

# Dry Bond Strength and Water Resistance of Konjac Glucomannan, Chitosan, and Polyvinyl Alcohol Blend Adhesive

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An environmentally-friendly wood adhesive was developed by blending konjac glucomannan (KGM), chitosan (CH), and polyvinyl alcohol (PVA) together. The viscosity of the KGM-CH-PVA (KCP) blend adhesive was determined, and the morphology of the film was observed using scanning electron microscopy (SEM). The KCP blend adhesive was applied to plywood during the manufacturing process, and the effects of the KGM, CH, and PVA contents on the bond strength was investigated. Results showed that KGM greatly increased the viscosity of the KCP blend adhesive, whereas the addition of PVA decreased the viscosity in the test range. The SEM observations showed that the KCP blend adhesive was homogeneous. The bond strength of the plywood that was treated with KCP blend adhesive increased with increasing KGM and CH concentrations, and desirable performance could be obtained with a total solids content of 4.6%. The KCP blend adhesive with 2.0% KGM, 2.0% CH, and 0.6% PVA exhibited a comparable bond strength with phenol formaldehyde. Findings suggest that the KCP blend adhesive can be used as a wood adhesive with all raw materials, having the advantage of being environmentally friendly.

*Keywords:* Konjac glucomannan; Chitosan; Polyvinyl alcohol; Wood adhesive; Viscosity; Dry bond strength; Water resistance

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## INTRODUCTION

Synthetic resin adhesives, such as urea formaldehyde (UF) and phenol formaldehyde (PF), have been widely used in industry because of their many advantages, such as high bonding strength and high water resistance (Moubarik *et al.* 2009). Most synthetic adhesives contain formaldehyde, which is a chemical agent that causes environmental problems and is a threat to public health (Girods *et al.* 2009). The increasing concerns over environmental pollution and public health protection have forced the wood industry to develop environmentally-friendly wood adhesives (Liu and Li 2007; Liu *et al.* 2014; Xia *et al.* 2015). Polymeric 4,4'-diphenylmethane diisocyanate (pMDI) is an environmentally-friendly wood adhesive that contains no formaldehyde and has a high bonding strength and resistance to water (Sonnenschein *et al.* 2005; Qi *et al.* 2012). However, the moisture content of wood significantly affects the reaction between the wood and pMDI, and pMDI is expensive, which reduces its potential applications in industry (He and Yan 2007). Natural polymers, such as casein and soybean protein, were used as wood adhesives until they were replaced by synthetic resin adhesives a few years

prior. Synthetic resin adhesives replaced natural polymer adhesives because of issues concerning low durability and water resistance (Narayanamurti 1956). To date, studies on new, natural, polymer-based adhesives are necessary for the development of wood adhesives for the wood industry. Protein, starch, tannin, lignin, and polysaccharides are examples of interesting bio-based polymers that have been suggested as good candidates for the development of new adhesives and binders (Roffael 2006; Pandey *et al.* 2011; Amini *et al.* 2013).

Konjac glucomannan (KGM), a polysaccharide, is extracted from the tubers of the amorphophallus Konjac tree (Liu *et al.* 2010; Wang *et al.* 2015). Konjac glucomannan is an attractive candidate for the preparation of many materials, including composite materials, biodegradable films, controlled-release matrix medicine, and cosmetics (Huang *et al.* 2015; Wang *et al.* 2015). In addition, KGM was used during World War Two as a paste for paper, and it has been more recently used as an ingredient of wood adhesives (Chen *et al.* 2003; Umemura *et al.* 2003; Chen *et al.* 2015; Shang *et al.* 2015).

Chitosan (CH) is a deacetylated product of chitin, which is a naturally occurring polysaccharide available in large quantities from the epidermis of crustaceans, such as crab and shrimp (Lagarto *et al.* 2015). Current applications of chitosan are usually concentrated in biomedical fields, such as tissue engineering, gene vectors, and drug carriers (Kumar 2000; Rabea *et al.* 2003). However, an increase in the commercial availability of chitosan has opened the door to the development of materials in the film, binder, and adhesive fields (Cuy *et al.* 2003; Fu *et al.* 2005). The structure and adhesion mechanism of chitosan were reviewed by Mati-Baouche *et al.* (2014).

Polyvinyl alcohol (PVA) is a synthetic polymer that is used in a wide range of commercial, medical, and food applications (Choi *et al.* 2012; Muppalla *et al.* 2014; Kim *et al.* 2015). In addition, polyvinyl alcohol is frequently used as an adhesive and a sizing agent in textiles (Harazono and Nakamura 2005). Recently, polyvinyl alcohol was applied for wood preservation against fungal decay and termites, and for improving wood anti-swelling properties (Mohareb *et al.* 2011; Basta *et al.* 2013; Liu *et al.* 2013).

