

Preparation and Properties of Oriented Cotton Stalk Board with Konjac Glucomannan-Chitosan-Polyvinyl Alcohol Blend Adhesive

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The use of formaldehyde-free, biomass-based composites has gained increasing attention in recent years because of their environmental benefits and superior strength properties. In this study, oriented cotton stalk board (OCB) was fabricated with an environmentally friendly, water-based konjac glucomannan-chitosan-polyvinyl alcohol (KCP) blend adhesive using hot pressing technology. The effects of pressing parameters on the physical and mechanical properties of oriented cotton stalk board were examined in order to obtain optimal pressing parameters. Interfacial bonding surface was also examined with a scanning electron microscope and a fluorescence microscope. The optimal physical and mechanical properties were obtained at a pressing temperature of 150 °C for 15 min with a target density of 0.8 g/cm³ during hot pressing. Adhesive content and hot pressing pressure had significant influences on adhesion. Mechanical interlocking was also observed between cotton stalks and the adhesive. OCB with KCP blend adhesive has comparable mechanical properties to that with urea formaldehyde resin or phenolic formaldehyde resin. OCB resinated with KCP blend adhesive is environmental friendly and has potential applications in furniture and interior decoration with less stringent requirements for water resistance.

Keywords: Cotton stalk; Konjac glucomannan; Chitosan; Polyvinyl alcohol; Adhesive

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INTRODUCTION

Cotton is a common crop throughout the world. China is the world's largest cotton-producing country, accounting for nearly 30% of annual global cotton production (Li *et al.* 2013). After cotton balls are harvested, cotton plant residues such as stalks, leaves, and shells remain. Unfortunately, these stalks are often burned as fuels or ground in the field as fertilizer (Du *et al.* 2013; Haykir and Bakir 2013; Xiong *et al.* 2014). Cotton stalks could be a promising alternative to wood because they have similar composition with wood (mainly composed of lignin, cellulose, and hemicellulose). Compared with other agricultural residues, cotton stalks have higher cellulose and lignin contents, and can produce longer fiber bundles with fast growth rate and high yield (Zhou *et al.* 2010). Cotton stalks have been used as a raw material for composites fabrication in a few previous studies (Nath and Chawla 2011; Qi *et al.* 2012; Fahmy and Mobarak 2013; Holt *et al.* 2014). Cotton stalk scrimbers were made by Song (2008) with urea

formaldehyde (UF) resin, exhibiting acceptable mechanical properties, but the final products were found to release formaldehyde. Qi *et al.* (2012) used high-density polyethylene (HDPE) as adhesive and maleic anhydride polyethylene as coupling agent to fabricate cotton stalk bundles and thermoplastic composites using hot-press molding. This product has great water resistance property, but its mat is required to cool down before removing out during consolidation.

Recent interest in developing environmentally friendly products has expanded the application of formaldehyde-free adhesives (Kim 2009; Pizzi and Mittal 2011; Ping *et al.* 2012). Transparent films have been prepared by blending aqueous konjac glucomannan solution with chitosan acetate solution (Xiao *et al.* 2000). Edible composite films based on konjac glucomannan, chitosan, and soy protein isolate have also been developed. Studies have examined the effects of polymer composition, glycerol concentration, and the pH of the film-forming solution on the water vapor permeability, tensile strength, and percentage elongation at break of the film (Jia *et al.* 2009). Our previous research developed a water-based adhesive by blending konjac glucomannan, chitosan, and polyvinyl alcohol and applied it to plywood in the laboratory (Gu *et al.* 2010). The performance of konjac glucomannan, chitosan, and polyvinyl alcohol blend adhesive depends on the dispersion and concentration of konjac glucomannan and chitosan in water as well as the complex degree of the three elements in the solution (Gu *et al.* 2010). However, the application of water-based KCP blend adhesive on the OCB and the properties of this kind of OCB have not been studied.

Many factors affect the final properties of OCB, such as the board density, pressing parameters, raw material properties, resin type, layer structure, and board moisture content. Board density and pressing parameters significantly influence both the physical and mechanical properties of the particleboard (Nemli 2009; Liu *et al.* 2011).

