# Effect of Polyvinyl Alcohol Treatment on Mechanical Properties of Bamboo/Polylactic Acid Composites

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Polylactic acid (PLA) and bamboo fiber are both green and biodegradable materials. However, the bonding of PLA and bamboo fiber is poor, which limits the physical properties of paper. The effects of polyvinyl alcohol (PVOH) on PLA fiber/bamboo fiber composites were studied by measuring the tensile strength, tear resistance, and breaking length of the paper. In addition, the morphology of paper comprised of PLA fiber and bamboo fiber were investigated by scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). The tensile index, tear index, and breaking length of paper made with the 4 wt% PVOH-treated bamboo fiber and the untreated PLA fiber compared favorably with the paper made of the untreated bamboo fiber and PLA fiber increased 21.0%, 8.6%, and 20.8%, respectively. However, compared with the paper made of the untreated bamboo fiber and PLA fiber, the tensile index, tear index, and breaking length of the paper made with the treated PLA fiber and the treated bamboo fiber with 4 wt% PVOH solution were dramatically reduced by 30%, 18%, and 30%, respectively.

*Keywords: Polyvinyl alcohol; Polylactic acid fiber; Bamboo; Mechanical property*

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## INTRODUCTION

Polylactic acid (PLA) is a commonly used biodegradable plastic material (Tokiwa *et al*. 2009; Vanstrom 2012) with good biocompatibility, gloss, transparency, thermal stability, and other characteristics (Cui *et al*. 2007; Jung 2008; Wu *et al*. 2015). Therefore, the application of this biofriendly material has grown extensively (Zhao and Gu 2014). If the PLA fiber can be processed directly into thin sheet material through the traditional papermaking method, it will have a greater cost advantage. Because PLA fibers do not form hydrogen bonding as plant fibers (Natarajan *et al*. 2014; Fan *et al*. 2017), the combination of PLA fiber cannot meet the requirements of paper formation. All of the PLA paper with traditional papermaking methods faces many difficulties. Plant fibers are abundant, renewable, and environmentally friendly materials found in nature (Meng *et al*. 2012; Khalil *et al.* 2013; Thakur *et al*. 2014), and they also have high biodegradability. The use of superior performance plant fibers and a PLA fiber mixture not only can improve the strength of the film material but also reduce the cost of production materials. In 2015, Wang and researchers successfully mixed PLA fiber with bamboo fiber through a paper forming process and found that bamboo fibers can improve the mechanical properties of fibrillated polylactic acid into paper (Wang *et al*. 2015). Thus, it is important to combine PLA fiber and bamboo fiber, thereby achieving better adhesion force and improving the paper's physical strength.

Polyvinyl alcohol (PVOH) is a linear water-soluble synthetic polymer produced *via* partial or full hydrolysis of polyvinyl acetate to remove the acetate groups (Baker *et al*. 2012). The polymer has structural regularity, high crystallinity, and a high number of hydroxyl groups in its molecular structure (Naebe *et al*. 2008; Ni *et al.* 2017). It is widely used by blending with other polymer composites, such as biopolymers and other polymers with hydrophilic properties; PVOH is utilized for various industrial applications to enhance the mechanical properties of films because of its compatible structure and hydrophilic properties (Limpan *et al.* 2012; Gaaz *et al.* 2015).

Therefore, this study aims to improve the strength of the PLA/bamboo fiber paper with the use of PVOH. In this study, PVOH pre-treated PLA fiber and bamboo fiber were used to improve the bonding property and the physical strength of PLA/bamboo composite paper.

## EXPERIMENTAL

### Materials

Bamboo pulp was bought from Guizhou Chitianhua Paper Industry Co., Ltd., (Guizhou China). The “bamboo fiber” means the “bamboo pulp” in the paper. The weighted average length of bamboo fibers was 0.71 mm. The average fiber width was 11.98μm.

The PLA fiber was bought from Haining Sprutop Chemical Technology Co., Ltd. (Zhejiang China). The fiber length and diameter was 6 mm and 1.3 μm, respectively, with the glass transition temperature ($T_g$) being 69.0 °C and 157.2 °C.

A partially hydrolyzed (87.1 mol % ± 1.0 mol %) PVOH was supplied by Kuraray Co., Ltd. (Shanghai, China). The $T_g$ was 68 °C and the molecular weight ($M_w$) ranged from 72600 g/mol to 81400 g/mol.

*Beating of bamboo fiber*

Bamboo pulp was refined in a Mark VI type PFI refiner (Hamjern Maskjn, Hamar, Norway) to a beating degree of 40 °SR for the experiment, while the PLA fibers were free of beating.

*Preparation of polyvinyl alcohol solution*

Polyvinyl alcohol and water at a mass ratio of 1:9 were mixed. The mixture was heated and stirred in a water bath. The mixture was then heated to 95 °C and held for 2 h. The stirring speed was 300 rpm/min. After the mixing and stirring of the heated PVOH solution at 75 °C, it was diluted into the mass fractions of 2 wt%, 4 wt%, and 6 wt%.