Blending is an effective approach for developing new adhesives. Generally, when the individual components used to blend are compatible, the blend can form a homogeneous structure and exhibit better physicochemical properties than the individual components themselves (Gao *et al.* 2008; Moubarik *et al.* 2013). Konjac glucomannan and chitosan were blended to form a new, natural, polymer-based wood adhesive (Umemura *et al.* 2003). In a previous study, an investigation of the rheological properties of konjac glucomannan, chitosan, and polyvinyl alcohol (KCP) blend adhesive was conducted (Gu *et al.* 2014), and the KCP blend adhesive was applied for plywood fabrication. However, the viscosity and cost of KCP was too high, which resulted in limitations with its use (Chen *et al.* 2015). The objective of this study was to develop and test KCP blend adhesive with a low viscosity in order to expand its applications in the wood industry. The effects of KGM, CH, and PVA content on the dry bond strength and wet bond strength of KCP blend adhesive was investigated.

## EXPERIMENTAL

### Materials

Konjac glucomannan (KGM) with a molecular weight of 256,000 and a glucomannan content of 97% was purchased from Qindong Konjac Food Co., Ltd.

(Shaanxi, China). The color of KGM flour was white to gray and the viscosity of 1.0 wt% aqueous solution was 20 Pa·s (25 °C).

Chitosan (CH), with a molecular weight of 213,000 and a deacetylation degree of 90 to 95%, was obtained from the Golden-Shell Biochemical Co., Ltd. (Zhejiang, China). The 1.0 wt% chitosan was dissolved in a 1.0 wt% acetic acid solution (30 mPa·s) at 20 °C. The color of the powder was almost white.

Polyvinyl alcohol (PVA) that was 98% acetylated and had a molecular weight of 14,000, as well as glycerol were obtained from the Tianjin Kemiou Chemical Reagent Co., Ltd. (Tianjin, China). All of the above reagents were used without further purification. The representative wood adhesives used here were phenol formaldehyde (PF) and polymeric 4,4'-diphenylmethane diisocyanate (pMDI). The PF and pMDI resins were obtained from Bayer AG (Leverkusen, Germany). The solids content and viscosity of pMDI resin were 100% and 1500 mPa·s at 25 °C, respectively, while those of PF resin were 52% and 1200 mPa·s at 25 °C, respectively.

Poplar rotary-cut veneer with a thickness of 1.5 mm was manufactured in the laboratory by a wood veneer lathe.

### The Formulations of Tested KCP Blend Adhesives

The preparation of KGM-CH-PVA blend adhesive was as follows (Chen *et al.* 2015). Chitosan and konjac glucomannan powders were weighed and dissolved in distilled water, sequentially, and the mixture was stirred at room temperature with a mechanical stirring machine. Glacial acetic acid was added while stirring. Polyvinyl alcohol was weighed and dissolved in distilled water to 10% w/w concentration at 95 °C. Then, the aqueous polyvinyl alcohol solution was added to the mixture. The adhesive was then stirred to obtain a clear, homogeneous polymer solution.

The formulations for investigating the effects of KGM, CH, and PVA content on the mechanical properties of the wood are shown in Table 1. In order to study the influence of the KGM content on the bonding strength, fixed levels of CH (2.0%) and PVA (0.6%) were used, and the KGM level varied from 0% to 2.5% using 0.5% intervals. The labels, K1 to K6 stood for the different levels of KGM contents. Similarly, CH1 to CH6 stood for the different levels of CH and P1 to P6 stood for the different levels of PVA. For PVA, KGM and CH were both fixed at 1.0% in order to lower the effect of KGM and CH, as shown in Table 1.

The experimental design of investigating the effects of the solids content on the KCP blend adhesive bonding strength is shown in Table 2. For all of the formulations, the ratio of KGM and CH was fixed as 1:1 (Jia *et al.* 2009; Gu *et al.* 2010), and the total solids content (including KGM content, CH content and PVA content) was varied from 1.6% to 6.6%. The different solids contents were identified as R1 to R6. Table 3 shows the comparison of the different KCP blend adhesives with the commercial PF, and pMDI resins. “CO1” to “CO8” were used to stand for the different prepared and commercial adhesives as shown in Table 3.

Three-layer plywood with the dimensions of 15×15 cm<sup>2</sup> and a moisture content of 10% was prepared using rotary-peeled polar veneer, as specified by Chen *et al.* (2015). The KCP adhesive was applied to the core veneer at a spread rate of 200 g/m<sup>2</sup> (Umemura *et al.* 2003) using a rubber roller. The PF resin and pMDI glue were also applied to the core veneer at a spread rate of 140 g/m<sup>2</sup> and 80 g/m<sup>2</sup>, respectively (Umemura *et al.* 2003).

