

# Mechanical Properties Research and Structural Characterization of Alkali Lignin / Poly(vinyl alcohol) Reaction Films

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With alkali lignin and PVA as the primary materials, glutaraldehyde as the cross-linker, and glycerin as the plasticizer, alkali lignin/PVA cross-linking reaction film was prepared by casting. The process conditions were evaluated one at a time for different variables. Mechanical properties of the composite film were tested. The performance of the reaction film was analysed by XRD, TG, SEM, and FT-IR. The results showed that when the mass ratio of alkali lignin/PVA was 1/5, glutaraldehyde content 1.67%, and glycerol content 7.1% (w/w) of the dry matter, respectively, the reaction film had satisfactory mechanical properties. Under the best conditions, the mechanical properties of the reaction film were better than that of pure PVA film. The thermal stability of the reaction film was higher than the stability of PVA film. Compared with pure PVA film, the crystallinity of the reaction film decreased a little. SEM images indicated that compatibility of alkali lignin and PVA was good. The FT-IR analysis showed that a cross-linking reaction occurred between alkali lignin and PVA(1788). But the aldol reaction between the lignin and PVA(1788) was weak.

*Keywords:* Alkali lignin; Polyvinyl alcohol; Cross-linking; Mechanical properties

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## INTRODUCTION

Lignin is the most abundant natural aromatic polymer on earth, and as such represents a vast and renewable reservoir of latent polymeric materials or aromatic chemicals. The industrial conversion of wood to paper separates lignin from the cellulosic material and in the process generates approximately 50 million tons of lignin annually. In addition, the recent increased interest in biomass high-value utilization is expected to generate further quantities of lignin en route to biomass-based products. Therefore, finding new value-added applications for lignin would help to reduce pollution associated with lignin and render lignin more useful (Sivasankarapillai and McDonald 2011).

Industrial lignin is a thermoplastic natural macromolecular material having a broad molecular weight. Because of its photosensitive groups such as carbonyl, lignin can be photodegraded. Lignin, with its high ability to absorb UV absorption, can be used as a filler for various film applications, or it can be added to agricultural mulch (Chiellini *et al.* 2003). Lignin degradation in the soil is the formation of the core material of the soil humus (Tuomela *et al.* 2000). So it can be used as the degradation primer applied to plastics to increase the plastic's degradation (Elizondo *et al.* 2009; Chen and Chen 1996).

Poly(vinyl alcohol) (PVA) ranks among the few forms of biodegradable synthetic polymers that are frequently modified owing to their properties. After being employed by industry, it is carried away in wastewater to treatment plants, where it is more or less successfully degraded (Kumar *et al.* 2012; Frone *et al.* 2011). Nevertheless, it remains in the soil and displays low biodegradation when applied in agriculture. Its time required for degradation, however, is quite lengthy, and continuous effort has been made for this reason, with the goal to shorten the biodegradation time of PVA to the maximum possible extent, while simultaneously attempting to preserve or improve its technological and usage properties (Kharade and Kale 1999; Xiong *et al.* 2008).

Lignin contains a large number of polar functional groups (Fernandes *et al.* 2006a). PVA, a linear polymer containing a large number of hydroxyl groups, also belongs to the polar substances. They have good compatibility. So excellent mechanical properties of materials can be prepared using the reaction between lignin and PVA. The development and utilization of alkali lignin not only can reduce environmental pollution but it also would help to alleviate the resources crisis (Fernandes *et al.* 2006b; Abdul Khalil 2011). PVA blends with alkali lignin can be used as a new material in packaging and in mulching plastic film. However, very limited studies on biodegradable films based on blends of alkali lignin with PVA have been found in the specialized literature. Thus, the aim of this work was to develop films based on blends of alkali lignin and poly(vinyl alcohol), and to study the effect of alkali lignin content in the blends on their mechanical properties.

## EXPERIMENTAL

### Materials

PVA(1788) (88% degree of hydrolysis) was obtained from Shangdong Chemical Plant. Industrial alkali lignin was purchased from Shangdong Paper Mill. Glutaraldehyde 50% solution was obtained from Tianjin Kemiou Chemical Reagent Co. Ltd. The glycerol and NaOH were of analytical reagent grade purchased from Tianjin Fuyu Fine Chemical Co., Ltd.