The objective of this study was to fabricate OCB with KCP blend adhesive by hot pressing molding. The effects of board density and hot pressing parameters on the physical and mechanical properties of OCB were investigated. The morphological characteristics of OCB were also observed by a scanning electron microscope (SEM) and a fluorescent microscope, aiming to find out the adhesion mechanism between cotton stalks and KCP blend adhesive.

EXPERIMENTAL

Materials

Konjac glucomannan (KGM) (100-mesh, molecular weight 256,000) was purchased from Qindong Konjac Food Co., Ltd. (Shaanxi, China). The glucomannan content was 97%, with a viscosity of 10 Pa·s at 1 wt. % concentration.

Chitosan (CH) (80-mesh, molecular weight 213,000) was obtained from Golden-shell Biochemical Co., Ltd. (Zhejiang, China) with an intrinsic viscosity of 130 mPa·s, and a deacetylation degree of 95.2%.

Polyvinyl alcohol (PVA) (98% acetalized, molecular weight 14,000) and glycerol were obtained from Tianjin Kemiou Chemical Reagent Co., Ltd. (Tianjin, China). All of the above reagents were used without further purification. Cotton stalks were obtained from local farmland in Shaanxi Province, China.

Cotton Stalk Preparation

The roots of cotton stalks were removed with a saw and crushed with a two-roller crusher after the barks and branches were removed by hand. The crushed cotton stalks were cut into pieces with a length of 450 mm and a thicknesses of 2 to 3 mm. Their moisture contents were reduced to around 15% by oven drying.

Blend Adhesive Preparation

In this study, the adhesive was fabricated using the method described by Gu *et al.* (2013). Firstly, chitosan and konjac glucomannan powders were sequentially weighed and dissolved in distilled water at 2.5% w/w concentration, and the mixture was stirred vigorously at room temperature with a mechanical stirring machine. Glacial acetic acid was added by 1% w/w while stirring. Secondly, polyvinyl alcohol was weighed and dissolved in distilled water to 10% w/w concentration at 95 °C until it was transparent. Finally, the aqueous polyvinyl alcohol solution was added to the mixture by 6% w/w. The adhesive was then stirred to obtain a clear, homogeneous polymer solution. The final concentration of blended adhesive was 2.5% chitosan, 2.5% konjac glucomannan, and 0.6% polyvinyl alcohol, and the blend adhesive with a solid content of 5.6% was used in this study. After 2 h of gelatinization and degassing, the blended adhesive could be used (Gu *et al.* 2010).

Oriented Cotton Stalk Board Fabrication

KCP blend adhesive was applied to the cotton stalks with a two-roller gluing machine. The gauge distance and rotation speed were controlled to obtain a specific adhesive content. As KCP blend adhesive is a water-based adhesive with low solids content, the moisture content of cotton stalks after applying adhesive was quite high and they were oven-dried at 100 °C to reduce the moisture content to around 15% before forming. The stalks resinated with KCP blend adhesive were weighed and manually formed into a mat with all stalks parallel to the longitudinal direction in a forming box. Release papers were placed on both surfaces of the mat to avoid the mat sticking on the caul plates. Mats were pressed into a 450 mm × 450 mm × 10 mm board with a hot press (Wei Di Electrical Technology Co., Ltd., Xianyang, China) (Qi *et al.* 2012). A three-step pressing schedule was used to avoid blistering (Gu *et al.* 2010), and the pressing curve is shown in Fig. 1. The target density of the OCB could be achieved by controlling the weight of raw materials, and the final moisture content of the OCB was approximately 6%.

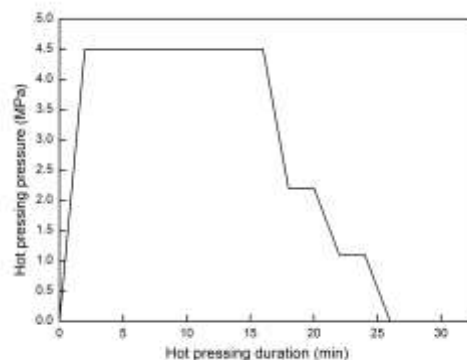


Fig. 1. Hot pressing curve during panel fabrication

Experimental Design

The effects of board target density and pressing parameters on the physical and mechanical properties of OCB were studied. The mechanical properties evaluated included modulus of elasticity (MOE), modulus of rupture (MOR), and internal bonding strength (IB), and the physical properties included water absorption (WA) and thickness swelling (TS). The fixed factors were KCP blend adhesive content (10% based on dry mass), board thickness (10 mm), and mat moisture (around 15%). Table 1 shows the single-factor experimental design of this study. Each sample was replicated three times.

