Preparation of Particleboard Using Dialdehyde Starch and Corn Stalk

Peiyao Ye,[a] Junjian An,[a,b] Guangyan Zhang,[a,b] Lei Wang,[a,b] Peng Wang,[a,b,]* and Yimin Xie [a,b]

Particleboard was manufactured using dialdehyde starch (DAS) as the adhesive and corn stalk as the matrix. The adhesive was prepared *via* the oxidation of tapioca starch in sodium periodate. The DAS was characterized by Fourier transform infrared (FTIR) spectroscopy. The effect of the hot pressing temperature, board density, and DAS dosage on the physical properties of particleboard was studied. The cross-section morphology of particleboard was observed *via* scanning electron microscopy (SEM). The absorption peak of the aldehyde group appeared at 1731 cm^{-1} after the tapioca starch was oxidized by sodium periodate, indicating that DAS had formed. Both the modulus of rupture (MOR) and modulus of elasticity (MOE) increased first and then decreased, with the increase of hot pressing temperature, board density, and DAS dosage. Within a certain range, the increase of hot pressing temperature, board density, and DAS dosage reduced the thickness swelling (TS) and improved the particleboard water resistance. During the particleboard hot pressing process, DAS filled the spaces of the corn stalk and acted as an adhesive to bind the corn stalks tightly together, thereby improving the physical, mechanical, and water resistance properties of the particleboard.

Keywords: Dialdehyde starch; Corn stalk; Particleboard; Adhesive

*Contact information: a: School of Materials and Chemical Engineering, Hubei University of Technology, 430068, Wuhan, China; b: Hubei Provincial Key Laboratory of Green Materials for Light Industry, Hubei University of Technology, 430068, Wuhan, China; *Corresponding author: ahwp1234@163.com*

INTRODUCTION

With the current global fast growth of the particleboard industry, the development of new adhesives has become a major necessity. Currently, urea-formaldehyde (UF), phenol-formaldehyde (PF), and melamine-formaldehyde (MF) have been widely used in the production of particleboards due to their excellent adhesion property and low cost. However, these adhesives release harmful formaldehyde gases during their utilization, which can cause serious environmental and health problems (Amini *et al*. 2013; Salleh *et al*. 2015). Therefore, there is an urgent need to find a green formaldehyde-free adhesive to replace formaldehyde adhesives. To solve the problem of particleboard formaldehyde emission, two methods have recently been proposed. Firstly, formaldehyde-free chemical adhesives, such as isocyanates, have been introduced, which give excellent properties to particleboard, particularly in improving the water resistance and the mechanical properties (Deppe and Ernst 1971). However, isocyanates are expensive and have poor anti-aging properties (Banks 1995) that limit their application. Secondly, natural polymers, such as soy protein, lignin, tannin, and starch are used as raw materials to prepare biomass adhesives (Wang *et al*. 2012). These natural substances are abundant in nature and have great potential to be raw materials for preparing biomass adhesives.

Starch is a natural macromolecule that is widely present in all plants (Petersen *et al*. 1999; Wang *et al*. 2011). Starches extracted from different plants have different chemical structures, which are known as amylose and amylopectin. Amylose is composed of glucose units linked by α-(1-4) bonds. In addition to α-(1-4) bonds, amylopectin also contains 4.2% to 5.9% of α-(1-6) bonds on the branches (Temperini 2014). Due to the structural properties, starch as an adhesive has many problems in practical applications, such as low adhesive strength, poor water resistance, poor stability, high production cost, and the complicated production process. To solve these problems, extensive research has been conducted on the modification of starch (Subramanian and Natarajan 1989; Tharanathan 2005; Sulaiman *et al*. 2013; Wang *et al*. 2017). Nwokocha (2011) prepared a cyanoethyl starch adhesive with better adhesion performance by cyanoethylating cassava starch. However, the adhesive performance of the cyanoethyl starch was still insufficient in high humidity environments. Pang *et al*. (2016) used polyvinyl alcohol (PVA) solution as a tackifier, flour as a filler, and polyisocyanate (PMDI) as an enhancer to prepare a modified corn starch with excellent properties. Unfortunately, the isocyanate used in the raw materials is expensive, resulting in a high production cost that limited development.

