# Effects of Treatment Temperature on Properties of Starch-based Adhesives

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Starch-based adhesives were developed by hydrolyzing starch with polyvinyl alcohol under alkaline and acidic conditions at various treatment temperatures. The chemical and physical structures of the corn starch and hydrolyzed starch were characterized with Fourier Transform infrared (FT-IR) spectroscopy, X-ray diffraction (XRD), and scanning electronic microscopy (SEM). Thermal degradation and bond strength were also evaluated. The results indicated that the bond strength of starch adhesives reached a maximum value at 40 °C. The FI-IR results showed that the amount of hydroxyl groups first increased and then decreased with increasing treatment temperature. When the treatment temperature was 55 °C the crystallinity of treated starch was the lowest and the thermal resistance also the weakest; it decreased by 10.1% and 13.6% respectively compared to untreated starch. Obvious erosion could be observed from the SEM images of treated starch. In addition, the interaction of polyvinyl alcohol (PVA) and starch also could be observed, and the results indicated that the compatibility between starch and PVA became better and better as the treated temperature was increased.

*Keywords: Starch adhesive; Acidic and alkaline treatment; Treatment temperature; Structure; Bond strength*

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## INTRODUCTION

Starch is one of the most abundant natural, renewable, biodegradable polymers and is produced by many plants as a source of energy. In recent years, starch has attracted great attention as a raw material for producing wood adhesives because of its relatively low price, abundance, non-toxicity, and its renewable and biodegradable properties (Xie 2008; Zhang *et al.* 2002). However, starch-based adhesives have not been widely applied at an industrial scale due to inadequacies such as water resistance, storage stability, and mechanical properties (Guo *et al*. 2007; Hu *et al.* 2009).

In light of the cited inadequacies, it is necessary to use effective ways to chemically, physically, or biologically modify starch to change its viscosity and solubility and improve other relevant properties when preparing starch-based adhesives (Wu *et al.* 2009, Wang *et al.* 2012). Chemical reagents such as acid (Zuo *et al.* 2013; Shi *et al.* 2009), alkali (Palacios-Fonseca *et al.* 2013; Uthumporn *et al.* 2012) and enzymatic (Hu 2007) treatments are often used in modification of starch. Acid treatment has been applied for the hydrolysis of starch granules and to produce soluble starch for many years. Hydrochloric acid, sulfuric acid, and citric acid are often used to alter the properties of native starch through causing scission of glucosidic linkage. Abdorreza *et al.* (2012) reported the effects of acid hydrolysis on physicochemical and rheological properties of sago starch. The molecular weight of amylopectin and amylose were decreased after 24 h of hydrolysis, whereas the gelatinization temperature and enthalpy of acid-hydrolyzed sago starch increased with increasing degree of hydrolysis. Both acid and alkali treatment starches are often used industrially as premodification steps for production of starch adhesives.

Agents such as NaOH and KOH also are frequently used as alkali treatment. Wang (2012) studied the effect of alkaline treatment on the structural and functional properties of pea starch granules using a range of characterization methods, which showed that there were small decreases in relative crystallinity and double helix content after 15 days of alkaline treatment. Deformations could be observed on the surface of alkali-treated granules, and there was evidence of adhesion between some of the granules. Recently, many researchers focused on formaldehyde-free wood adhesives, which are prepared through the reaction between some special polymers and starch. These polymers, such as starch/polyvinyl alcohol (PVA) (Syed *et al*. 2001), starch/isocyanate (Ramizi *et al*. 2011), and starch/tannin (Moubarik *et al*. 2010), were blending with treated starch to improve their bonding performance. Among the above polymers, PVA is suitable for blending with starch since it is highly polar and can also be manipulated in water solution (Nadras *et al*. 2011). It is well known that polymeric methylene diphenyl diisocyanate (pMDI) can be used as a crosslinking agent to improve the bond strength and water resistance of certain polymers (Tan *et al*. 2011; Hamid *et al.* 2006; Gu *et al.* 2010).