*Treatment of bamboo fiber and PLA fiber*

The experiment was divided into three samples. Solutions of 2 wt%, 4 wt%, and 6 wt% of PVOH solution were added to 6 wt% of bamboo pulp or PLA fiber (relative to the mass ratio of the mixture). Then, the mixture was stirred at 75 °C with a magnetic stirrer at a speed of 300 rpm for 30 min and then the bamboo pulp or PLA fiber was removed. The treated bamboo pulp was washed six times with 2000 mL of distilled water, centrifuged, and placed in a sealing bag for 24 h at room temperature. Then, the dryness of the treated bamboo fiber and PLA fiber was measured.

*Papermaking process*

The bamboo fiber: PLA fiber ratios were fixed at 7:3 for papermaking with a basis weight of 80 g/m$^2$. This study was conducted on four distinct samples. Sample 1 consisted of the paper made of untreated bamboo fiber and untreated PLA fiber. Sample 2 was the paper made of the treated bamboo fiber and untreated PLA fiber. Sample 3 was the paper made of the treated bamboo fiber and treated PLA fiber. Sample 4 was the paper made of untreated bamboo fiber and treated PLA fiber. The papermaking process is diagramed in Fig. 1. Then, the wet sheets obtained were subsequently pressed at 4.8 MPa and dried at 85 °C.

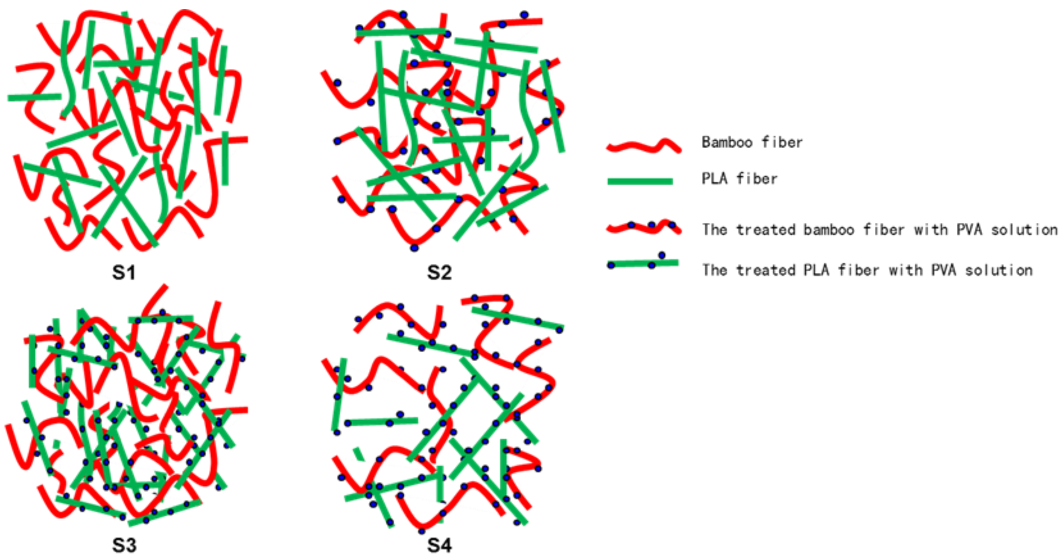


**Fig. 1.** The process of papermaking: S1: paper made of untreated bamboo fiber and untreated PLA fiber; S2: paper made of the treated bamboo fiber and untreated PLA fiber; S3: paper made of untreated bamboo fiber and treated PLA fiber; and S4: paper made of the treated bamboo fiber and treated PLA fiber

## Methods

The surface morphology of samples, including untreated/treated PLA fiber, untreated/treated bamboo fiber, and PLA/bamboo composite paper were observed using a scanning electron microscope (SEM; S-3700N, Hitachi, Ltd., Tokyo, Japan) with an acceleration voltage of 11.0 KV. All samples were sputter-coated with approximately a 2-nm thickness of gold foil prior to the experiment. The SEM images were taken at magnifications between 100× and 5000×.

The standard atmospheric conditions for the testing papers were a relative humidity of 50% ± 2% and a temperature of 23 °C ± 1 °C according to ISO 187 (1990). The tensile strength and breaking length properties of the resulting papers were examined using a tensile strength tester (L&W CE062, Kista, Sweden). The tear resistance of the resulting papers was examined by the tear tester (L&W 009, Kista, Sweden). Thus, the tensile index (N·m/g) of paper was calculated with Eq. 1,

$$X = \frac{S}{G} \times 1000 \tag{1}$$

where $G$ is the basis weight (g/m$^2$) and $S$ is the tensile strength (kN/m) of paper.

The tear index (mN·m$^2$/g) of paper was calculated with Eq. 2,

$$X = \frac{a}{G} \tag{2}$$

where $G$ is the basis weight (g/m$^2$) and $a$ is the tear resistance (mN) of paper. The experimental data (the tensile strength, breaking length and tear resistance) in this paper were average values obtained from five replicates.

## RESULTS AND DISCUSSION

### Mechanical Properties of PVOH-treated Bamboo Fiber Paper

The tear index, tensile index, and breaking length of the paper made from the treatment of bamboo fiber with different concentrations of the PVOH solution and the untreated PLA fiber are shown in Figs. 2 and 3.