Zhang *et al*. (2011) prepared a modified starch adhesive through the oxidation of starch with hydrogen peroxide in which many hydroxyl groups in starch were converted to carbonyl or carboxyl groups, and then, graft copolymerization occurred with the silane coupling agent and olefin monomer. The results showed that the adhesive strength and the water resistance of the starch adhesive obtained were significantly improved. The adhesive strength reached 7.88 MPa in the dry state and 4.09 MPa in the wet state. Therefore, high-performance starch adhesives with high adhesive strength and a high water resistance can be synthesized by oxidizing starch. During the oxidation of starch with sodium periodate, the hydroxyl groups of C_2 and C_3 on starch glucose units are oxidized to form aldehyde groups (Wongsagon *et al*. 2005; Fiedorowicz and Para 2006; Martucci and Ruseckaite 2010; Yu *et al*. 2010). The dialdehyde starch (DAS) obtained has greatly improved reactivity and a stronger binding strength. Currently, DAS has been used as a wet strength agent in the paper industry and as a tanning agent in the leather industry (Weakley *et al*. 2010; Yu *et al*. 2017). Korhonen *et al*. (2017) added a complex of DAS and chitosan to paper to strengthen the network structure of the wood fiber, therefore resulting in a significant increase in the wet strength of the paper. Mu *et al*. (2010) showed that the aldehyde groups in DAS can form cross-links with collagen, which causes the collagen to have a high stability at high temperatures and in enzyme reactions. It has been observed that DAS can form strong bonds with fibers due to the presence of a large number of aldehyde groups in the structure. Therefore, DAS can be used as a new type of adhesive for the preparation of formaldehyde-free particleboards.

The global production of waste crop stalks has been estimated to be approximately 8 billion tons/year (Lal 2005), and most of them are burned after harvest, causing serious environmental problems. However, the low cost and rich hemicellulose content of crop stalks make it an attractive potential substrate for binder-free board production. Studies have shown that crop stalks have great potential for producing high-quality particleboards (Salim *et al*. 2008). Therefore, all different crop stalks, such as rice straw, wheat straw, and corn stalk, can be used as raw materials for particleboard manufacturing (Halvarsson *et al*. 2009a, 2009b; Chen *et al*. 2018).

In the present study, sodium periodate was used to oxidize starch for the preparation of a DAS-based adhesive. Corn stalk was used as a raw material for producing formaldehyde-free particleboards with excellent mechanical and water resistant properties.

The effect of the hot-pressing process on the properties and the morphology of the particleboards was investigated.

EXPERIMENTAL

Materials

Tapioca starch was supplied by a factory in Guangxi Province, China. Sodium hydroxide, anhydrous ethanol, ethylene glycol, and 98% concentrated sulfuric acid were purchased from Sinopharm Chemical Reagent Co., Shanghai, China. Sodium periodate was purchased from Aladdin Biochemical Technology Co., Shanghai, China. Potassium bromide was purchased from Shanghai Macklin Biochemical Co., Shanghai, China. The potassium bromide was of spectral grade, and all other reagents were of analytical grade. Corn stalks were collected from a suburb of Mianyang City in Sichuan Province, China. The corn stalks were washed with deionized water and oven-dried at 50 ºC to a moisture content between 10% and 12%. The dried corn stalks were ground in a vegetation disintegrator and sieved through a 120-mesh screen. The ground samples were used for the experiment.

Methods

Preparation of dialdehyde starch

A sample weighing 2 g of dried tapioca starch and 100 mL of distilled water was added into a 250-mL three-necked round bottom flask. The flask was tightly wrapped with aluminum foil, placed in a water bath at a constant temperature of 40 ºC, and then flushed with nitrogen gas. Sodium periodate was added into the flask in a 1:1 mass ratio of sodium periodate to starch. The mixture was adjusted to a pH of 3 with 0.5 mol/L of sulfuric acid and was stirred for 4 h. At the end of the reaction, an appropriate amount of 0.1 mol/L ethylene glycol solution was added to remove the unreacted sodium periodate in the mixture. The mixture was then filtered and the filter cake was washed with plenty of distilled water followed by absolute ethanol to remove glycol. Finally, the obtained DAS was dried in a vacuum drying box at room temperature.

Characterization of dialdehyde starch by Fourier transform infrared (FTIR)

Approximately 1 mg of the dried DAS was milled with 200 mg of potassium bromide and then compressed for FTIR analysis. The infrared spectra was obtained with a Nicolet 6700 FTIR spectrophotometer (ThermoFisher Scientific Inc., Waltham, MA, USA). Each sample was scanned 32 times in a wavelength region of 4000 cm^{-1} to 400 cm^{-1} at a resolution of 4 cm^{-1}. Background spectra were collected before each experiment.