Treatment temperature is an important parameter during the process of preparing starch adhesives. The temperature could affect the chemical reaction in the experiment, thereby influencing the physical and chemical properties of starch adhesives. Zuo *et al.* (2012) studied the effects of acid hydrolysis temperature on the properties of starch. The results showed that the crystallinity and gelatilization temperature increased first and decreased with the increasing of acid hydrolysis temperature. Gelatilization viscosity was rapidly decreased with the increasing of acid hydrolysis of temperature. Different starch types have different gelatilization temperature, and increasing temperature under alkali and acid treatment could influence gelatilization temperature of starch (Uthumporn *et al.* 2012). Besides molecular structure, properties of starch adhesives such as the liquidity and viscosity would finally be changed (Samson *et al.* 1990). Chen *et al.* (2010) studied the effects of water contents, temperature, time, and pH on granules morphology of chestnut starch. Results suggested that the heating temperature could promote the swelling of granules. Heating temperature had more effect on granules morphological changes compared with heating time, whereas higher alkali concentration had stronger effects on the swelling of granules than neutral and acid condition.

Starch-based wood adhesives were prepared by mixing starch with PVA during alkali treatments, followed by acid treatment, using pMDI as a crosslinking agent. Considering the treatment temperature under acid and alkali condition can affect the degree of starch hydrolysis and properties of adhesives. The objective of this study was to investigate the effect of treatment temperature on the structure and properties of the starch-based adhesives. Fourier-transform infrared (FI-TR) spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM), and thermal gravimetric analysis (TGA) were used to analyze the chemical and physical structures of the adhesives. The bond performance of these adhesives was also evaluated.

## EXPERIMENTAL

### Materials

Corn starch was supplied by Shandong Hengren Industry Corp. (Shandong, China). PVA (PM 1799/1788) was provided by Tianjin Yongda Chemical Reagent Corp. (Tianjin, China). Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were purchased from Jiangsu Dongsheng Chemical Reagent Corp. (Jiangsu, China). The pMDI was obtained from Bayer Co. (Tianjin, China). All other reagents were of analytical grade.

### Methods

*Preparation of starch-based wood adhesives*

Adhesive preparation was carried out in a three-necked, round-bottom flask equipped with a condenser, a magnetic stirrer, and a thermometer. About 10 g of PVA-1788, 10 g PVA-1799, and 150 mL of de-ionized water were added to the flask. The mixture was heated to 90 °C and kept at 90 °C for 50 min to ensure that the PVA completely dissolved. The mixture was then cooled to a certain temperature (40, 45, 50, 55, or 60 °C). About 100.0 g of starch-water solution (1:1, w/w) was poured into the mixture, and the pH of the mixture was adjusted to 10.0 with NaOH solution (30%, w/w) and stirred for 30 min. The pH was then adjusted to 3.0 with HCl solution (10%, w/w), and the mixture was stirred for another 30 min. The pH was once again adjusted to 10.0 for 30 min and then to 3.0 for 30 min. The pH was adjusted back to 10.0, and the mixture was stirred for 30 min. Finally, the pH was adjusted to 6.5 with HCl solution and the mixture was cooled to room temperature to yield the treated starch emulsion. The crosslinking agent (pMDI) was added to the emulsion at a 4/1 (w/w) ratio before the starch-based wood adhesives were used.

*Lap shear strength test*

The lap shear (bond) strength of the adhesives with and without the pMDI crosslinking agent was tested according to Chinese industry standard GB/T 17657-1999 (1999). Wood veneers with 1 mm thickness were cut into rectangles with dimensions of 2.5×10 $cm^2$. Adhesive was applied to one side of each piece of veneer. The adhesive-coated area was 2.5×1.3 $cm^2$ at one end of each veneer. The spread rate of the adhesive was 250 $g/m^2$. The coated areas of two adhesive-coated veneers were overlapped with their grains parallel to each other and pressed with 0.45 MPa pressure and 25 °C for 72 h. The bonded samples were soaked in hot water (63 °C) for 3 h before shear testing. The shear test was carried out using a MWD-100 universal mechanics testing machine (Jinan Times Shijing Corp., China) while the samples were still wet. The test speed was 1 mm/min. Twelve replicates were tested for each condition.

*Fourier transform-infrared spectroscopy (FT-IR) measurement*

The original corn starch and treated starch emulsion samples were dried and milled with KBr before being pressed into a thin sheet for FT-IR analysis using an IRPrestige-21 FT-IR spectrometer (Shimadzu Corp., Japan). Each sample was scanned 32 times from 4000 to 400 $cm^{-1}$ at a resolution of 4 $cm^{-1}$.