Table 1. Single-Factor Experimental Design

Sample	Hot Pressing Duration (min)	Target Density (g/cm ³)	Hot Pressing Temperature (°C)
A1 to A5	5,10,15,20,25	0.7	150
B1 to B5	15	0.5, 0.6, 0.7, 0.8, 0.9	150
C1 to C5	15	0.7	110, 130, 150, 170, 190

The optimum results of the single-factor experiment were further analyzed by orthogonal experiment (L9 (3, 4)), and Table 2 gives the details of orthogonal experiment design. Each sample was replicated three times.

Table 2. Orthogonal Experimental Design

Sample	Hot Pressing Duration (min)	Target Density (g/cm ³)	Hot Pressing Temperature (°C)
T1	1	1	1
T2	1	2	2
T3	1	3	3
T4	2	1	3
T5	2	2	1
T6	2	3	2
T7	3	1	2
T8	3	2	3
T9	3	3	1

Note: 1, 2, and 3 stand for three different levels of each factor.

Physical and Mechanical Properties Test

The mechanical (MOE, MOR, and IB) and physical (WA and TS) properties of the OCB were tested according to ASTM D1037-06a (2006) standard methods. All specimens were conditioned to equilibrium at a temperature of 20 °C and 65% relative humidity. Ten specimens were used for the physical and mechanical tests for each sample.

Surface Characteristics of OCB

Cotton stalks before pressing and stalks in the OCB were observed under SEM. Stalks were frozen in liquid nitrogen and then cut into specimens. The test specimens were oven dried at 100 °C to reduce the moisture. Dried specimens were coated with gold powder in a sputter coater for examination. The microstructure of the samples was observed with a SEM (Hitachi, Model S2400, Japan) at an acceleration voltage of 5 kV.

Surfaces of OCB without adhesives and OCB with KCP blend adhesive were observed under a fluorescence microscope. Specimens with a size of 5 mm × 15 mm × 10

mm were used for fluorescent microscope observation. Test specimens were placed onto a microscope slide and fixed with clay, and were observed with a fluorescence microscope (Olympus, Model DP72, USA).

Statistical Analysis

Data for each test were statistically analyzed with the Statistical Package for Social Sciences (SPSS version 13.0; IBM, USA) for Windows. All data were expressed as the mean value \pm the standard deviation. Figures were drawn with Origin Pro 8.5 software (Origin Lab Co., USA).