Preparation of particleboard

The corn stalk was mixed with the DAS in concentrations of 0, 4, 8, 12, 16, and 20%. After being air-dried to 12% moisture content, the mixture was manually made into a homogeneous mat of dimensions 220 mm × 200 mm by using a forming box. The formed mats were prepressed by hand and silicone paper was placed on the top and bottom surfaces of the mats. Then, the mats were transferred to a hot-press machine (BY302X2/15, Xinxieli Group Co., Ltd., Suzhou, China) and pressed under various conditions: hot pressing temperatures of 150, 160, 170, 180, and 190 ºC; hot pressing time of 10 min; hot pressing pressure of 3 MPa; and targeted densities of 0.9, 1.0, 1.1, and 1.2 g/cm^3. Distance bars that were 3-mm-thick were inserted between the hot platens during hot pressing.

Mechanical properties of particleboard

The modulus of rupture (MOR), the modulus of elasticity (MOE), and the thickness swelling (TS) of the particleboard were tested according to the China national standard GB/T 17657 (2013).

Scanning electron microscopy (SEM) analysis of particleboard

A SEM analysis was conducted by using a scanning electron microscope (Quanta-FEG 250, Hillsboro, America) to investigate the morphological changes of the fracture surface of particleboard. Before the analysis, the fracture surface of particleboard was mounted on cylindrical aluminum stubs with a double-sided adhesive tape and coated with a thin layer of gold. Photographs were taken at 1000× and 5000×.

RESULTS AND DISCUSSION

FTIR Analysis of Starch and DAS

Figure 1 shows the FTIR spectra of the native starch and the DAS. Characteristic peaks of the native starch were observed at 3436 cm^{-1} (O-H stretching and vibration), 2927 cm^{-1} (C-H asymmetrical stretching and vibration), and 1640 cm^{-1} (O-H vibration of physically adsorbed water).

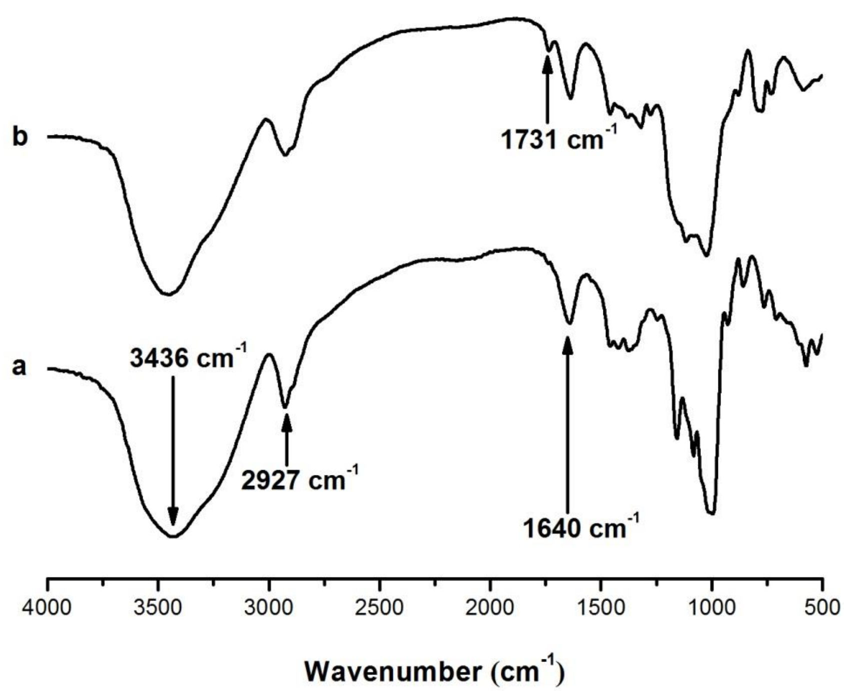

Fig. 1. FTIR spectra of (a) native starch and (b) DAS

The absorbances between 1000 cm^{-1} and 1200 cm^{-1} were assigned to the C-O stretching and vibration, and the peak below 1000 cm^{-1} was the absorption peak of C-C in the glucose ring of the starch (Landauer 1952; Nilsson and Bergenståhl 2007; Francl and Kingery 2015; Zuo *et al.* 2017). Compared to the FTIR spectra of the native starch, the DAS had an obvious absorption peak occurring at 1731 cm^{-1}, which was assigned to the characteristic peak of the C=O groups (Zhang *et al.* 2010). This indicated that the sodium periodate mainly broke down the C-OH bonds at the C_2 and C_3 positions of the anhydroglucose units to form aldehyde groups (Yu *et al*. 2010). Furthermore, the determination of the aldehyde groups showed that the native starch had many aldehyde groups after the selective oxidation with sodium periodate and the content of the aldehyde groups was 1979.57 μmol/g.