*X-ray diffraction (XRD) measurement*

The X-ray diffraction data were collected at room temperature using an XRD-6000 diffractometer (Shimadzu Corp., Japan) with CuKα radiation operating at 40 kV and 30 Ma. The corn starch and treated starch emulsion samples were tested. The treated starch emulsion was freeze-dried and cut into small pieces. The test samples were made by pressing these pieces into slices. All samples were scanned from 5 to 40° at a scanning speed of 5°/min with a step size of 0.04°. The relative crystallinity of the samples was calculated using the machine's software.

*Scanning electron microscopy (SEM) measurement*

Images showing the granular morphology were obtained using a TM-1000 SEM (Hatachi, Ltd., Japan) operated at 15000 kV accelerating voltage. The freeze-dried emulsion and the corn starch samples were mounted on aluminium specimen holder using double-sided carbon tape and sputter-coated with a thin gold film.

*Thermal gravimetric analysis (TGA)*

Thermal gravimetric analysis was conducted using a STA409c thermal analyzer (NETZSCH Scientific Instruments Trading (Shanghai) Ltd., Germany). The samples were freeze-dried, and about 5 to 10 mg were weighed for measurement. The heating rate was 10 °C/min, and the measured temperature range was from 25 to 600 °C.

## RESULTS AND DISCUSSION

### Effect of Treatment Temperature on Bond (shear) Strength

The bond strength of the adhesives treated at different temperatures is shown in Fig. 1. The adhesives without crosslinking with pMDI had very low bond strength. Adding pMDI dramatically improved the bond strength to over 0.8 MPa. This suggests that the -NCO groups in the pMDI reacted with the -OH groups in the starch, resulting in a crosslinked network that improved the bond strength and water resistance (Shi *et al.* 2008).

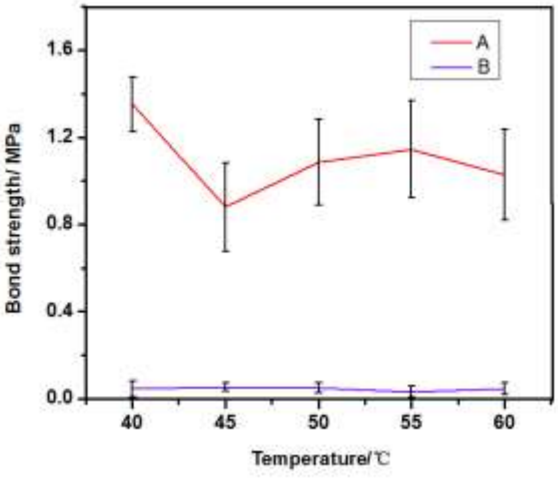


**Fig. 1.** Effects of treatment temperature on bond strength of starch adhesives A: starch treated with pMDI B: starch treated without pMDI

The bond strength of the starch-based adhesives, with the addition of pMDI, was also affected by the treatment temperature. The adhesive had the highest bond strength (1.35MPa) when the treatment temperature was 40 °C. Because steric hindrance among starch granules and PVA was the smallest at low 40 °C, comparing with other samples the starch swelled the least, which could lead to low viscosity and high flowability. This adhesive could easily penetrate into the adherent material and reinforce it with high bond strength (He *et al*. 2003; Shi *et al*. 2008). When the treatment temperature was increased, the bond strength first increased, and then decreased. The bond strength value stopped increasing as the temperature exceeded 55 °C. This was likely related to the amount of free hydroxyl groups present on the starch molecular chains. Shi (2008) suggested that unreacted hydroxyl groups in adhesive systems could enhance the interaction between the adhesive and the wood surface, leading to high bond strength. The treatment of starch with acid and alkali broke the hydrogen bonds between the starch molecules and increased the amount of free hydroxyl groups. The amount of free hydroxyl groups started to decrease at 60 °C, yielding to the bond strength decrease.

## Effect of Treatment Temperature on Structures

### *FT-IR analysis*

The starch and treated starch samples were analyzed using FT-IR. Figure 2 shows IR spectra of the various starch samples.

