the poplar veneer. This indicated a strong interaction between the adhesive and wood interface.
- 3. Scanning electron microscopy observations of film structure indicated that the blended adhesive exhibited a net-like structure that increased the bonding strength. These results provide the scientific basis for the development of an environmentally friendly wood adhesive.

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