RESULTS AND DISCUSSION

Single Factor Experiment Results

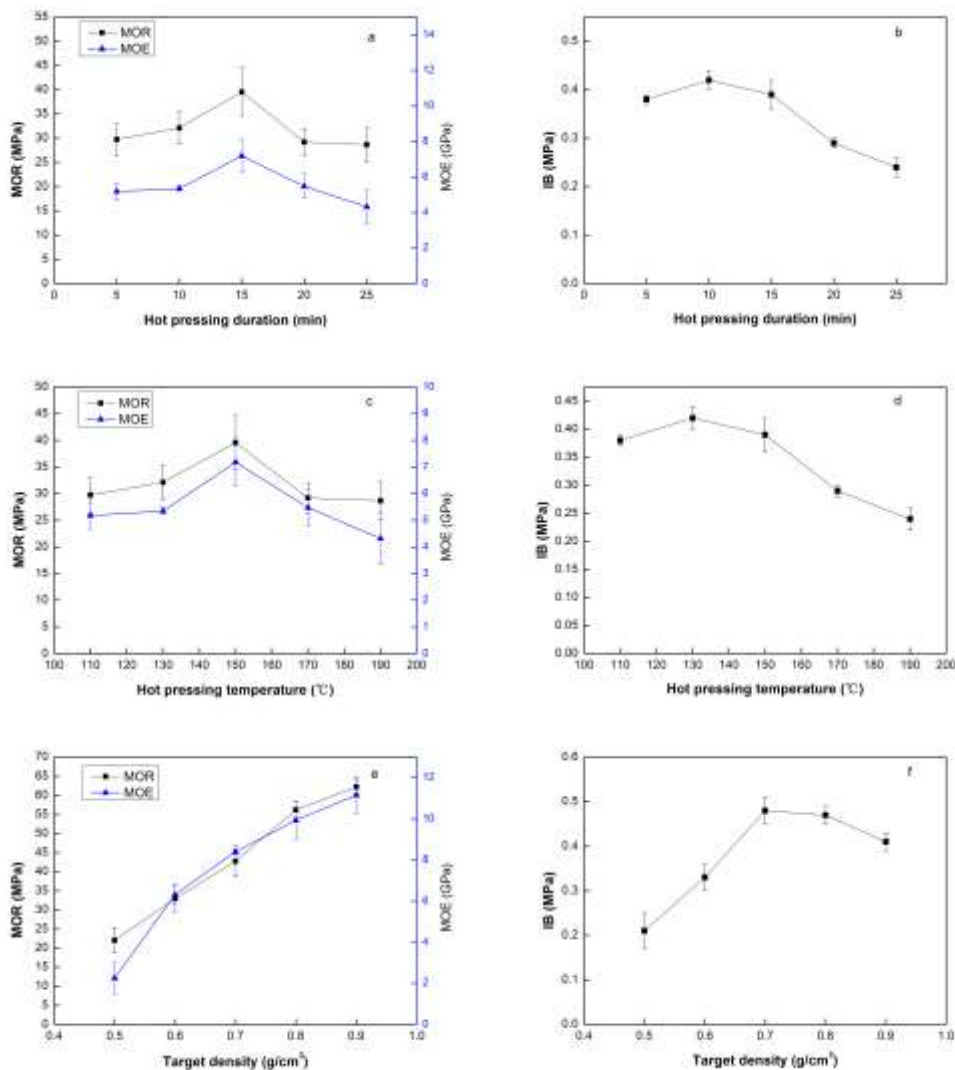


Fig. 2. Single factor experiment results : (a) Effects of hot pressing duration on MOR and MOE; (b) Effect of hot pressing duration on IB; (c) Effects of hot pressing temperature on MOR and MOE; (d) Effect of hot pressing temperature on IB; (e) Effects of target density on MOR and MOE; and (f) Effect of target density on IB

Figure 2 displays the effects of the hot pressing parameters on the mechanical properties of OCB. As shown in Fig. 2a, the values of MOE and MOR initially increased with pressing duration, and then they decreased when pressing duration was over 15 min. The maximum MOR and MOE (35.99 MPa and 7.18 GPa, respectively) were observed at a hot pressing duration of 15 min, while the lowest MOR and MOE occurred at 5 and 25 min, respectively. The IB of the boards was higher at 10 and 15 min and then decreased with prolonged pressing duration (Fig. 2b). Within a certain range of hot pressing duration, heat transmitted from the surface of the mat to the center and the cotton stalks deformed with increasing pressing duration. The adhesive also needed enough time to cure to bind the stalks together (Prasittisopin and Li 2010). Therefore, the MOR, MOE, and IB increased with the increasing pressing duration. However, the adhesive could decompose under the cooperation of high temperature and long pressing duration, and lose its bonding prosperity (D'Amico *et al.* 2010; Júnior *et al.* 2011; Zanetti *et al.* 2014), leading to a reduction of the mechanical properties of OCB. Therefore, the mechanical properties decreased when the pressing duration was too long. According to the results in Fig. 2a and Fig. 2b, three levels (10, 15, and 20 min) of hot pressing duration were selected for orthogonal experiment to replace levels 1, 2, and 3 of pressing duration (Table 2).