### Methods

First, industrial alkali lignin was dissolved in aqueous alkali at room temperature, and then the alkali lignin solution was dried by spray-drying technology. The alkali lignin (AL) and PVA were simultaneously put in a three-neck flask with different alkali–lignin proportions with the content from 10% to 50% of the dry matter. A certain amount of distilled water was added to the flask. The alkali lignin, PVA, and water were then mixed with constant stirring (300 rpm) in the flask connected to a water bath at 90 °C (Li *et al.* 2012). Every 30 min from the start of stirring, glycerol and glutaraldehyde were successively added in. After stirring, the solution was filtered for de-foaming. The film-forming solution was then poured and spread evenly over a PVC plastic surface (Belbekhouche *et al.* 2011). Films were dried at room temperature. Prior to characterization, the films were conditioned at 23 °C and 50% RH for 72 h in desiccators with a saturated solution of  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  for the mechanical tests, and for 48 h for penetrability, and for 1 week for the FTIR analysis. Films of PVA with no alkali lignin were also prepared and analyzed in the same way.

## Film Characterization

### *Mechanical performance testing*

Thickness of the films was measured using an electronic digital readout micrometer with a 0 to 25 mm range and accuracy of 0.001 mm. The film thickness was determined from an average of 15 measurements made at five different locations. Mechanical properties were determined using the tensile test (tensile strength, TS, and elongation at break, ELO) using a small material testing machine (model LDX-200). The tensile test was performed according to the standard method GB 130022-91. Rectangle-shaped samples were cut for each film, 160 mm in length and 2 mm in width. The initial grip separation was set at 60 mm and the drawing speed at 45 mm/min. At least five samples from each film were evaluated (Mikkonen *et al.* 2008).

### *Atomic absorption spectrophotometer*

The measurement of Na<sup>+</sup> was performed with a Purkinje General TAS-990 atomic absorption spectrometer equipped with a sodium hollow cathode lamp and a deuterium background corrector at respective wavelengths using an acetylene flame. The set instrument parameters were: a hollow cathode lamp at 590.0 nm wavelength with slit width of 0.4 nm, current of 6.0 mA, a acetylene flow rate of 1300 mL/min, at a burner height of 5 mm. The measurement was carried out in triplicates.

### *X-ray diffraction (XRD)*

XRD measurements were performed on a D/max-r B X-ray diffractometer (Rigaku Corp., Tokyo, Japan) using Cu K $\alpha$  radiation. The samples were scanned over a 2 $\theta$  (°) range varying from 10° to 50° with a scanning rate of 4°/min.

### *Thermogravimetric analysis (TGA)*

TGA was performed on a Netzsch TG-209F3 under flowing nitrogen (20 mL/min) atmosphere at 10 °C/min. Samples of 6 mg were placed into platinum pans.

### *Scanning electron microscopy (SEM)*

Freeze-fractured surfaces and plane were observed using a Quanta200 environmental scanning electron microscope. The samples were coated with gold powder and observed under 1000 $\times$  magnification.

### *Fourier transform infrared spectroscopy (FTIR)*

Infrared spectra of films were recorded between 4000 and 400 cm<sup>-1</sup> at 16 cm<sup>-1</sup> resolution with a Spectrum One (Magna-IR560, Nicolet USA) spectrometer, supplied with a universal attenuated total reflectance (ATR) accessory.

## RESULTS AND DISCUSSION

### **Mechanical Properties**

The effect of the different mass ratio (alkali lignin/PVA) on tensile property is shown in Fig. 1. Both the tensile strength and elongation at break of alkali lignin/PVA films first increased and then decreased in agreement with the findings of others (Julinová *et al.* 2010; Sahoo *et al.* 2011; Huang *et al.* 2012). When the mass ratio of alkali lignin and PVA was 2/10, the reaction film had the best tensile properties. Elongation at

break and tensile strength were 283% and 36 MPa, respectively. Tensile properties of pure PVA film were 254% and 30 MPa. These results suggest that the incorporation of alkali lignin into PVA provides a gain in tensile strength and elongation to breakage. Because lignin has rigid particles, a small amount of lignin showed enhancement of the film characteristics. Luo Xue Gang and others reported that liginosulfonate/PVA blended film also had better tensile properties than pure PVA film (Li and Luo 2011; Julinová *et al.* 2010).