**Table 1.** The KGM, CH, and PVA Contents of the KCP Blend Adhesive

Samples	KGM (%)	CH (%)	PVA (%)	Spread rate (g/m <sup>2</sup> )
K1-K6	0, 0.5, 1.0, 1.5, 2.0, 2.5	2.0	0.6	200
CH1-CH6	2.0	0, 0.5, 1.0, 1.5, 2.0, 2.5	0.6	200
P1-P6	1.0	1.0	0, 0.2, 0.4, 0.6, 0.8, 1.0	200

**Table 2.** Effects of the Solids Content on the Bond Properties of KCP Blend Adhesive

Samples	KGM (%)	CH (%)	PVA (%)	Spread rate (g/m <sup>2</sup> )
R1	0.5	0.5	0.6	200
R2	1.0	1.0	0.6	200
R3	1.5	1.5	0.6	200
R4	2.0	2.0	0.6	200
R5	2.5	2.5	0.6	200
R6	3.0	3.0	0.6	200

**Table 3.** Comparison of KGM, CH, and PVA Adhesives with Commercial PF and pMDI Resins

Samples	KGM (%)	CH (%)	PVA (%)	PF	pMDI	Spread rate (g/m <sup>2</sup> )
CO1	2.0	0	0	no	no	200
CO2	0	2.0	0	no	no	200
CO3	2.0	2.0	0	no	no	200
CO4	2.0	0	0.6	no	no	200
CO5	0	2.0	0.6	no	no	200
CO6	2.0	2.0	0.6	no	no	200
CO7	0	0	0	yes	no	140
CO8	0	0	0	no	yes	80

### Plywood Fabrication and Pressing Conditions

The KCP blend adhesive was added to previously assembled three-ply veneers that were cold-pressed under a pressure of 0.98 MPa for 2 h (to eliminate the inner force of the veneer) and then hot-pressed at 130 °C at 3.0 MPa for 15 min (Gu *et al.* 2010).

The UF resin adhesive was added to previously assembled three-ply veneers that were cold-pressed at a pressure of 0.98 MPa for 30 min and then hot-pressed at 150 °C at 3.0 MPa for 10 min. To prevent blistering, degassing was done after a hot-pressing time of 5 min. When the pMDI glue was applied, the assembled three-ply veneers were cold-pressed at 0.98 MPa for 10 min and then hot-pressed at 170 °C at 3.0 MPa for 10 min. Degassing was also done after hot-pressing.

### Bond Strength Measurements

Conditioning was achieved at a temperature of 20 °C and a humidity of 65% for a minimum of 7 days. The plywood panels were cut into specimens with the dimensions of 10 × 2.5 cm<sup>2</sup> to determine the bond strength and water resistance, according to the China National Standard (GB/T 17657 2013). Each specimen had a bonded area of 2.5 × 2.5 cm<sup>2</sup>. Both dry and wet bond strengths were tested using a SANS testing machine (MTS Systems Corp., Shenzhen, China), with a loading rate of 5 mm/min. At least 10 replicates were used for each test. For wet bonding strength, the specimens were soaked in 63 ± 3 °C water for 3 h and then dried at room temperature for 10 min before testing. The mean values and standard deviations of the bonding strengths and the average wood failures were calculated for each sample. The wood failure measurements were carried out in accordance with the China National Standard (GB/T 17657 2013).

### Viscosity Measurements

The viscosity of the adhesives were measured using a NDJ-5S rotary viscometer (Geological Instrument Co., Ltd., Shanghai, China). The spindle heads (1#, 2#, and 3#) were wiped with distilled water to prevent contamination from the adhesive that was tested. Testing was carried out with the spindle head sticking into the adhesive for 5 min of spinning at room temperature. Five readings were recorded, and the average viscosity was calculated.

### SEM Observation

Solutions of KGM, CH, KGM/CH blend, and KCP were prepared, and sample solution was added onto the slide by 0.5 g/cm<sup>2</sup>. Then, the slides were oven-dried at 60 °C until a consistent weight was obtained (Huang *et al.* 2015). The film was scraped using a knife and cut into the dimensions of approximately 0.5 × 0.5 cm<sup>2</sup>. Then, the films were coated with gold powder, and the surface morphologies were observed using a scanning electron microscope (SEM) (Model S2400, Hitachi Global, Ltd., Tokyo, Japan) at an acceleration voltage of 5 kV.