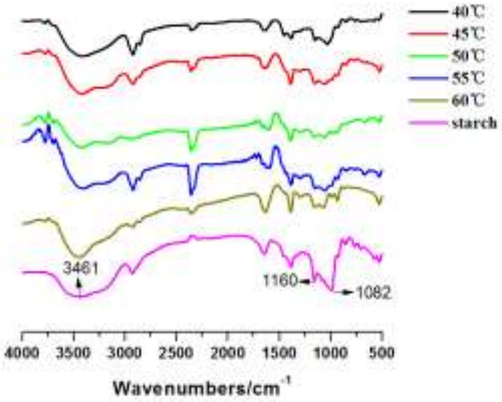


**Fig. 2.** FTIR spectra of corn starch and treated starch

The spectra show that hydroxyl absorption in the starch was located at approximately 3461 cm$^{-1}$, but the hydroxyl absorption in the treated starch samples was located between 3500 and 3400 cm$^{-1}$ with stronger, narrower peaks. This suggests that the treatment with acid and alkali broke the molecular interaction and increased the number of free hydroxyl groups. The intensity of the hydroxyl peak first increased, then decreased, after reaching a maximum when the treatment temperature was increased, which is in agreement with the results of bond strength testing. The bands at 1082 and 1160 cm$^{-1}$ were the feature peaks of the C-O-C and C-O bond of the glycosidic linkages in the starch (Goodfellow and Wilson 1990). The intensities of these two peaks visibly decreased in the treated starch samples because the treatment with acid and alkali resulted in the breakage of glycosidic bonds.

### *XRD analysis*

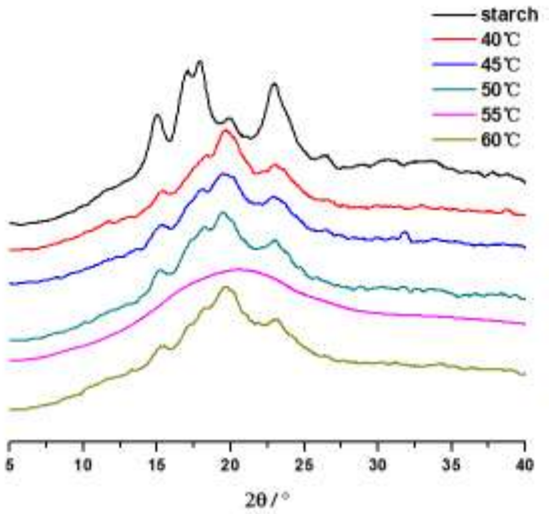


**Fig. 3.** X-ray diffraction patterns of the corn starch and treated starch samples

The X-ray diffraction patterns for starch and treated starch are shown in Fig. 3. The diffraction patterns of corn starch had strong peaks at $2\theta$ values of 15.06, 17.10, 18.06, and 2h3.02°, and its crystallinity pattern was A-type (Liu 2003). After treatment, the peak at $2\theta$= 17.10° became weaker, and the absorption peaks became broader in general. However, the peak at $2\theta$ = 18.06° became stronger because acid hydrolysis occurred first in the amorphous part of the starch molecule, forming short molecular chains from the broken molecular chains. These short chains easily rearranged to form small lattices of microcrystals (Sun *et al*. 2004). Unlike the acid hydrolysis, the alkali hydrolysis could hydrolyze the crystalline part of the starch more easily (Jung-Ah and Seung-Taik 2004), making the crystalline peaks weaker.

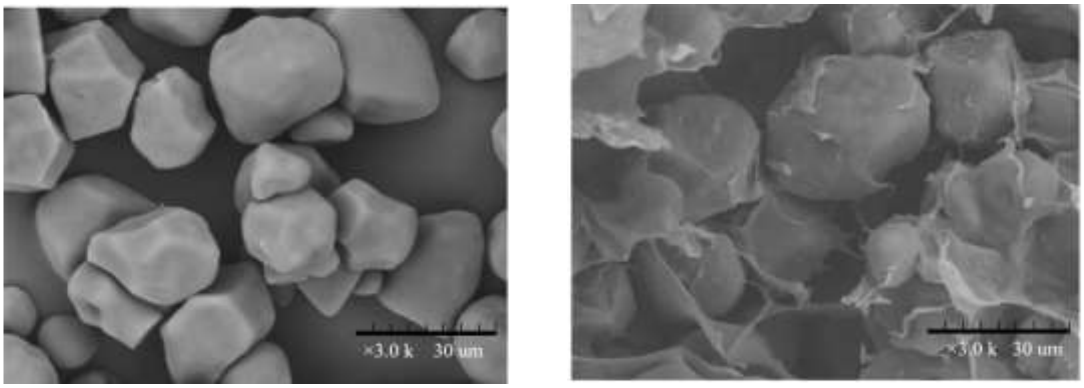
**Table 1.** Crystallinity of Starch and Treated Starch Samples

| | Sample | Crystallinity (%) |
|---|---|---|
| | Corn starch | 57.0 |
| Treatment temperature (°C) | 40 | 53.9 |
| | 45 | 58.2 |
| | 50 | 52.5 |
| | 55 | 51.2 |
| | 60 | 53.3 |