**Table 1.** Mechanical Properties of the Paper Sheets

| Sample | Modifier PVOH Dosage (wt%) | Tensile Strength (kN/m) | Breaking Length (km) | Tear Resistance (mN) |
|---|---|---|---|---|
| S1 | 0 | 2.292 | 2.88 | 1785.9 |
| S2 | 2 | 2.39 | 2.91 | 1803.2 |
| | 4 | 3.00 | 3.48 | 2094.2 |
| | 6 | 2.33 | 2.74 | 1800.5 |
| S3 | 2 | 2.56 | 3.17 | 1774.4 |
| | 4 | 2.58 | 3.26 | 1852.8 |
| | 6 | 2.44 | 3.04 | 1754.2 |
| S4 | 2 | 1.56 | 1.99 | 1429.5 |
| | 4 | 2.39 | 2.91 | 2058.5 |
| | 6 | 2.01 | 2.38 | 1618.7 |

As shown in Fig. 2, it can be found that when the PVOH solution concentration was 4 wt%, the tensile index and breaking length of the paper reached its highest value, 34.12 N·m/g and 3.48 km, respectively. This was attributed to the fact that PVOH was coated on the fiber surface when the bamboo fiber was treated. As is well known, the tensile index and breaking length of the paper are mainly affected by the binding force between fibers and the average fiber length (Rohde *et al.* 2011; Naito *et al.* 2012). Also, PVOH is a hydrophilic polymer (Naebe *et al.* 2007). The molecular structure contains a lot of hydroxyl groups, which are easy to form strong hydrogen bonds between molecules. During PVOH treatment, molecules were coated on the fiber surface, and the hydroxyl groups on the surface of PVOH molecules preferentially formed hydrogen bonds with the hydroxyl groups on the surface of bamboo fiber.