Effect of Hot Pressing Temperature on Physical Properties of Particleboards

Figures 2 and 3 showed the effects of hot pressing temperature on the MOR, MOE, and TS values of the particleboards. When the hot pressing temperature gradually increased from 150 ºC to 170 ºC, the MOR and MOE values gradually increased. The maximum MOR and MOE values, which were 15.3 MPa and 2168.5 MPa, respectively, were obtained at a hot pressing temperature of 170 ºC. The TS value decreased noticeably when the hot pressing temperature increased from 150 ºC to 170 ºC, and the minimum TS value was 28.3% at a hot pressing temperature of 170 ºC. This indicated that the water resistance of the particleboard gradually increased with the increase in hot pressing temperature.

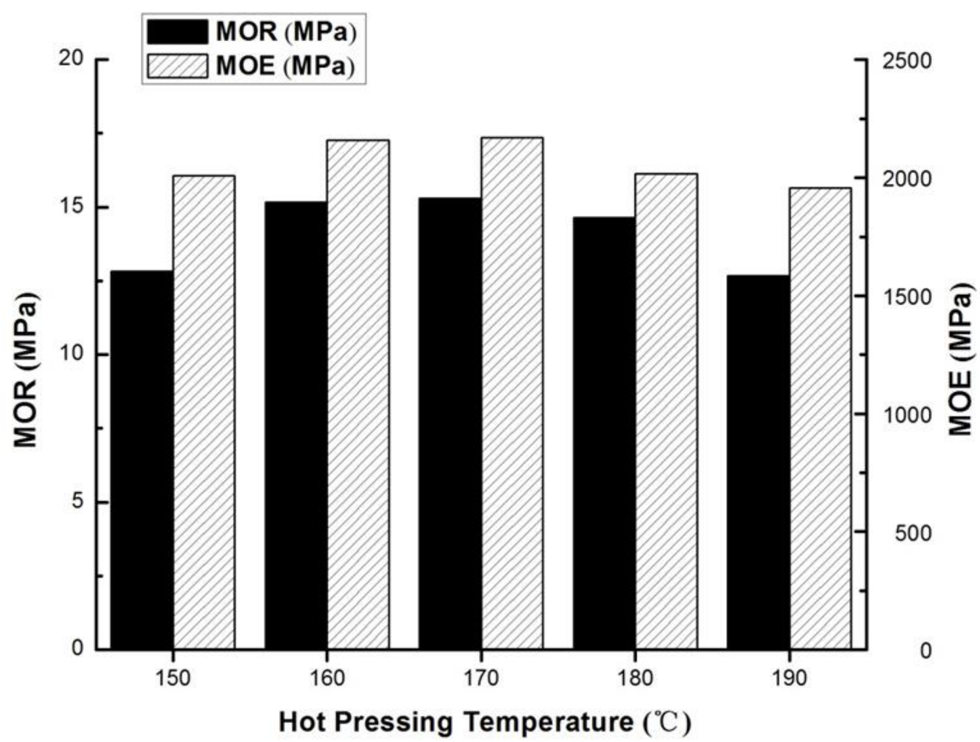

Fig. 2. Effects of hot pressing temperature on the MOR and MOE of the particleboard (Board density: 0.9 g/cm^3 and DAS dosage: 8%)

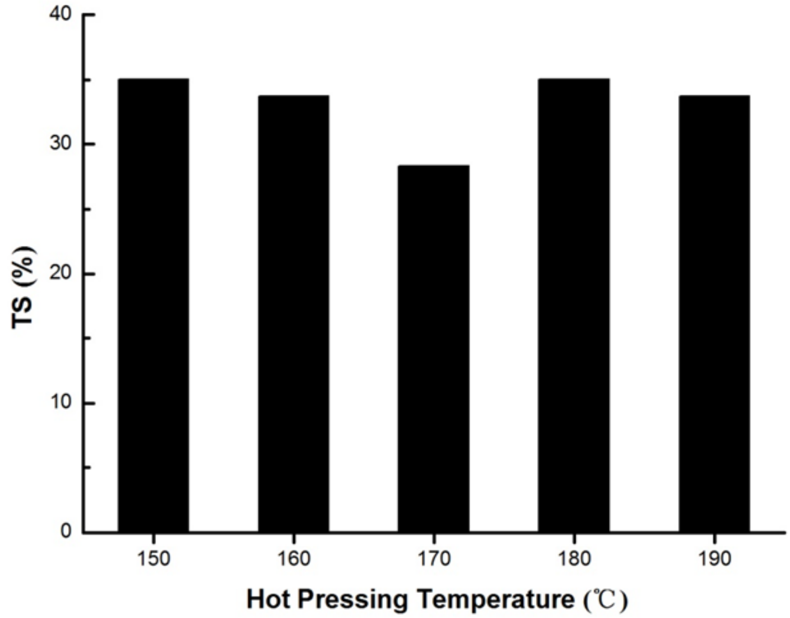

Fig. 3. Effects of hot pressing temperature on the TS of the particleboard (Board density: 0.9 g/cm^3 and DAS dosage: 8%)