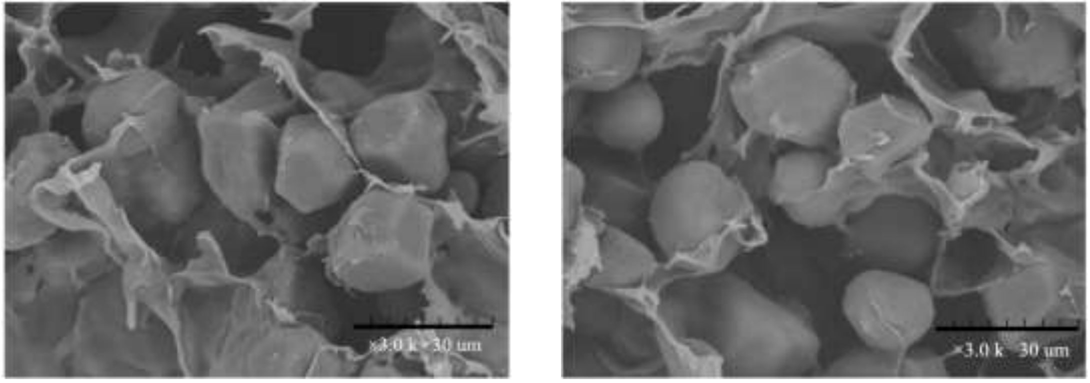
The crystallinities of starch and treated starch samples at various temperatures were calculated from the X-ray diffraction data, as shown in Table 1. The crystallinity of starch treated at 45 °C was slightly higher than that of the corn starch, probably because at this treatment temperature, Hydrolysis is generally faster in amorphous regions than in crystalline regions, leading to a higher ratio of crystalline part-to-amorphous part (Sun *et al*. 2004). However, the crystallinity generally decreased at other treatment temperatures because both amorphous and crystalline parts were hydrolyzed. The crystallinity decreased with increasing treatment temperature and the highest decrease, 10.1%, occurred when the treatment temperature was 55 °C. The crystallinity increased slightly as the temperature increased beyond 55 °C due to increased crosslinking between the starch and PVA (Zhang *et al*. 2002). The hydrogen bond interactions between the starch and PVA first decreased, and then it increased with increasing treatment temperature, resulting in the same variation in crystallinity.

*SEM analysis*

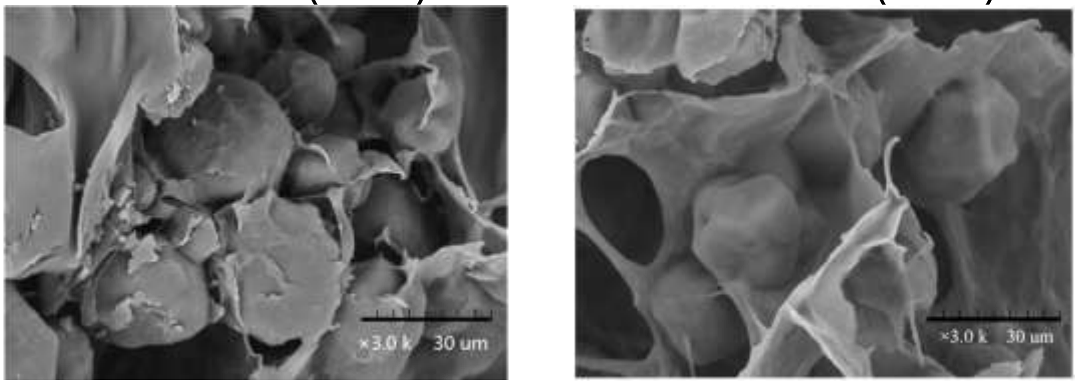
Figure 4 shows the SEM photos of the corn starch and treated starch samples. The untreated corn starch contained dispersed or agglomerated particles that were irregular polyhedrons with smooth surfaces. The surfaces were corroded when starch was treated with acid and alkali.