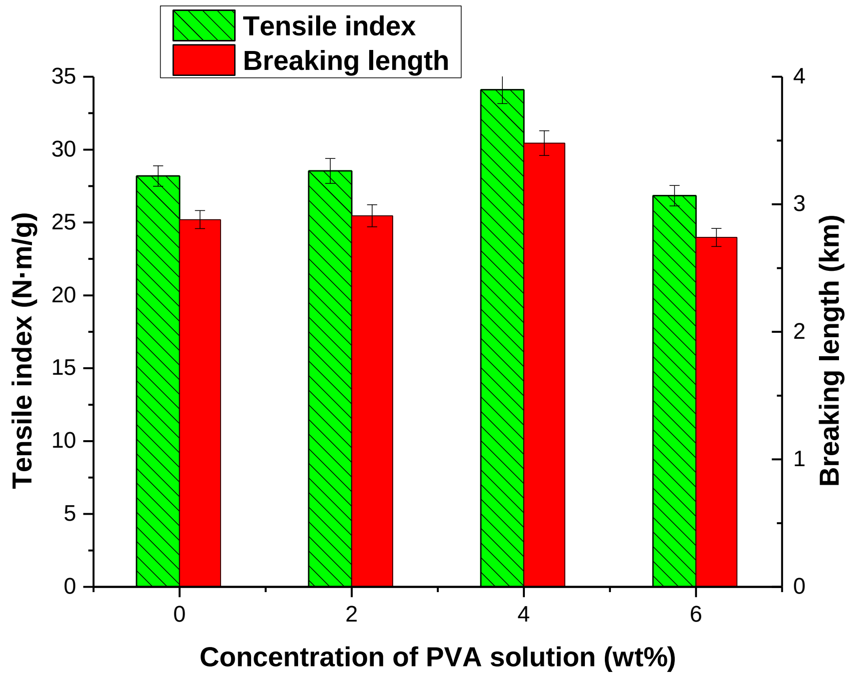


**Fig. 2.** Tensile index and breaking length of the bamboo fiber paper after PVOH solution treatment

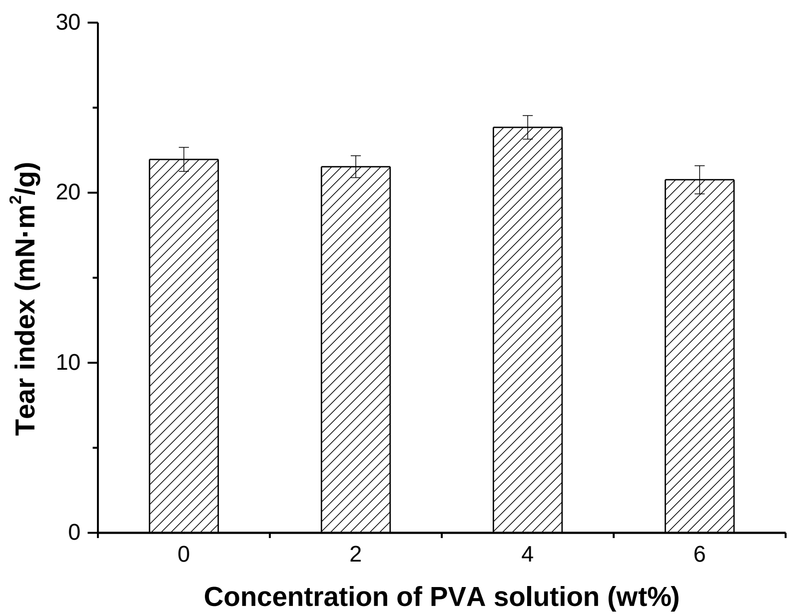


**Fig. 3.** The tear index of the bamboo fiber paper after PVOH solution treatment

This coating not only increased bamboo fibers bonding and reduced the chemical polarity of bamboo fiber surface, but it also improved the combination between bamboo fiber and PLA fiber. Therefore, the tensile index and breaking length of the paper increased. Because the concentration of 2 wt% PVOH solution was low, in this environment its impact on the bamboo fiber surface was minimal. Thus, the tensile index and breaking length slightly increased. When the concentration of PVOH solution was increased to 4 wt%, the tensile index and breaking length of paper increased 21% and 20.8%, respectively. However, the higher concentration of PVOH solution, the worse dispensability of the treated bamboo fiber in the pulp suspension. At the same time, it easily generated its own cross-linked flocculation, which reduced its combination with the PLA fiber. Therefore, it can be seen that the tensile index and breaking length of the paper decreased after the treatment of bamboo fiber with 6 wt% PVOH solution.

The tear index of the paper made from the treated bamboo fiber and untreated PLA fiber is demonstrated in Fig. 3. The factors that affect the tear index of the paper are mainly the average length and strength of the fiber, followed by the fiber binding force, fiber orientation, and fiber interlacing (Todoroki *et al.* 1997). Thus, the tensile index and tear resistance of paper is not a straight line (Nissen *et al.* 2008). Therefore, the tensile strength of soft paper is high, but the tear resistance is low. After the bamboo fiber was treated with 2 wt% PVOH solution, the tensile index of the paper slightly increased, but the tear index was mildly reduced. This may have been because the 2 wt% PVOH solution had a lower viscosity. Therefore, the bamboo fiber could be fully exposed to PVOH solution when PVOH solution treated the bamboo fiber. The PLA fiber and bamboo fiber were distributed evenly during papermaking, resulting in a slight increase in most of the paper. Bamboo fiber that was treated with 4 wt% PVOH solution experienced an 8.6% in the tear strength of the paper. In this environment, the binding force of fibers increased, while the paper structure was tight. PVOH is a water-soluble polymer that readily reacts with different cross-linking agents (Koski *et al.* 2004). When more PVOH is present on the fiber surface, the hydroxyl groups on the PVOH molecules are conjugated to form hydrogen bonds (Chang *et al.* 2005). When the concentration of PVOH was 6 wt%, the fiber easily generated their own cross-linking. The treated bamboo fiber and PLA fiber distribution appeared uneven in the paper. As a consequence, the combination of the two fibers decreased. Therefore, when the bamboo fiber was treated with 6 wt% PVOH solution, the tear index of the paper was reduced.

## Mechanical Properties of Paper After PLA Fibers Treated by PVOH Solution

The tear index, tensile index, and breaking length of the paper made from the PLA fiber with different PVOH solution concentrations and the untreated bamboo fiber are shown in Figs. 4 and 5.

Figure 4 indicates that when the PVOH solution concentration was 4 wt%, the tensile index and breaking length of paper reached its highest, 31.9 N·m/g and 3.26 km, respectively. In contrast, when the PLA fiber was treated with 6 wt% PVOH solution, the values were lowest, 29.82 N·m/g and 3.04 km, respectively. Compared to the untreated bamboo fiber paper and PLA fiber paper, these values still increased 5.8%.

As shown in Fig. 5, the tear index of the paper was slightly reduced after 2 wt% and 6 wt% PVOH treatment of the PLA fiber. However, the tear index of the paper increased 4% when treated with 6 wt% PVOH solution, overall a minimal change in the tear index. It can be said that the treatment of PLA fiber improved the hydrophilic properties of the PLA fiber surface and the binding force of PLA fiber and bamboo fiber, but it did not have much effect on the strength of the PLA fiber itself. Therefore, there was minimal impact on the tear strength of the paper after PVOH solution treatment of the PLA fiber.