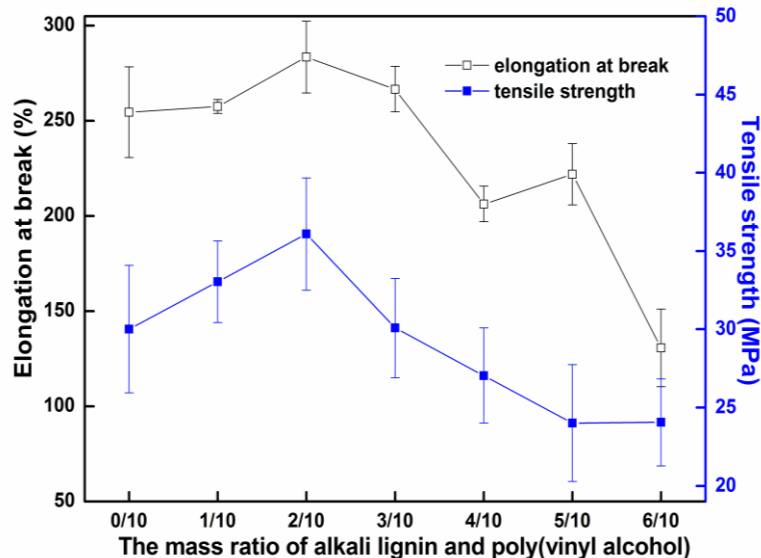


Fig. 1. The effect of different mass ratio of alkali lignin and PVA on tensile property

The effect of different glutaraldehyde content on tensile property is shown in Fig. 2. Alkali lignin/PVA was 2/10 in these films. Elongation at break and tensile strength of alkali lignin/PVA reaction film first increased and then decreased with increasing glutaraldehyde amounts. Alkali lignin and PVA were cross-linked by the glutaraldehyde.

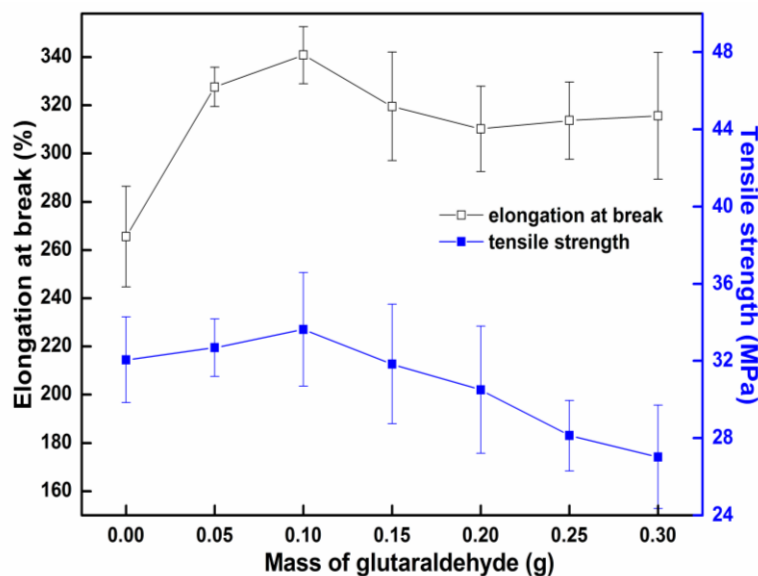


Fig. 2. The effect of different mass of glutaraldehyde on tensile property

Alkali lignin and PVA underwent an acetal reaction, and a network structure of macromolecules was formed. Tensile strength and elongation at break improved to some extent in the end (Farsheh *et al.* 2011). Cross-linking combined with the increasing glutaraldehyde, which resulted in a colloidal solution in a very short time. The obtained film was uneven, so the tensile strength and elongation at break decreased. After adding 0.1 g glutaraldehyde, 1 g of alkali lignin, and 5 g of PVA, the mechanical properties of the film were the best.

The effects of different glycerol content on tensile properties are shown in Fig. 3. In these films the mass of alkali lignin was 1 g, PVA 5 g, and glutaraldehyde 0.1 g. Plasticizer glycerol had a greater impact on the mechanical properties of the films. The plasticizer was used to improve the brittleness of the film. With glycerol increasing, elongation at break increased and tensile strength first increased and then decreased. The higher contents of glycerol and the films with lower tensile strength were the same as results reported by Souza *et al.* 2012. When the amount of glycerol was 0.426 g, the mechanical properties of the film were the best.