### Statistical Analysis

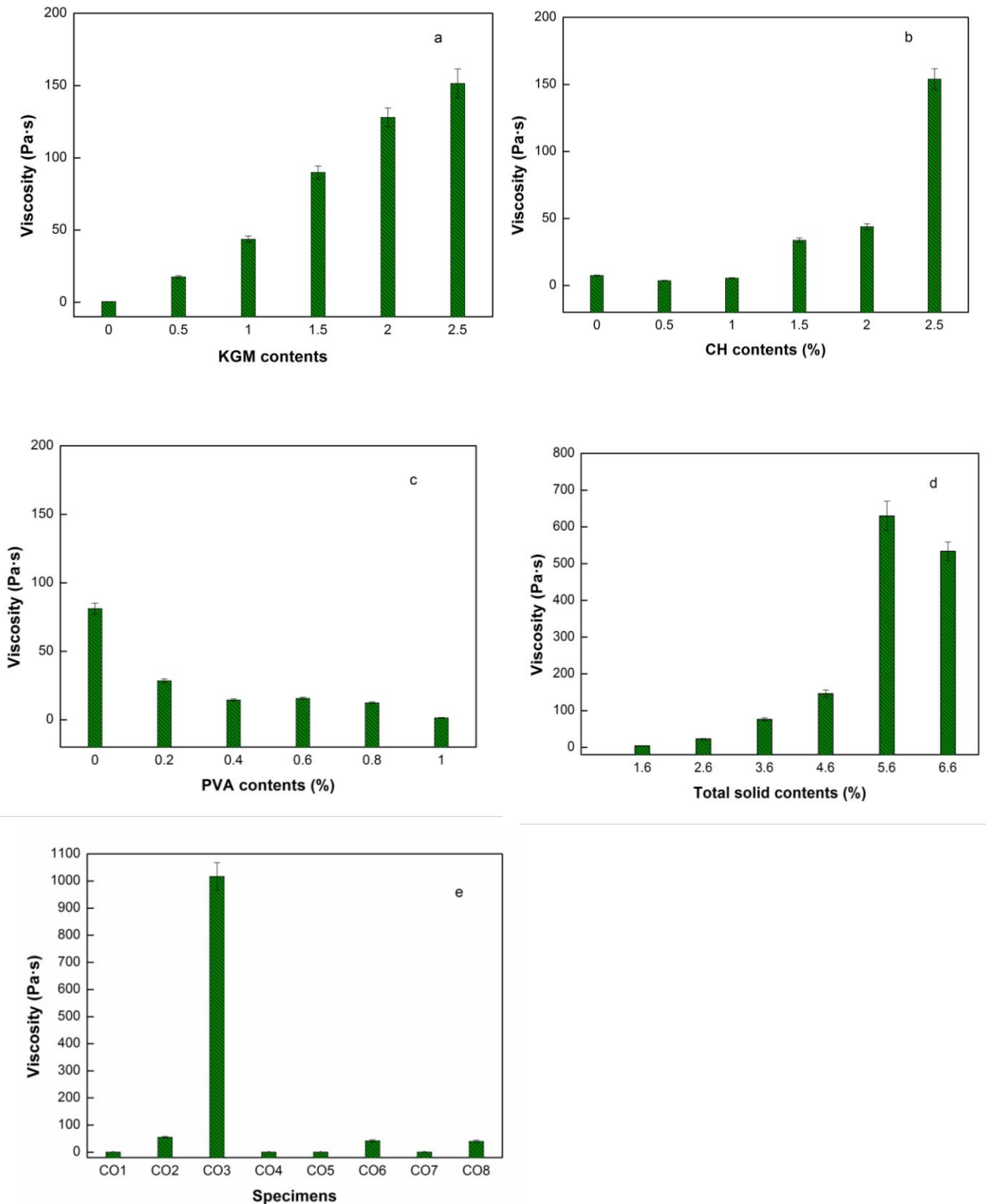
Data from each test were statistically analyzed using the Statistical Package for Social Sciences (SPSS version 13.0; IBM, USA) for Windows (Microsoft Corp., Redmond, WA). The student's t test was used to make comparisons between selected groups. A *P* value of <0.05 was considered significant. Figures were developed using the Origin Pro 8.5 software (Origin Lab Co., Northampton, MA, USA).

## RESULTS AND DISCUSSION

### Viscosities of the Various Adhesives and Adhesive Blends

Figure 1 shows the viscosities of various KCP blend adhesives. From Fig. 1a, the viscosities increased almost linearly as the quantity of KGM increased. Therefore, it can be concluded that the KGM favorably affected the viscosity of the KCP blend adhesives. The viscosity trends of the CH series adhesives are shown in Fig. 1b. The viscosities of this series changed slightly along with the CH content before reaching 2.5%. The PVA content may be responsible for decreasing the viscosity of the KCP blend adhesives, as shown in Fig. 1c.