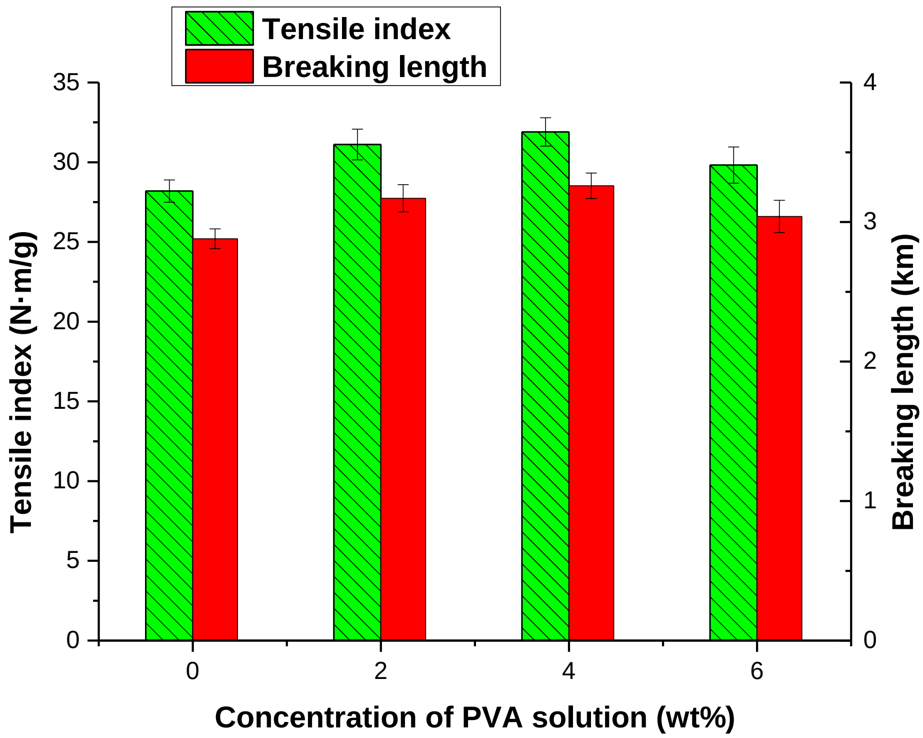


**Fig. 4.** The tensile index and breaking length of the PLA fiber paper after PVOH solution treatment

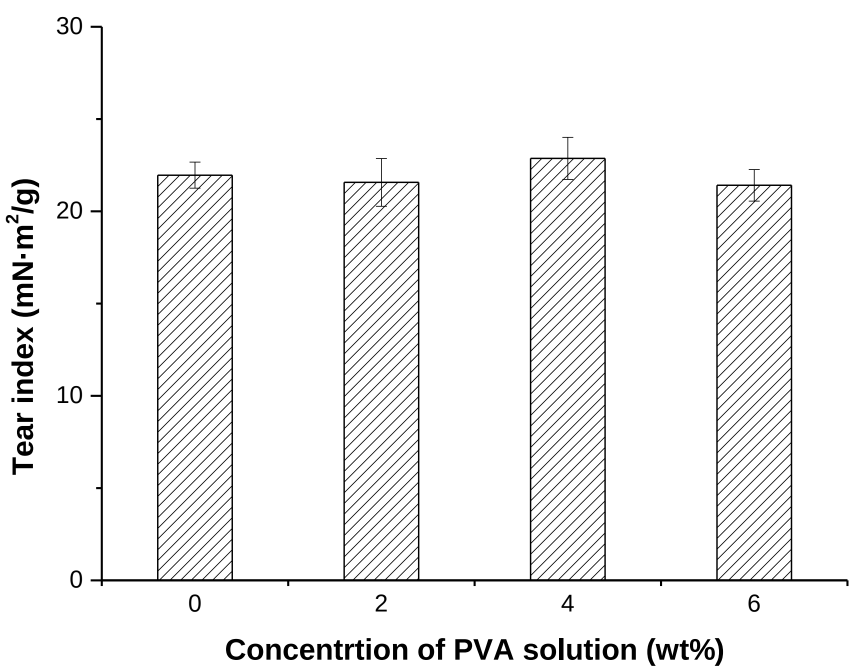


**Fig. 5.** Tear index of the PLA fiber paper after PVOH solution treatment

## The Mechanical Properties of Paper After the Bamboo Fiber and PLA Fibers were Treated by PVOH Solution

The tear index, tensile index, and breaking length of the paper made of the treated PLA fiber and the treated bamboo fiber with different concentrations of the PVOH solution are shown in Figs. 6 and 7.