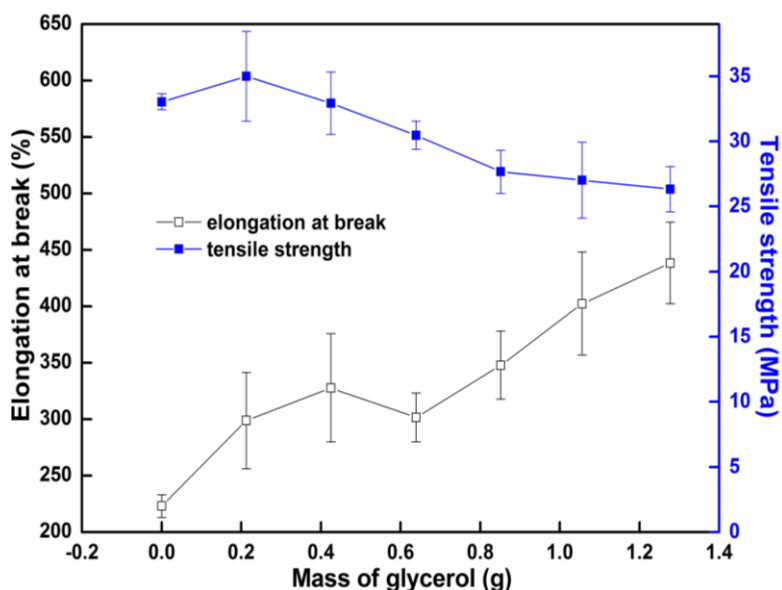


Fig. 3. The effect of different glycerol content on tensile property

### Crystallization Characteristics

XRD patterns (Sionkowska *et al.* 2009) for alkali lignin/PVA film, alkali lignin, and PVA film are shown in Fig. 4. The PVA was shown to be a crystalline type of polymer. There was a strong diffraction peak at  $2\theta = 19.8^\circ$  and unobvious broad peaks at  $2\theta = 11^\circ$  and  $40^\circ$  (Naveen Kumar *et al.* 2010). The alkali lignin was shown to be a typical amorphous polymer, with X-ray diffraction pattern exhibiting a significant dispersion peak. However, Fig. 4 shows that the alkali lignin exhibited two strong diffraction peaks. This phenomenon was caused by the presence of sodium hydroxide particles in the alkali lignin. With the use of a contrasting reaction film with PVA film at  $2\theta = 11^\circ$ , the crystallinity of the reaction film was reduced. Lignin is an amorphous substance, and since the cross-linking reaction existed between the PVA and the alkali lignin, the crystalline structure of PVA was destroyed. But the crystallinity change was very small.

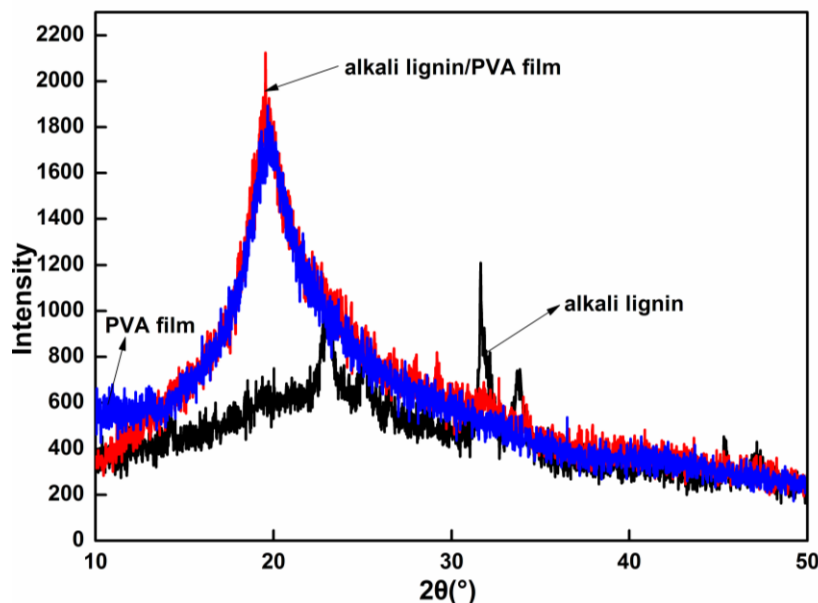


Fig. 4. X-ray diffraction patterns for alkali lignin/PVA film, alkali lignin, and PVA film

### Thermogravimetric Analysis

The thermal stability and degradation of alkali lignin, PVA film, and alkali lignin/PVA reaction film with a ratio of 1:5 was examined by TGA, and the results are presented in Fig. 5. The TG curves showed similar behaviours at the initial stage of thermal degradation. As temperature increased, the thermal decomposition of alkali lignin slowed down (Li *et al.* 2012), and the maximum weight loss temperature ( $T_{max}$ ) took place at 405 °C (Song *et al.* 2011).

In Fig. 5(b), the alkali lignin/PVA reaction films and PVA films exhibited a fast weight loss, respectively, in the vicinity of 285 °C and 300 °C (Yang *et al.* 2004; Zhen *et al.* 2012; Wang *et al.* 2010). The peak at around 285 °C of alkali lignin/PVA reaction films was short and broad. However, the peak around 300 °C of PVA films was long and sharp. This may indicate that reaction films had a lower thermal degradation rate than PVA films. In addition, the peak at around 650 °C in alkali lignin disappeared in lignin, which was caused by the presence of sodium hydroxide (Fig. 5(b)).