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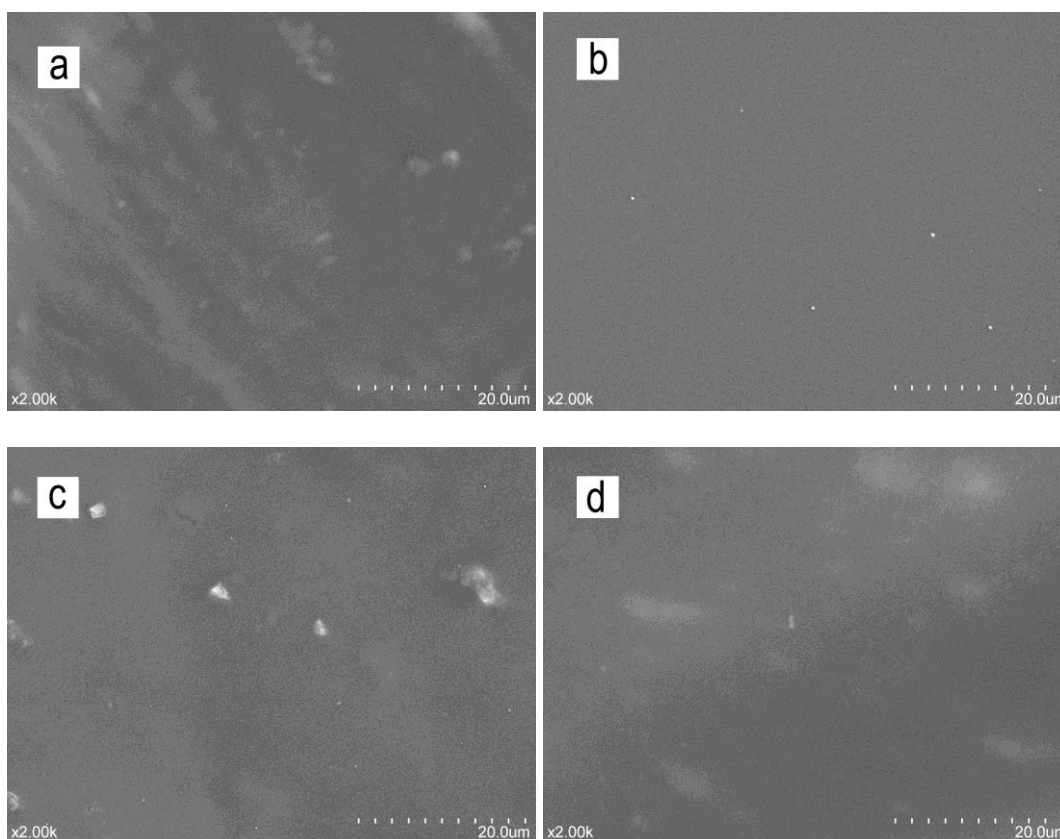
**Fig. 1.** Viscosities of various KCP blend adhesives, PF, and pMDI resins: (a) viscosities of various KGM series adhesives; (b) viscosities of various CH series adhesives; (c) viscosities of various PVA series adhesives; (d) viscosities of various solids content series adhesives; (e) viscosities of various KCP blend adhesives, PF, and pMDI adhesives

The viscosities of the KCP blend adhesives increased slightly when the total amount of KGM and CH was less than or equal to 5.6%, as shown in Fig. 1d. Figure 1e shows that the viscosity of the KGM and CH adhesive (CO3) was the highest. The viscosities of the KGM/PVA (CO4) and PVA adhesives (CO5) were lower than the KGM (CO1) and CH adhesives (CO2), respectively, which was attributed to the PVA content in the adhesives. The viscosity of the KCP blend adhesive (CO6) was higher than the PF resin (CO7) and was comparable with the pMDI resin (CO8). As a result, KCP blend adhesive could be sprayed by using the common spraying equipment.

### SEM Observations of the Film from the Adhesives

The results from the SEM images of the films are shown in Fig. 2(a-d). The film from the KGM adhesive (Fig. 2a) showed strips on the surface, which may have been caused by a shrinking effect when the KGM was heated. A homogeneous background of beads was exhibited in Fig. 2b (CH adhesive) and Fig. 2c (KGM and CH adhesive). The film from the KCP blend adhesive (Fig. 2d) exhibited a smooth and homogeneous surface morphology, suggesting high miscibility and blend homogeneity between the KGM, CH, and PVA adhesives.