Figures 2c and 2d show that the MOE and MOR of the boards were higher at 130, 150, and 170 °C and the IB of the boards were higher at 130 and 150 °C. As the hot pressing temperature increased, the adhesive cured, and the stalks deformed (Barcikowski *et al.* 2006). Studies show that hemicellulose, cellulose, and lignin are the primary material affecting the MOE and MOR of the boards. When the temperature reaches 140 °C with enough time, hemicellulose begins to degrade, whereas cellulose and lignin are less affected under such temperature, mainly due to their different structures and components (Liu *et al.* 2009). Hemicellulose is the primary reason that the board absorbs water, so lowering hemicellulose content may improve the thickness swelling and IB of the board (Yildiz *et al.* 2013). Therefore the values of MOE, MOR, and IB increased with increasing hot pressing temperature. However, the adhesive could cure excessively if hot pressing temperature is too high. In addition, the high pressing temperature is related to degradation of cotton stalks and can decrease the values of mechanical properties of OCB (Pizzi and Cameron 1981; D'Amico *et al.* 2010; Lei *et al.* 2014). Therefore, temperatures of 130, 150, and 170 °C were therefore chosen for orthogonal experiment to replace levels 1, 2, and 3 of pressing temperature in Table 2.

The MOE and MOR increased continually with increasing board target density (Fig. 2e). The structure of OCB became more compact when the board density increased, allowing them to withstand higher loads. Thus, the mechanical properties of the boards were improved when the board target density increased. The IB reached its highest value at 0.7 g/cm³ density and then decreased slightly with increasing board target density. When the density reached 0.8 g/cm³, the MOE and MOR curves became smooth and the speed of increase became slow. With the density increasing, the needed pressing pressure on the board increased, leading to a larger internal stress and a higher spring-back (Wong *et al.* 1999). This might destroy the adhesive line structure in the inner part of the board and weaken the adhesion between the stalks (Deppe and Schmidt 1986; Varga *et al.* 2004). Furthermore, higher density requires greater requirements for the pressing machine (Xu *et al.* 2004). Above all, three levels (0.6, 0.7, and 0.8 g/cm³) of board target density were selected for orthogonal experiment to replace levels 1, 2, and 3 of board target density in Table 2.

Optimization of the Pressing Parameters

In order to optimize the pressing process, the results of the single-factor experiments were used to select appropriate pressing parameters for the design of the orthogonal experiment (Cai *et al.* 2011; Wei *et al.* 2013).

Table 3. Results of Orthogonal Experiments

Sample	Hot Pressing Duration (min)	Target Density (g/cm ³)	Hot Pressing Temperature (°C)	MOE (GPa)	MOR (MPa)	IB (MPa)
T1	10	0.6	130	5.86 (0.55)	32.08 (2.56)	0.38 (0.034)
T2	10	0.7	150	8.03 (0.43)	38.96 (3.64)	0.42 (0.033)
T3	10	0.8	170	8.35 (0.45)	40.19 (4.01)	0.40 (0.030)
T4	15	0.6	170	6.38 (0.46)	34.70 (2.27)	0.34 (0.022)
T5	15	0.7	130	7.32 (0.68)	39.80 (3.28)	0.46 (0.041)
T6	15	0.8	150	9.85 (0.94)	48.13 (3.78)	0.48 (0.037)
T7	20	0.6	150	7.30 (0.65)	35.41 (2.20)	0.37 (0.023)
T8	20	0.7	170	7.25 (0.56)	35.18 (2.71)	0.37 (0.030)
T9	20	0.8	130	8.55 (0.43)	41.73 (2.74)	0.47 (0.046)
K1 ^a	7.41 (0.623)	6.513 (0.466)	7.246 (0.619)			
K2 ^a	7.852 (0.432)	7.533 (0.731)	8.39 (0.767)			
K3 ^a	7.7 (0.746)	8.916 (0.565)	7.327 (0.482)			
R ^a	0.442 (0.039)	2.403 (0.224)	1.144 (0.112)			
K1 ^b	37.076 (2.872)	34.062 (2.567)	37.867 (3.403)			
K2 ^b	40.878 (2.54)	37.982 (2.226)	40.835 (2.176)			
K3 ^b	37.44 (3.014)	43.35 (2.94)	36.691 (2.401)			
R ^b	3.802 (0.297)	9.288 (0.632)	4.144 (0.302)			
K1 ^c	0.399 (0.027)	0.365 (0.025)	0.439 (0.034)			
K2 ^c	0.425 (0.035)	0.416 (0.022)	0.423 (0.032)			
K3 ^c	0.406 (0.027)	0.449 (0.04)	0.368 (0.029)			
R ^c	0.026 (0.002)	0.084 (0.006)	0.071 (0.004)			

(ⁱ): Values in parentheses are the standard deviation. K_i is obtained by adding any number of columns corresponding to ⁱ level. R_i is the difference between the maximum value and the minimum value of K_i of any columns. ^a MOE; ^b MOR; ^c IB

The arrangements and results of the orthogonal experiment are shown in Table 3. Results showed that the board target density had the largest impact on the MOE, MOR, and IB, followed by hot pressing temperature and hot pressing duration. The optimal pressing parameters for the mechanical properties of OCB were at 150 °C for 15 min with a target density of 0.8 g/cm³.