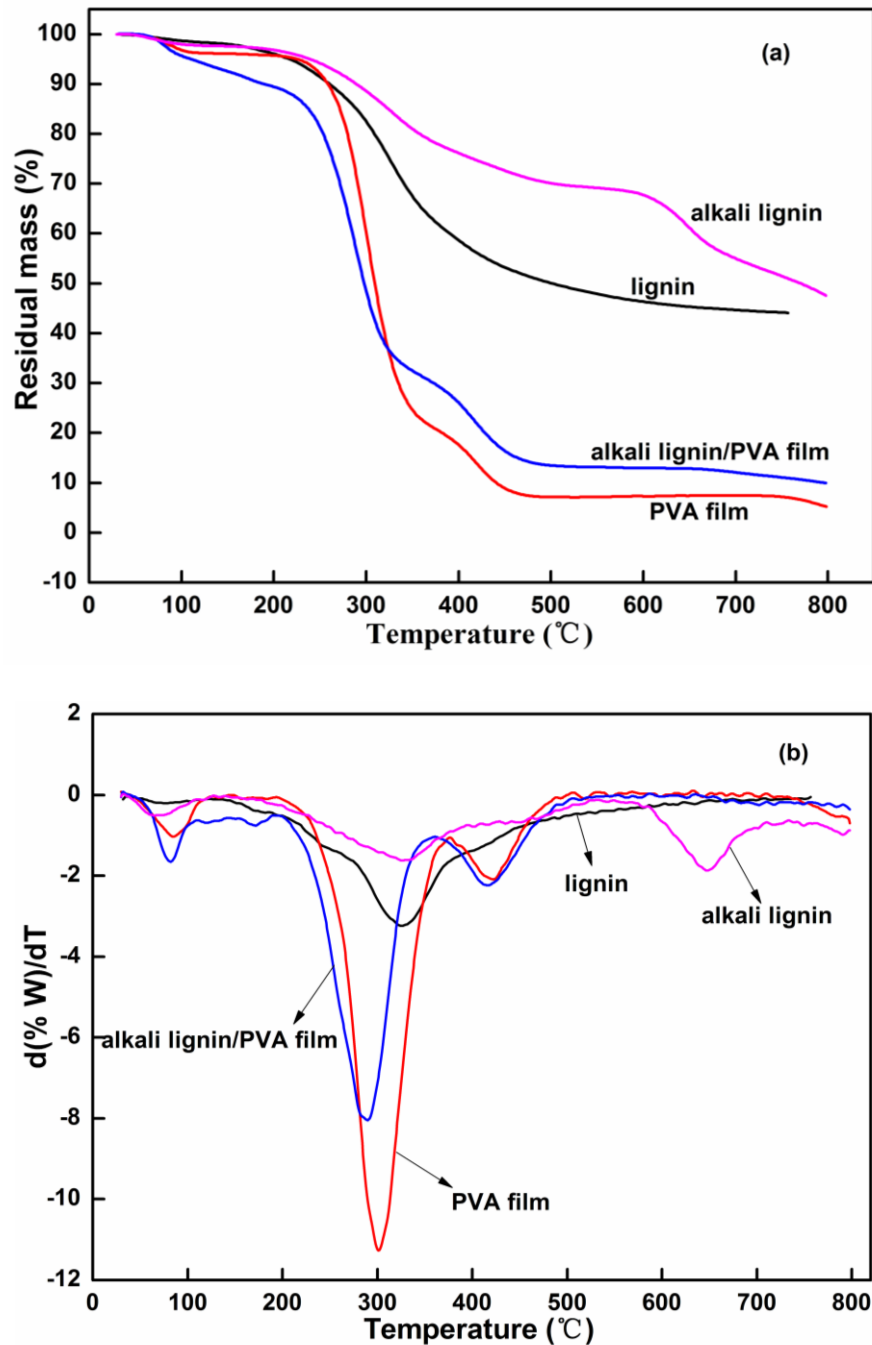
The content of the sodium hydroxide in the composite film was very little. By atomic absorption spectrophotometer, the amounts of NaOH in alkali lignin and in the composite film were 22.09% and 3.68%, respectively. So the peak at around 650 °C also disappeared in the composite film (Fig. 5(b)). In Fig. 5(a), the residual mass at 600 °C is 14.16% for reaction films and 7.99% for PVA films. The amount of NaOH in composite film 3.68% was smaller than the residual mass 14.16%.