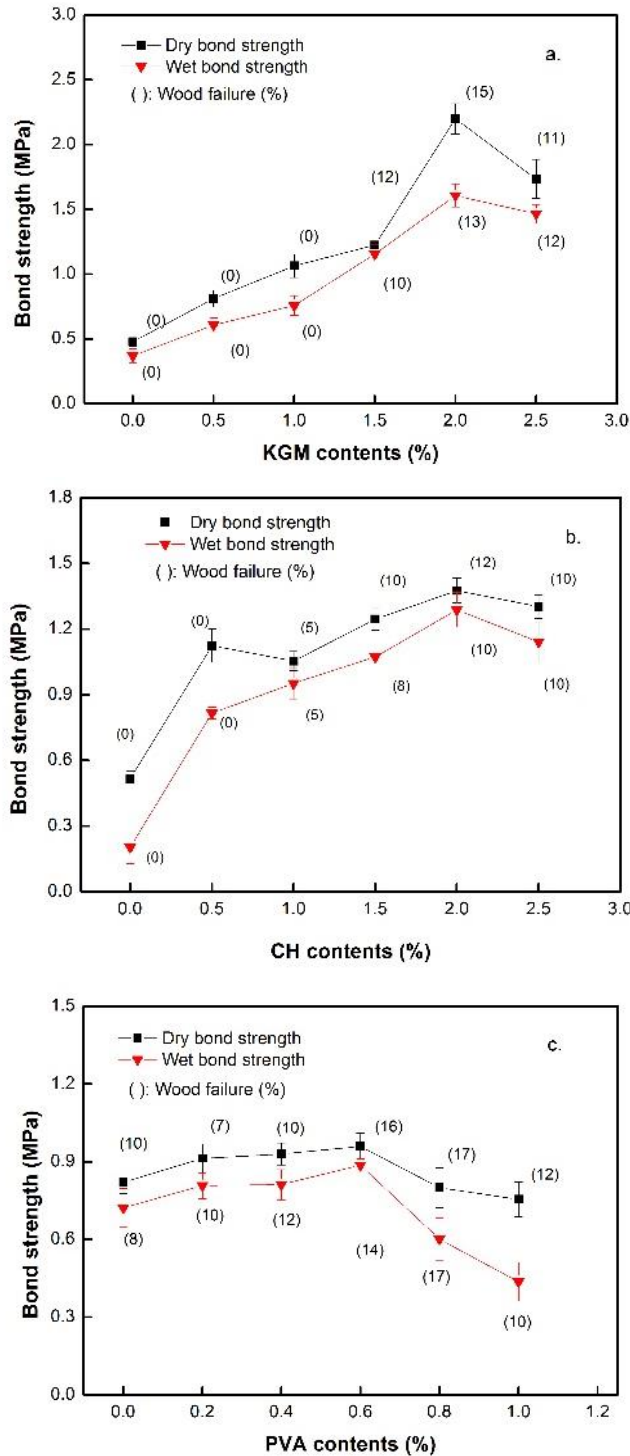
Favorable miscibility and hydrogen bonding between the KGM, CH, and PVA may have resulted in an enhancement of the dry and wet bond strengths, which was verified by previous research (Umemura *et al.* 2003).



**Fig. 2.** SEM observations of various KCP blend adhesives: a) film from KGM adhesive; b) film from CH adhesive; c) film from KGM/CH adhesives; d) film from KCP blend adhesive

### Effects of the KGM, CH, and PVA Contents on the Properties of KCP Blend Adhesive

Figure 3a displays the bond strength of plywood glued with adhesives of varying KGM concentrations. The CH and PVA levels were kept at a constant quantity and the KGM level varied from 0% to 2.5%, as shown in Table 1.



**Fig. 3.** Bond strengths of plywood glued with various KGM, CH, and PVA contents: a) KGM; b) CH; and c) PVA



Bond strengths of the plywood increased with increasing KGM up to approximately 2.0%, and then decreased slightly. The maximum average bond strengths were 2.2 MPa and 1.6 MPa for dry and wet conditions, respectively. This was likely because of the positive effect of KGM on bond strength, which was also reported by Andrés-Bello *et al.* (2013). However, with increasing KGM concentrations, the viscosity of the KGM blend adhesives increased significantly, which decreased the wettability of the adhesive to the veneer. As a result, the bond strength of the plywood decreased as the concentration of KGM increased. The wet bond strength decreased by approximately 27% compared to the dry bond strength at 2.0% KGM. The wood failure of the samples glued with these adhesives exhibited low values (10 to 15%).