The increase of the hot pressing temperature could favor the condensation reaction between the aldehyde groups on the DAS and the hydroxyl groups on the corn stalks to form acetal and/or hemiacetal structures, thereby improving the strength and water resistance of the particleboard (Zhang *et al*. 2015; Wang *et al*. 2016). When the hot pressing temperature was further increased from 170 ºC to 190 ºC, the MOR and MOE values gradually decreased and the TS value increased. This may have been due to the degradation of the main raw materials at high temperatures, which lead to a decline in the properties of the particleboard (Fu *et al*. 2015; Le *et al*. 2016). The results of the physical properties of the particleboards showed that the hot pressing temperature at 170 ºC was appropriate.

Effect of Board Density on Physical Properties of Particleboards

Board density has a strong influence on the properties of the particleboard (Laemsak and Okuma 2000). The effects of board density on the MOR, MOE, and TS were investigated when DAS was applied as an adhesive, as shown in Figs. 4 and 5. Firstly, the MOR and MOE values increased when the board density was increased from 0.9 g/cm^3 to 1.1 g/cm^3, and then it decreased noticeably when the board density was increased from 1.1 g/cm^3 to 1.2 g/cm^3. The maximum MOR and MOE values were 24.8 MPa and 5606.5 MPa, respectively. In this process, the TS value decreased first and then increased, and the minimum was at 11.6% when the board density was 1.1 g/cm^3. This was mainly because the spaces between the corn stalks and DAS were reduced with increased board density. Furthermore, the combination of these was more compact, which was favorable for the reaction between the hydroxyl groups on the corn stalks and the aldehyde groups on the DAS to generate more acetal and/or hemiacetal structures. Therefore, the strength and water resistance of the particleboards were improved. However, when the board density exceeded 1.1 g/cm^3, the MOR and MOE values started to decrease, and the TS value increased. This may have been due to the excessively high board density hindering the escape of gases generated during the hot pressing process, which resulted in delamination of the particleboard.

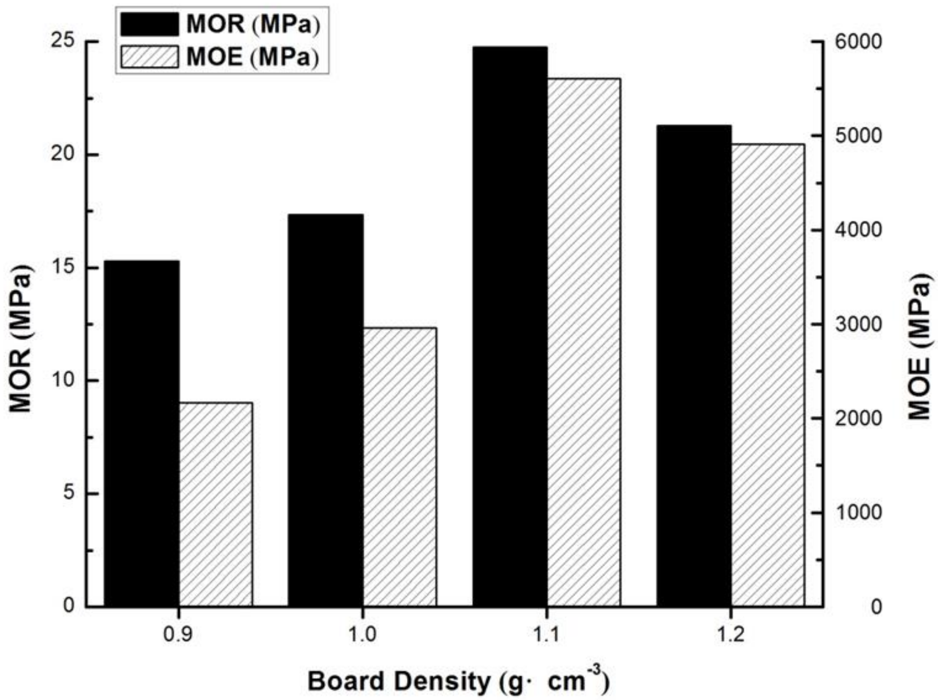

Fig. 4. Effects of board density on the MOR and MOE of the particleboard (Hot pressing temperature: 170 ºC and DAS dosage: 8%)