The thermal stability of the reaction film was increased due to alkali lignin added. The other reason may be that alkali lignin molecules inserted into the PVA molecules formed an ether bond (Fig. 8). So the thermal stability of the reaction film was higher than the stability of the PVA film.

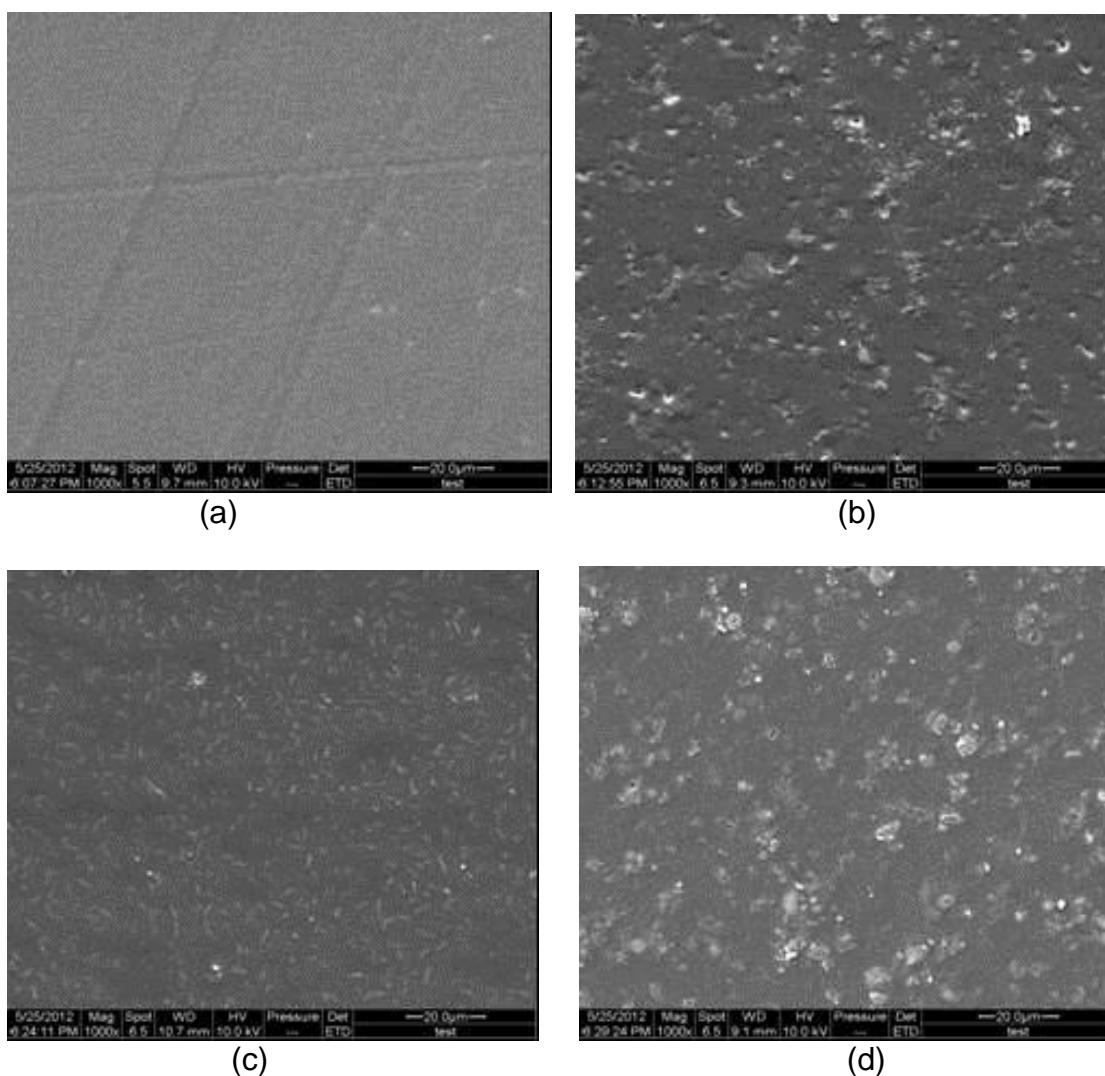
### Surface Morphology

Scanning electron microscopy (SEM) images of PVA and alkali lignin (AL)/PVA film samples were also recorded to investigate the surface morphology of the films (Luo 2008). Figure 6(a) shows the smooth surface of pure PVA film. Figure 6(b) depicts the

rough, irregular surface of reaction films. Comparing (b), (c), and (d) images, it was clear that pits and protrusions were formed on the membrane surface after adding glutaraldehyde. From (b), (c), and (d), it could be seen that glycerol made the surface smooth and glutaraldehyde made the surface rough. The small white points on the film surface indicated the presence of NaOH crystals.