Figure 3b shows the bond strength of the plywood glued with adhesives of varying CH concentrations. The KGM and PVA levels remained at a constant quantity, while the CH level varied from 0% to 2.5%, as shown in Table 1. The dry bond strength of the plywood increased significantly ( $P < 0.05$ ) as the CH concentration increased from 0% to 0.5%, then it increased slightly until the CH concentration was 2.0%, and then it decreased slightly. For the 2.0% CH level, the average bond strengths were 1.37 MPa and 1.28 MPa for dry and wet conditions, respectively. The positive effect of CH on the bond strength of blend adhesives was similar to results by Mati-Baouche *et al.* (2014). The wood failure of the samples glued with these adhesives exhibited low values (5% to 12%).

Figure 3c shows the bond strength of the plywood glued with adhesives of varying PVA concentrations. The KGM and CH levels were maintained at a constant quantity, and the PVA level ranged from 0% to 1.0%, as shown in Table 1. The bond strength of plywood increased slightly as the concentration of PVA increased, and then decreased beyond a certain point.

When the PVA concentration was 0.6%, the average bond strength reached peak values of 0.96 MPa and 0.88 MPa for dry and wet conditions, respectively, which was inferior to those of adhesives of various KGM and CH contents. The reason for this may have been the lower KGM and CH concentrations in the adhesives with varying PVA concentrations (1.0% KGM and 1.0% CH, as shown in Table 1). It was shown that PVA exhibited a positive effect on the bond strength, although this effect was not as pronounced compared to those of KGM and CH. The wood failure of the samples glued with these adhesives exhibited low values (8% to 16%).

### Effects of the Total Solids Content on the KCP Blend Adhesive

The total solids content is an important factor in the performance and application of wood adhesive (Li *et al.* 2004). Figure 4 shows the bond strength of plywood glued with various total solids contents. The bond strength of the plywood increased with the increased total solids content. For a total solids content of 4.6% (KGM 2.0%, CH 2.0%, and PVA 0.6%), the average bond strengths were 1.49 MPa and 1.44 MPa for dry and wet conditions, respectively. Likewise, plywood with a total solids content of 6.6% (KGM 3.0%, CH 3.0%, and PVA 0.6%) exhibited average bond strengths of 1.68 MPa and 1.58 MPa for dry and wet, respectively, which increased by 12.7% and 9.7% when compared to plywood with a 4.6% total solids content. It was speculated that the bonding strengths of specimens increased because of increasing hydrogen bond strength between the CH and KGM adhesives, which was also reported by Li *et al.* (2006). The wood failure of the specimens manufactured with a total solids content of 4.6% were 35% and 32% for dry and wet conditions, respectively. And the wood failure of the specimens with

6.6% total solids content was 38% for dry condition, increasing by only 8.5% compared with that of solids content of 4.6%. The wood failure of the specimens with 6.6% total solids content was 30% for wet condition, with decreasing by 6.6% compared with that of solids content of 4.6%.

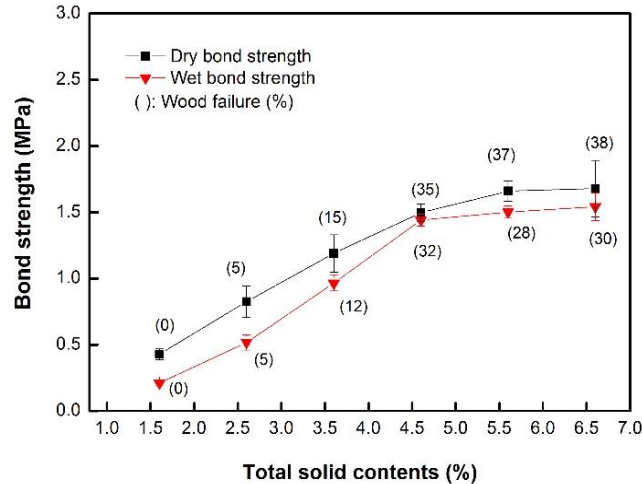


Fig. 4. Bond strengths of plywood glued with various total solids contents

### Various KCP Blend Adhesives Compared with PF and pMDI Resins

Figure 5 shows the bond strengths of specimens glued with various KCP blend adhesives, PF, and pMDI resins. The average dry bond strength of KGM adhesive (CO1) was similar to that of KGM and PVA adhesive (CO4), with an average value of 1.37 MPa. The wet bond strength of CO4 was greater than that of CO1 because of the addition of polyvinyl alcohol. The polyvinyl alcohol had some influence on dry and wet bond strengths. This was also observed by contrasting the bond strength of CH adhesive (CO2) with that of CH and PVA adhesive (CO5), which showed that dry and wet bond strengths of CO5 were higher than CO2.