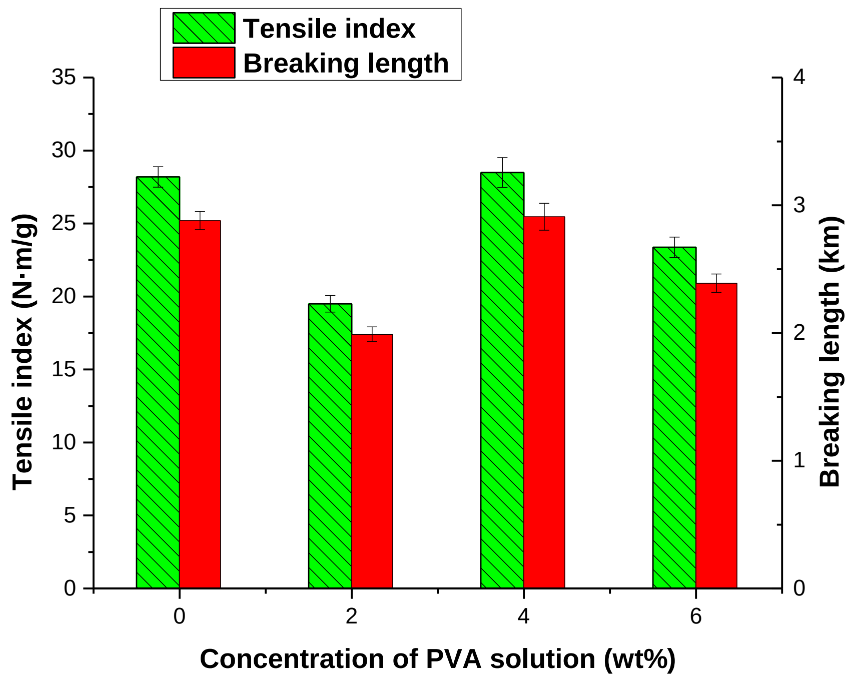


**Fig. 6.** The tensile index and breaking length of the bamboo fiber and PLA fiber paper after PVOH solution treatment

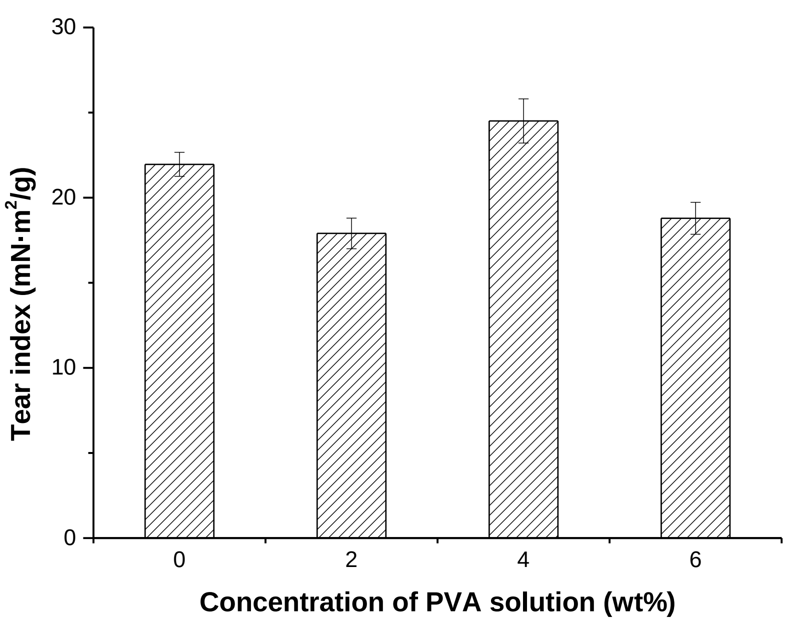


**Fig. 7.** The tear index of the bamboo fiber and PLA paper after PVOH solution treatment

As shown in Figs. 6 and 7, when the bamboo fiber and PLA fiber were treated with 4 wt% PVOH solution, the mechanical properties (tensile index, tear index, and breaking length) of the paper were 28.50 N·m/g, 24.51 mN·m$^2$/g, and 2.91 km, respectively. Even though the tensile index and breaking length of the paper increased, it was not much change relative to the paper that was made from the untreated bamboo fiber and PLA fiber. The difference was that the tear index of the paper increased 11.6%. It was reasonable to infer that the treated PLA fiber surface was covered with a certain amount of PVOH molecules, and that PVOH molecular structure contains a large number of hydroxyl groups, so some hydroxyl groups are exposed on the surface of the PLA fiber. When the bamboo fiber was treated, the hydroxyl groups on the surface of the fiber immediately combined with the hydroxyl groups on the PVOH molecule to produce hydrogen bonds, which improved bonding of the bamboo fibers (Fatehi and Xiao 2010) while reducing the hydrophilic nature of surface of the bamboo fiber. When the two fibers were treated with 2 wt% PVOH solution, the two kinds of fibers were coated with a small amount of PVOH molecules. When the two kinds of fibers formed paper, the PVOH molecules on the surface of fiber gave priority for the cross-linking among the molecules to occur, which weakened the combination of the treated bamboo fiber and the treated PLA fiber. At the same time, it could cause the flocculation of the same kind of fiber, so that the mechanical properties (the tensile index, tear index, and breaking length) of paper were clearly reduced 30%, 18%, and 30%, respectively. However, when the two fibers were treated with 6 wt% PVOH solution, the two kinds of fibers were coated with a lot of PVOH molecules, the tensile index, tear index, and breaking length of paper were reduced 17%, 14%, and 17%, respectively. When the PLA fiber was treated with 4 wt% PVOH solution, the presence of PVOH molecules had no difference in the binding force between the two fibers and the binding force of the two fibers themselves. However, when the bamboo fiber was treated with 4 wt% PVOH solution, it markedly increased the bonding among bamboo fibers bonding, and the tear index of the paper increased 11.6%.

## Fiber Morphology Observation

As indicated from the above discussion, and based on the experimental results, the mechanical properties of bamboo fiber/PLA fiber material were obviously changed by the treatment of bamboo fiber and PLA fiber with PVOH solution. To confirm the PVOH solution's effect and understand the bonding and distribution of the treated fiber and untreated fiber, a SEM observation was conducted.

After the fiber was beaten, the cell wall was displaced and deformed, while the primary and secondary walls were removed, and the fiber produced fine fibers (Willison *et al.* 1977). Consequently, the flocculation of fibers was scattered, and numerous fibers started to fibrillate, which led to more free hydroxyl groups (Wang *et al.* 2015). The SEM images of the untreated bamboo fiber and treated bamboo fiber are illustrated in Figs. 8 (a) and (b). There are many wrinkles on the untreated bamboo fiber, as shown in Fig. 8 (a). However, the treated bamboo fiber surface is relatively smooth for the untreated bamboo fiber as shown in Fig. 8 (b). Based on the result, it can be concluded that the fiber surface is smooth and inerratic after PVOH solution treatment, which is mainly attributable to PVOH coated on the fiber surface. Meanwhile, the SEM images of the untreated bamboo fiber and untreated PLA fiber paper and the paper made of the treatment of bamboo fiber with 4 wt% PVOH solution and untreated PLA fiber are illustrated in Figs. 8 (e) and (f). It can be clearly seen that compared with the untreated bamboo and untreated PLA fiber, the distribution and contact between the treated bamboo fiber and untreated PLA fiber was more complete. Thus, the PVOH molecules on the surface of the bamboo fiber promoted the combination of the bamboo fiber and PLA fiber. Furthermore, it also supported the theoretical basis for the improvement of the mechanical properties of the treated bamboo fiber paper with 4 wt% PVOH solution and the untreated PLA fiber paper.