**Fig. 5.** TG (a) and DTG (b) curves of PVA film, alkali lignin/PVA film, alkali lignin, and unmodified lignin

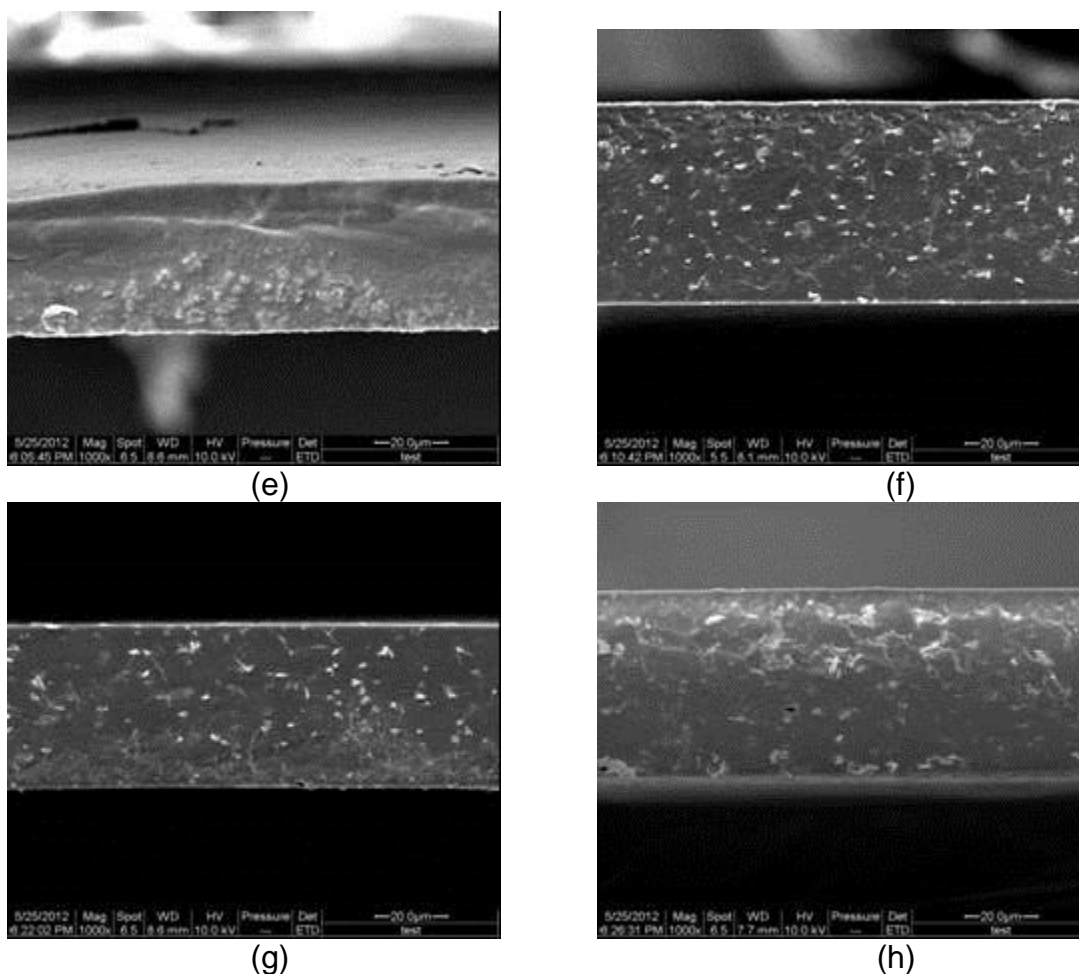


**Fig. 6.** SEM images of films: (a) AL/PVA=0:10, (b) AL/PVA=2:10 with glycerol and glutaraldehyde, (c) AL/PVA=2:10 with glycerol, (d) AL/PVA=2:1 with glutaraldehyde

SEM images of the freeze-fractured surface of the films are shown in Fig. 7. The white dots appearing in Fig. 7(f) are crystals of NaOH evenly dispersed on the film. The glycerol made the freeze-fractured surface (g) smooth. When only glutaraldehyde was added without glycerol plasticization, the freeze-fractured surfaces were obviously uneven (h) and the high molecular weight polymer deposited at the bottom surface.