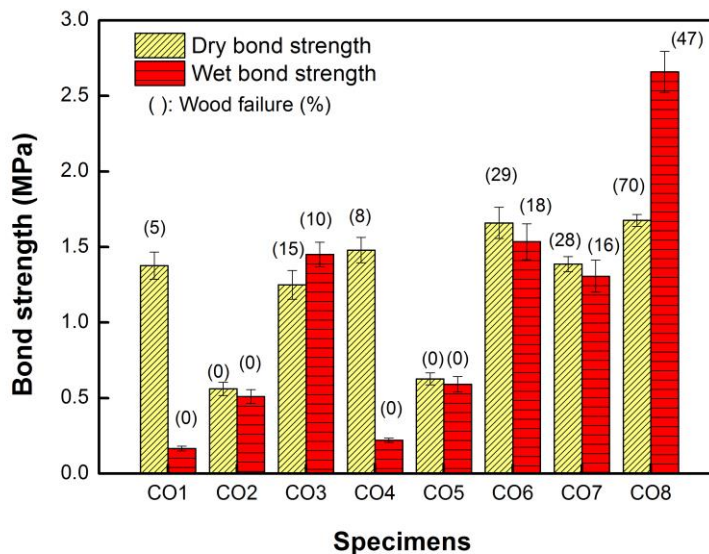


Fig. 5. Bond strengths of plywood glued with various KCP blend adhesives, PF, and pMDI resins

The dry bond strength of KGM/CH adhesive (CO3) was 1.25 MPa, which was similar to CO1. The wet bond strength of CO3 was notably greater than that of CO1. The main reason for this increase in bond strength was because CH exhibited a positive effect for improving the water resistance, which was also reported by Umemura *et al.* (2003). With contrasting the bond strengths of CO1 and CO2, it was found that the dry bond strength of CO1 was notably greater than CO2, and that the wet bond strength of CO2 was greater than CO1. It was determined that the dry bond strength of the KGM adhesive was greater than CH, and dry bond strength of CH adhesive is better than KGM.

The bond strength of the KCP blend adhesive (CO6) reached peak values of 1.77 MPa and 1.53 MPa for dry and wet conditions, respectively, that were greater than those of CO3 because of the quantity of PVA in the adhesive. The CO6 level had a higher quantity of PVA than CO4, and higher than those of CO5 on account of KGM. The bond strength of KCP blend adhesives has been reported in previous research (Gu *et al.* 2010). The KCP blend adhesives from a study by Gu *et al.* (2010) exhibited a bond strength of 3.2 MPa and 1.6 MPa for dry and wet conditions, respectively, with a spread rate of 800 g/m<sup>2</sup>. The decrease in the bonding strength of the KCP blend adhesive, as reported in this study, was likely because of the difference in the spread rates. The cost of the KCP blend adhesive was considerably lower than what was reported from Gu *et al.* (2010) because of a lower solids content of the adhesive and a lower spread rate. The bond strengths of KGM and CH composites reported by Umemura *et al.* (2003) were 2.1 MPa and 1.2 MPa for dry and wet conditions, respectively. This was mainly attributed to the different blending methods.

The dry and wet bond strengths of the KCP blend adhesive had advantages compared to PF resins (CO7), which have been commonly used in the furniture and decor industries. Because of formaldehyde release and its implications for public health, PF resins are oftentimes unsuitable, and interests in environmentally-friendly wood adhesives are of great interest. However, the properties of the KCP blend adhesive was inferior to those of pMDI resins (CO8); the pMDI resin exhibited more favorable bonding properties and was more environmentally friendly. Since pMDI resins are expensive, the applications of pMDI resins are limited in the industry. The adhesion of the KGM blend adhesive is assured by the penetration of the adhesive in the micro-cavities or pores of the cotton stalk surface before solidification. After the adhesive curing, the mechanical interlocking formed between the cotton stalks. The roughness and the asperities are favorable to the mechanical interlocking (Grujicic *et al.* 2008). The above can be regarded as the main mechanism for adhesion of the KGM blend adhesive. Besides, intermolecular forces and electrostatic attraction might also exist between the adhesive and the cotton stalks (Speranza *et al.* 2004; Nezu *et al.* 2005; Baji *et al.* 2015).

## CONCLUSIONS

1. The KGM had a significantly positive effect on dry bond strength of the KCP blend adhesive. The CH showed a distinctly positive effect on water resistance of the KCP blend adhesive. The PVA exhibited a positive effect on dry and wet bond strengths of the KCP blend adhesive to some extent.

2. The bond strength of the KCP blend adhesive was comparable to that of PF resins and could be used as a wood adhesive when conditions of water resistance are unnecessary.
3. The viscosity of the KCP blend adhesive could be lowered by reducing the concentration of KGM; in doing so, the viscosity notably improved. With this change, the application of KCP blend adhesives could be widely used in the wood industry.

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