The SEM images of the untreated PLA fiber and treated PLA fiber are illustrated in Figs. 8 (c) and (d).

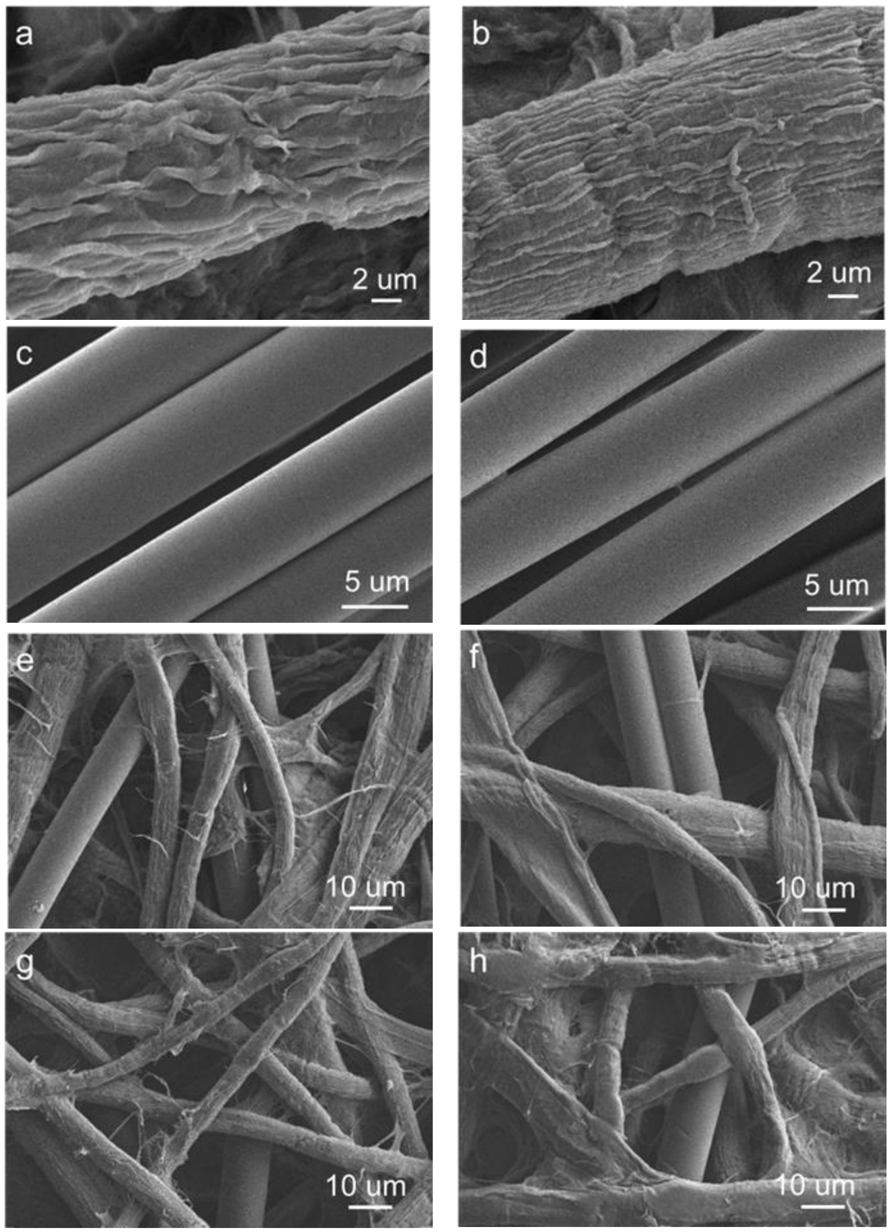


**Fig. 8.** SEM images of fibers and paper sheets: a) untreated bamboo fiber; b) treated bamboo fiber with 4 wt% PVOH solution; c) untreated PLA fiber; d) treated PLA fiber with 4 wt% PVOH solution; e) paper sheet made of untreated bamboo fiber and untreated PLA fiber; f) paper sheet made of bamboo fiber with 4 wt% PVOH solution and untreated PLA fiber; g) paper sheet made of bamboo fiber and PLA fiber with 4 wt% PVOH solution; and h) paper sheet with untreated bamboo fiber and PLA fiber with 4 wt% PVOH solution

As shown, the surface of the untreated PLA fiber was very smooth and the interface between the fibers was clear. The surface of the treated PLA fiber was also smooth, but there was adhesion between the fibers, which was due to the effect of the PVOH molecules on the surface of fiber. Due to the PVOH molecules containing a large number of hydroxyl groups, it made the PLA fiber surface hydroxyl and increased the affinity between the PLA fibers when the PVOH molecules attached to the surface of PLA fiber. As shown in Figs. 8 (e) and (h), the distribution of bamboo fibers is increased around the treated PLA fiber.