The four images of Fig. 7 (e), (f), (g), and (h) exhibited the different degrees of subsidence, which was similar to the blend films described by Xu *et al.* (2013). This result may be attributed to the high thickness of the cast film and long drying time. In the process of drying, the water evaporated and high molecular weight polymer in the film first dissolved out, which caused the uneven section. The morphology of (b) and (c), due to the bridging role of glycerol, was conducive to the dispersion of the dry matter, and there was not any obvious evidence of dissolution (Su and Wu 2011).





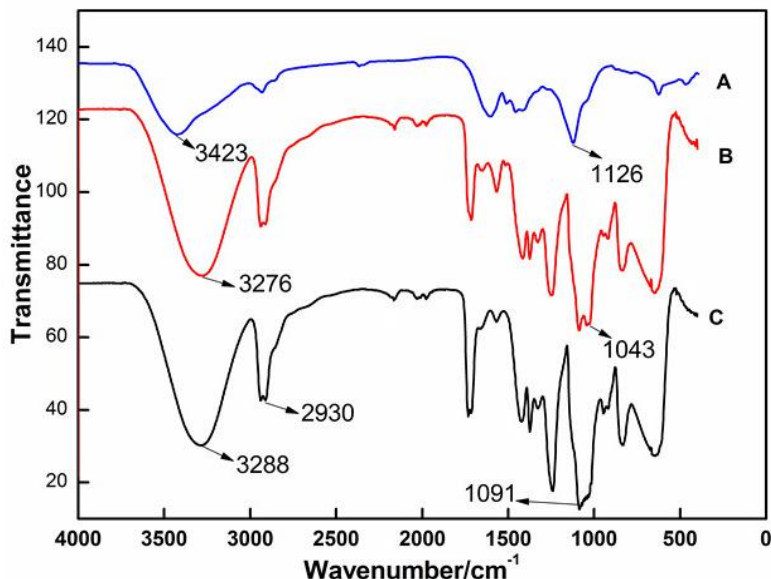
**Fig. 7.** SEM images of the freeze-fractured surface: (e) AL/PVA=0:10, (f) AL/PVA= 2:10 with glycerol and glutaraldehyde, (g) AL/PVA= 2:10 with glycerol, (h) AL/PVA=2:10 with glutaraldehyde

### FT-IR Analysis

The FT-IR spectra of alkali lignin (A), alkali lignin/PVA film (B), and PVA film (C) are shown in Fig. 8. In the spectra of PVA film, the  $3282\text{ cm}^{-1}$  and  $2930\text{ cm}^{-1}$  peaks are the  $\text{-OH}$  and  $\text{-CH}_2$  stretching vibrations (Krumova *et al.* 2000), respectively. The  $\text{C-O}$  stretching range is at  $1091\text{ cm}^{-1}$ .

In the spectra of alkali lignin, the  $3438\text{ cm}^{-1}$  peak is the  $\text{O-H}$  stretching vibration of the alcoholic hydroxyl group and phenolic hydroxyl. The  $\text{O-H}$  stretching vibration at  $3282\text{ cm}^{-1}$  was reduced towards lower wavenumbers a bit (Fig. 8B). The  $\text{C-O}$  stretching range at  $1091\text{ cm}^{-1}$  was reduced (Tao and Luo 2007).

The absorption peak at  $1030\text{ cm}^{-1}$  indicated that an ether bond ( $\text{C-O-C}$ ) was formed (Fig. 8B). But the absorption peak of the ether bond in Fig. 8B was small. This showed that the aldol reaction between the lignin and PVA(1788) was weak. Future work is planned in an attempt to achieve a greater extent of reaction in such films.



**Fig. 8.** FT-IR spectra of alkali lignin (A), alkali lignin/PVA film (B), and PVA film (C)

## CONCLUSIONS

1. The mechanical properties of the films improved after alkali lignin was added. When the mass ratio of L/PVA(1788) was 1/5, glutaraldehyde content 1.67%, and glycerol content 7.1% (w/w) of the dry matter, respectively, the best tensile properties were obtained. After the aldol reaction, the mechanical properties of the films were improved.
2. The thermal decomposition rate of alkali lignin was slow. PVA film and alkali lignin/PVA reaction film both had a fast weight loss in the vicinity of 300 °C and 285 °C, respectively. The reaction films had a lower thermal degradation rate than PVA films. The thermal stability of the reaction film was increased due to alkali lignin added.
3. SEM showed white dots, which indicated the presence of crystals of NaOH evenly dispersed in the film. Glycerol made the surface smooth and glutaraldehyde made the surface rough. The infrared analysis showed that a cross-linking reaction of alkali lignin and PVA occurred and an ether bond was formed. The reaction was feasible. But the absorption peak corresponding to the ether bond was small. This indicated that the aldol reaction between the lignin and PVA(1788) was weak.

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