A verification test was run with these parameters, and the results showed that the MOE, MOR, and IB were 8.745 GPa, 46.264 MPa, and 0.485 MPa, respectively. These parameters are similar with those from previous studies of OCB composites (Li *et al.* 2003; Song *et al.* 2008; Qi *et al.* 2012; Hou *et al.* 2014).

The cost of KCP blend adhesive was analyzed by Gu (2010), who found that the cost was half that of white latex, comparable to that of UF resins, and one third that of pMDI. Although the MOE, MOR, and IB of OCB from KCP blend adhesive cannot compare with those of pMDI (Chauhan *et al.* 2014), the mechanical properties of OCB are comparable with those of PF and UF (Ye *et al.* 2007). Most importantly, KCP blend adhesive is environmentally friendly, with no harm to the environment and peoples' health.

The physical properties of OCB did not meet ASTM D1307-06a (2006) standards (the WA reached 30% to 60% and the TS reached 25% to 55%), but could be improved by treatment with cotton stalks in later research, such as using wax or heat treatment (Qi *et al.* 2012).

Morphological Structure of the Stalk and the Adhesion Surface

Morphological structure observations could explain some of the mechanisms of adhesion. The morphological structures acquired by SEM of cotton stalks are shown in Fig. 3. Figure 3a illustrates cotton stalk structure before pressing. The cell wall was intact and the pores naturally open.

Obviously, the cell wall was crushed and the pores were closed after pressing (Fig. 3b). This could indicate that the density of the board increased as well as the board density, MOE, and MOR increased under the effect of pressing pressure (Wang and Cooper 2005; Nayeri *et al.* 2014). It was also observed that adhesives filled the cell wall and lumen, creating mechanical interlocking between the adhesive and the stalks, as reported by Münchow *et al.* (2013).

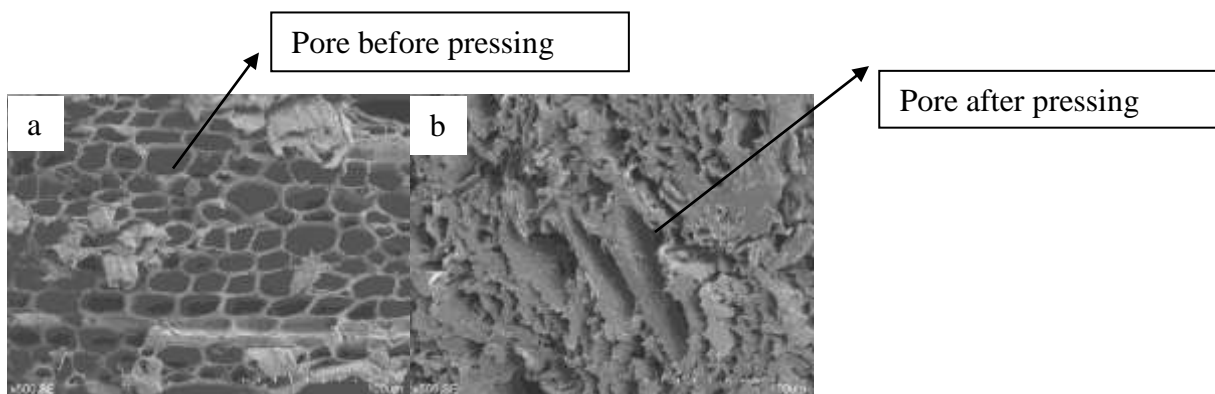


Fig. 3. SEM micrograph of OCB: (a) cotton stalk before pressing and (b) OCB after pressing