The SEM images of the treated bamboo fiber and treated PLA fiber paper are shown in Fig. 8(g). As shown, the distribution of the two fibers was not uniform in the paper, and the distribution of treated bamboo fibers was concentrated. In addition, as can be seen in Figs. 8 (f) and (g), the affinity and distribution between the treated PLA fiber and the untreated bamboo fiber were more relative to the untreated bamboo fiber and the untreated PLA fiber.

## FTIR Analysis

Figure 9 illustrates the infrared spectra of the paper sample made of the treated fiber material and untreated fiber material. As shown in Fig. 9, the characteristic absorption peak of the four paper samples were similar, which meant that no new absorption peaks appeared. However, there were slightly different degrees of difference in strength, which indicated that no chemical reaction happened and no new group was introduced into the PLA/bamboo fiber composite paper by PVOH-treated PLA and bamboo fibers. Only the vibration intensity of the original group had some impact.

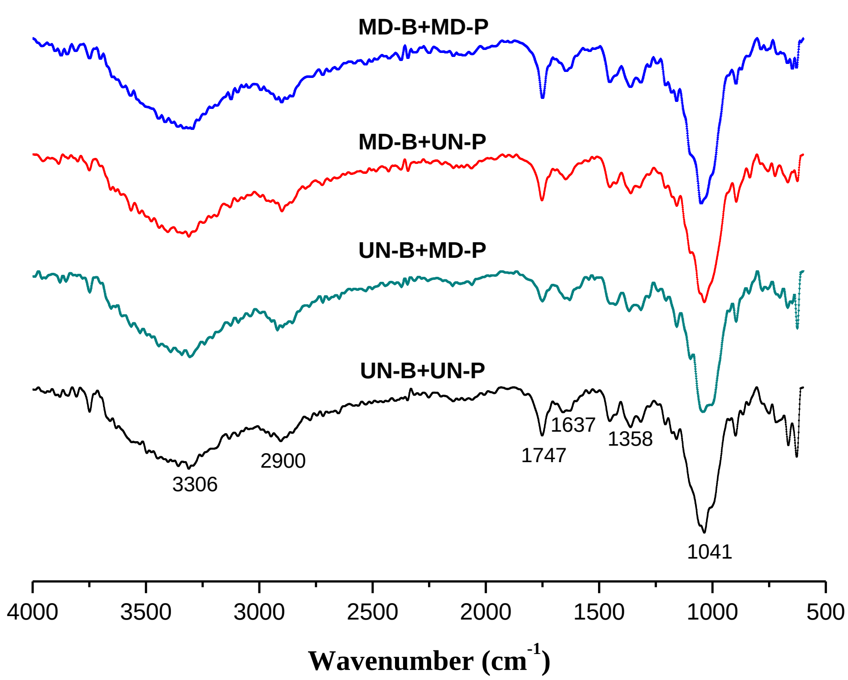


**Fig. 9.** Infrared spectra of the paper sample: UN-B+UN-P: paper made of untreated bamboo fiber and PLA fiber; MD-B+UN-P: paper made of untreated PLA fiber and bamboo fiber with 4 wt% PVOH solution; MD-B+MD-P: paper made of PLA fiber and bamboo fiber with 4 wt% PVOH solution; and UN-B+MD-P: paper made of untreated bamboo fiber and PLA fiber with 4 wt% PVOH solution

It can be concluded that the PVOH-treated fibers did not undergo a derivatization reaction. The absorption peak at 3400 $cm^{-1}$ was the stretching vibration of -OH on the molecular chain of plant fiber, and the characteristic absorption peak in the range of 2900 $cm^{-1}$ to 2800 $cm^{-1}$ was the stretching vibration of -CH on the acetylglucosamine unit. The characteristic absorption peak of 1747 $cm^{-1}$ was attributable to the C=O vibration of COO-, and the peak intensity of untreated fiber material is weaker than the treated fiber material, which indicated that the treated PLA fiber or the treated bamboo fiber promoted the formation of hydrogen bonds.

## CONCLUSIONS

1. The use of a polyvinyl alcohol (PVOH) solution to pretreat polylactic acid (PLA) fiber and bamboo fiber had great influence on the mechanical properties of the resulting paper.
2. When the bamboo fiber was pretreated with PVOH solution, the strength of the treated bamboo fiber and PLA fiber paper was considerably improved. When the PLA fiber was pretreated by PVOH solution, the strength of the paper comprised of the treated PLA fibers and bamboo fibers were only slightly improved. However, if the bamboo fiber and PLA fiber were both pretreated with PVOH solution, the mechanical properties of paper made of the treated PLA fibers and treated bamboo fiber were conspicuously reduced.
3. The physical properties (tensile index, tear index, and breaking length) of the 4 wt% PVOH-treated bamboo fiber paper and the untreated PLA fiber increased 21.0%, 8.6%, and 20.8%, respectively. However, the physical properties (tensile index, tear index, and breaking length) of the paper made from the treated PLA fiber and the treated bamboo fiber with 4 wt% PVOH solution were dramatically reduced 30%, 18%, and 30%, respectively.

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