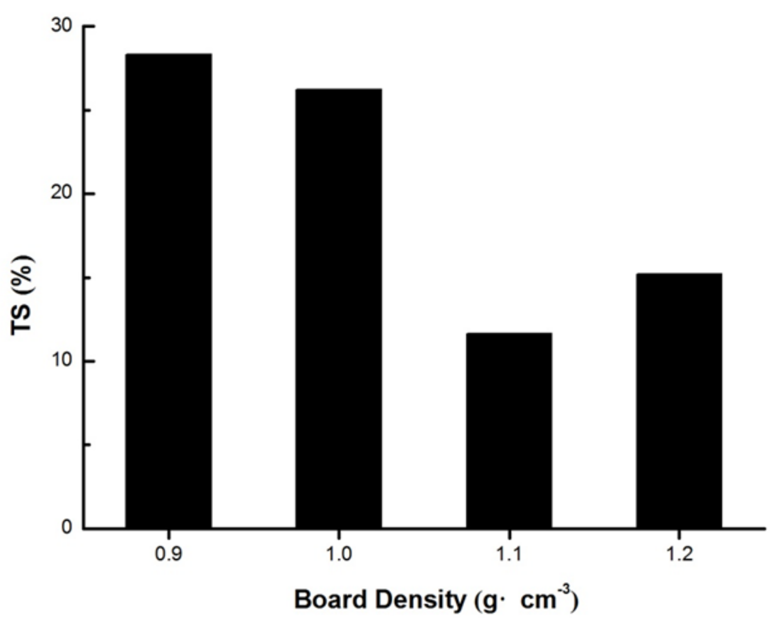

Fig. 5. Effects of board density on the TS of the particleboard (Hot pressing temperature: 170 ºC and DAS dosage: 8%)

Delamination caused a reduction in the bonding force between the fibers of the corn stalks and increased the spaces between the fibers, resulting in a decrease of the strength and the water resistance of the particleboard. In addition, there were many black spots on the surface of the particleboard, which indicated that the excessively high board density could lead to the degradation and carbonization of DAS. This could be another important reason for the decrease in the strength and water resistance of the particleboard.

Effect of Dialdehyde Starch Dosage on Physical Properties of Particleboards

Figures 6 and 7 show that DAS dosage had a significant positive effect on the MOR and MOE of the particleboard. With the increase of DAS dosage, the water resistance of the particleboards had also been significantly improved. When DAS dosage was in the range of 0 to 20%, the MOR value increased first and then decreased as DAS dosage increased. The maximum MOR value was 25.38 MPa when the DAS dosage was 8%. The MOE value showed an overall upward trend. This indicated that tighter bonding between corn stalks and DAS formed with the increase of DAS dosage, and thus the strength of the particleboard was greatly improved. However, when DAS dosage exceeded 8%, the rigidity of the particleboard increased, resulting in a decrease in the MOR of the particleboard. The TS value decreased with the increase of DAS dosage, and the minimum value was 11.59% when the DAS dosage was 20%. This indicated that more acetal and/or hemiacetal structures were formed between corn stalks and DAS with the increase of DAS dosage. Meanwhile, the water resistance of the particleboard was greatly enhanced.