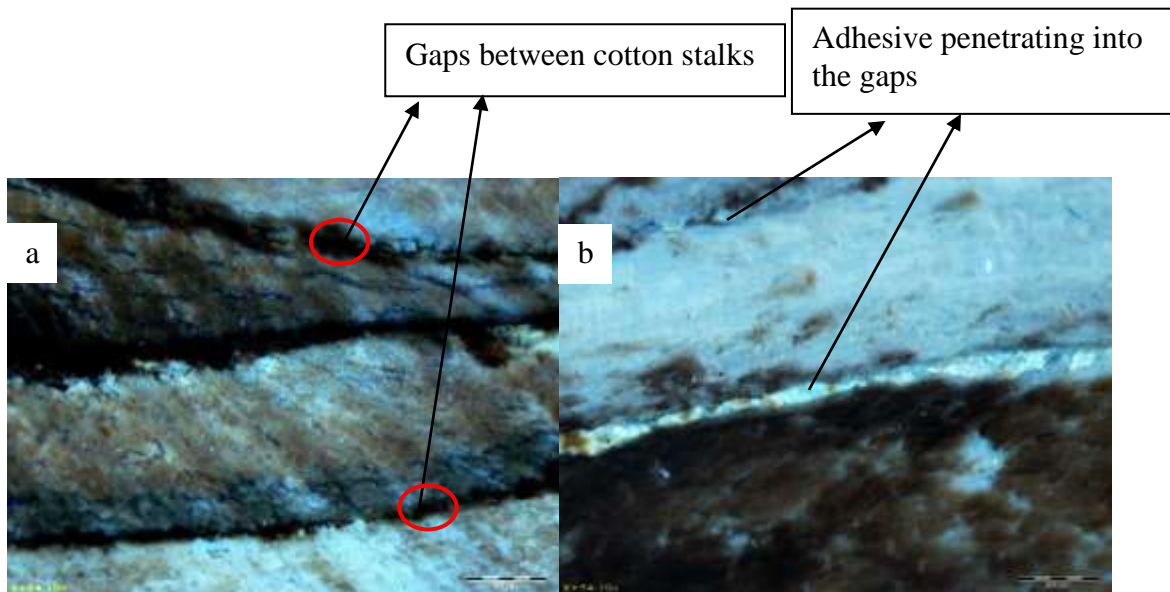


Fig. 4. Fluorescence microscope photos of OCB: (a) without adhesive and (b) with adhesive

Fluorescence microscope photos of the composite are shown in Fig. 4. Figure 4a shows the OCB without adhesive. The gaps between the stalks are evident. When the adhesives were glued onto the stalks and pressed into the OCB (Fig. 4b), they attached to the surface of the stalks and filled the gaps between stalks. This could improve the IB, MOE, and MOR of the OCB (Khristova *et al.* 1996; Guntekin and Karakus 2008). However, the adhesive coating was not uniform, and the adhesive did not penetrate deeply into the stalks. This may be caused by the high viscosity of the KCP blend adhesive, which made the penetration of the adhesive into the gaps between stalks difficult. Improved penetration of KCP blend adhesive could have a positive effect on mechanical interlocking between stalks, meanwhile improving physical properties of the boards. On the other hand, decreasing the viscosity of the adhesive and hot treatment of the stalks could have positive effects on penetration of the adhesive (Zhang *et al.* 1997; Gong *et al.* 2010).

CONCLUSIONS

1. The optimal pressing parameters for OCB were at a pressing temperature of 150 °C, for 15 min, and with board target density of 0.8 g/cm³. The values of the MOE, MOR, and IB using these parameters were 8.745 GPa, 46.264 MPa, and 0.485 MPa, respectively.
2. The scanning electron microscope and fluorescence microscope observations indicated the positive effects of pressing pressure and resin content on interfacial bonding between cotton stalk bundles.
3. This work offers a promising solution to a significant ecological problem by converting biomass into a value-added product suitable for furniture manufacturing and interior decoration with less stringent requirements for water resistance.

ACKNOWLEDGEMENTS

The authors appreciate the financial assistance from the special fund for agro-scientific research in the public interest of the Chinese Ministry of Agriculture (Grant No. 201003063-07).

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Article submitted: February 6, 2015; Peer review completed: April 5, 2015; Revisions received and accepted: April 28, 2015; Published: April 30, 2015.

DOI: 10.15376/biores.10.2.3736-3748