**a: Corn starch** **b: Treated starch (T=40 °C)**



**c: Treated starch (T=45 °C)** **d: Treated starch (T=50 °C)**



**e: Treated starch (T=55 °C)** **f: Treated starch (T=60 °C)**

**Fig. 4.** Scanning electron micrographs of corn starch and treated starch samples

Rough surfaces indicated that both the amorphous and crystalline regions contributed to rough surfaces because both the amorphous and crystalline parts were hydrolyzed by the acid and alkali (Abdorreza *et al.* 2012; Palacios-Fonseca *et al.* 2013). PVA filled the spaces in the starch particles to a certain extent and covered the surfaces of the starch particles. The coverage first increased and then slightly decreased with increasing treatment temperature due to interaction between PVA and starch. Hydrolysis produced free hydroxyl groups in the starch, increasing the compatibility between PVA and starch. More free hydroxyl groups resulted in more compatibility between PVA and starch, and thus more coverage of starch particles with PVA. The coverage reached was greatest following treatment at 55 °C, indicating that hydrolysis reached its highest degree at this temperature.

### *TGA analysis*

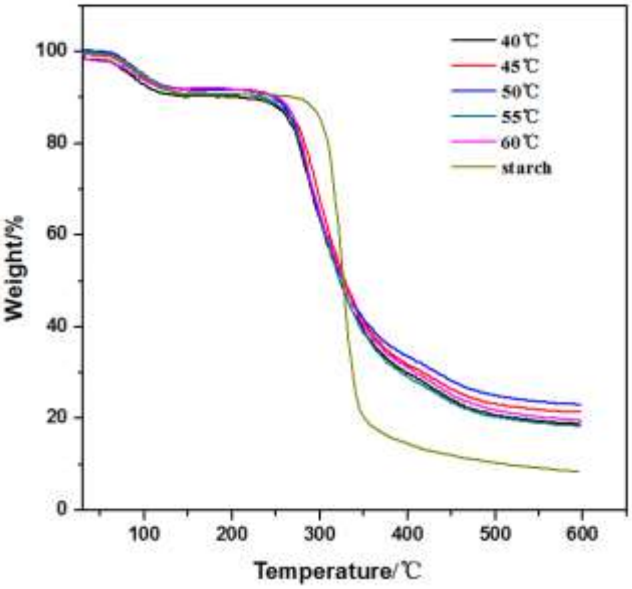


**Fig. 5.** TGA curves of starch and treated starch samples

The corn starch and treated starch samples were subjected to thermal gravimetric analysis, and the results are shown in Fig. 5.

All curves indicated that there were two weight loss sections. The first weight loss, at temperatures close to 100 °C, was small and was due to water evaporation and volatilization of small molecules from the samples. The second weight loss, between 255 and 470 °C, was significant and was due to the degradation of the starch macromolecules themselves (Wang and Les 2012).

The initial degradation temperatures of the treated starch samples were 264.6, 270.6, 268.2, 265.6, and 266.0 °C with treatment temperatures of 40, 45, 50, 55, and 60 °C, respectively, lower than that of untreated starch (307.5 °C). This suggests that the hydrolysis of starch reduced its thermal resistance to some extent. The degradation curves were similar to each other among the treated starch samples, but the initial degradation temperature was different. It was highest in the samples treated at 45 °C and decreased to a minimum at 55 °C, and then increased as temperature was increased further. Hydrolysis reduced the crystallinity of the starch samples, reducing the initial degradation temperature because the amorphous part can be thermally degraded first.

## CONCLUSIONS

1. Starch hydrolysis at different treatment temperatures affected the chemical structure, crystallinity, surface morphology, thermal degradation, and bonding performance of the adhesives produced.
2. Results showed that the amount of hydroxyl groups first increased and then decreased with increasing treatment temperature.
3. The crystallinity of the treated starch decreased the most at 55 °C.
4. The compatibility between starch particles and PVA was the best, and the initial thermal degradation temperature was lowest, when the starch samples were treated at 55 °C.
5. The bond strength reached the maximum value at the lowest temperature 40 °C. With the increasing of temperature, the bond strength value of all samples first increased and then decreased.

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