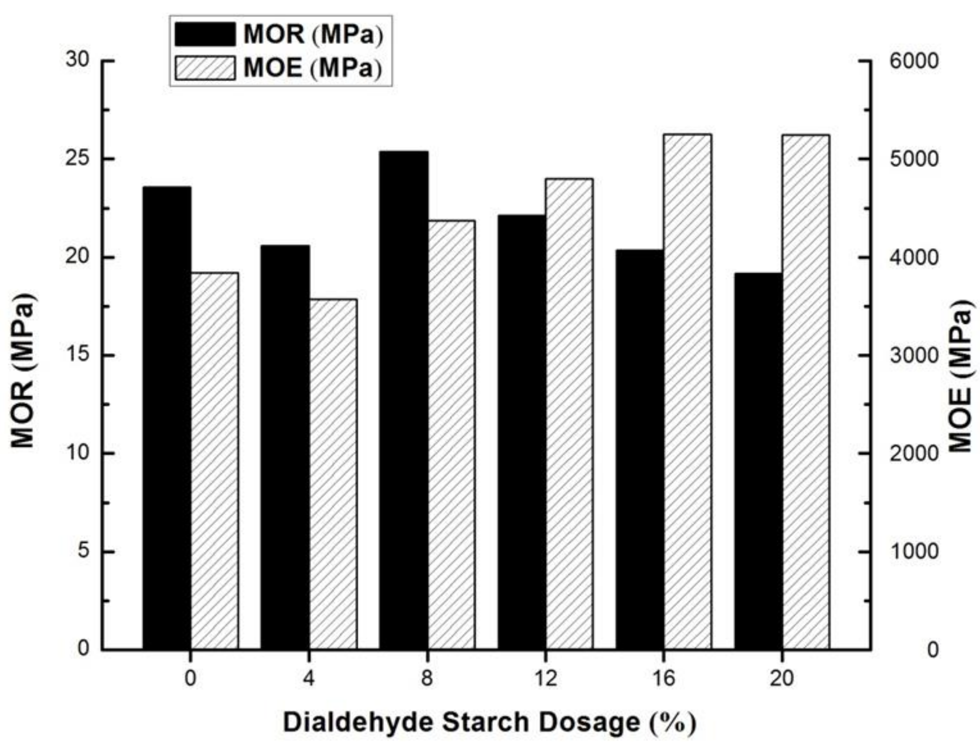

Fig. 6. Effects of DAS dosage on the MOR and MOE of the particleboard (Hot pressing temperature: 170 ºC and board density: 1.0 g/cm^3)

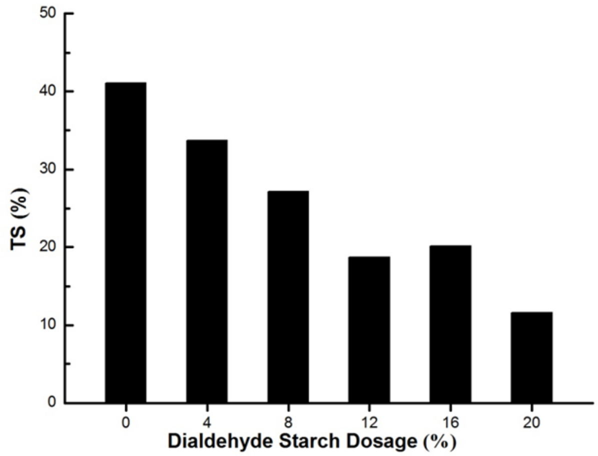

Fig. 7. Effects of DAS dosage on the TS of the particleboard (Hot pressing temperature: 170 ºC and board density: 1.0 g/cm³)

SEM Analysis of Particleboards

Bonding between the DAS and the corn stalk was characterized with SEM graphs of the cross-sections of the particleboard specimens. In Fig. 8, micrographs of the particleboards bonded without and with DAS are shown with different magnifications.

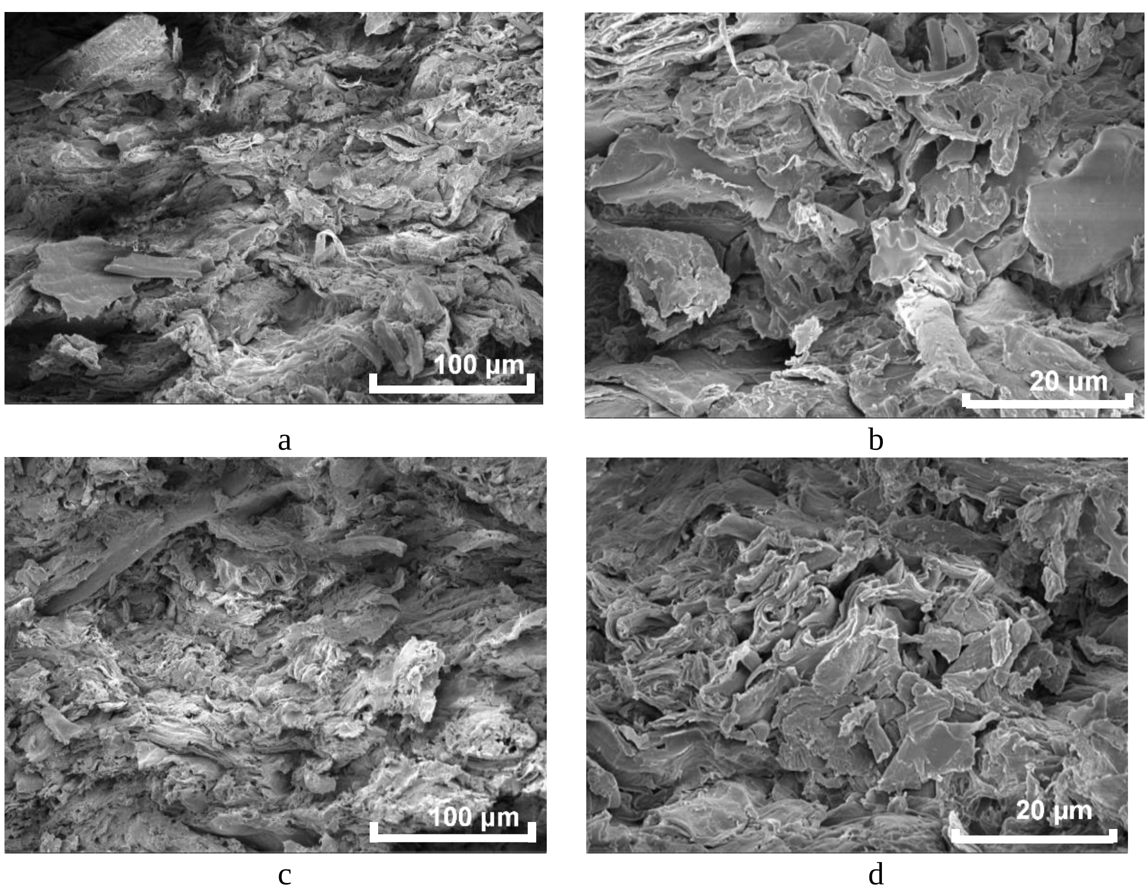

Fig. 8. SEM micrographs of cross-sections of particleboards bonded without (a and b) and with DAS (c and d), with magnifications of 1000 × (a and c) and 5000 × (b and d)

Figures 8a and 8b showed that the particleboard bonded without DAS had an uneven and porous cross-section with many gullies and small fragments, which indicates that the corn stalks did not bind tightly. When the board was bent, the loosely bonded corn stalks were separated and scattered on the cross-section. Thus, the strength of the particleboard was poor. Figures 8c and 8d showed that after adding DAS, the spaces on the cross-section were significantly reduced, and the corn stalks were bonded more tightly. It was apparent that the DAS was dispersed evenly into the spaces, and the condensation reaction occurred between the DAS and the corn stalks, forming acetal and/or hemiacetal structures under hot-pressing conditions. Therefore, the strength and water resistance of the particleboard were enhanced. Figure 9 showed a proposed bonding mechanism of corn stalks and DAS (Saito and Isogai 2006; Sun *et al*. 2015).

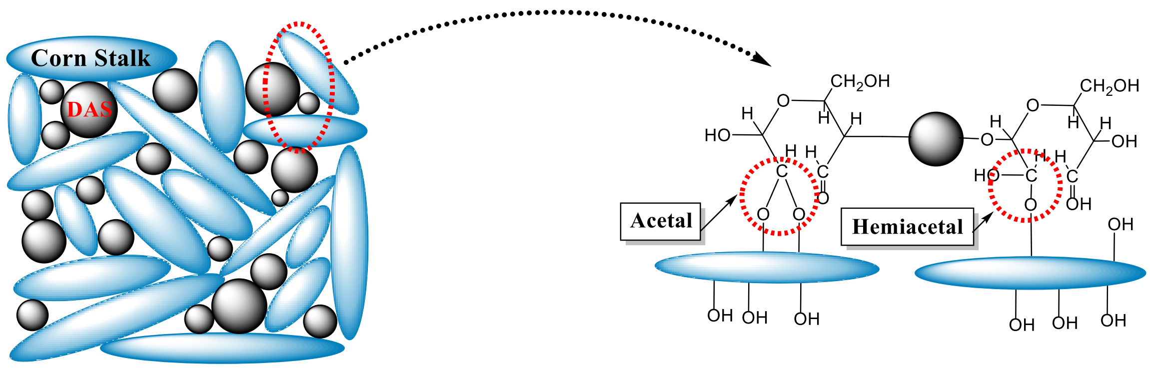

Fig. 9. The proposed bonding mechanism for the interaction between corn stalks and DAS

CONCLUSIONS

1. Sodium periodate can selectively oxidize tapioca starch to form dialdehyde starch (DAS), by breaking the C_2 and C_3 bonds of the anhydroglucose units and oxidizing the hydroxyl groups on these carbons to aldehyde groups.
2. Hot-pressing temperature, board density, and DAS dosage were important factors affecting the modulus of rupture (MOR), modulus of elasticity (MOE), and thickness swelling (TS) of the particleboard. MOR and MOE of the particleboard first increased and then decreased with the increase of the hot pressing temperature, while TS first decreased and then increased. With the increase of board density, MOR and MOE of the particleboard tended to increase first and then decrease, and TS decreased first and then increased. With the increase of DAS dosage, MOR of the particleboard first increased and then decreased, MOE showed an overall increasing trend, and TS showed an overall decreasing trend.
3. The scanning electron microscopic (SEM) analysis of the cross sections of the particleboards showed that the DAS was filled into the spaces of the corn stalks. Under the conditions of high temperature and high pressure, the condensation reaction between DAS and corn stalks occurred, and acetal and/or hemiacetal structures were formed in the particleboard, which tightly bonded the corn stalks together, thereby improving the strength and water resistance of the particleboard.

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