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Liquefaction of Banana Pseudo-stem and Preparation of Polyurethane Adhesive from Liquefied Products

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Abstract: Banana pseudo-stem was liquefied in the mixture of polyhydric alcohols of polyethylene glycol (PEG400) and glycerol. Hydroxyl value of liquefied products ranged from 294.8 to 370.2 mg KOH/g and $\overline{M}_n$ was about 430. Liquefied products (LBPP) could be used as raw materials for polyurethane by reacting with 4, 4'-diphenylmethane diisocyanate (4, 4'-MDI) and PEG400 to synthesize liquefied product-based polyurethane (LBPP-PU) adhesive. To analyze in depth the creation of urethane linkage among LBPP , PEG400 and 4, 4'-MDI, factors which had effects on the residue content were all investigated. They were characterized by FT-IR and TG. The shear strength of LBPP-PU adhesive was improved when decreasing the percentage of the substitution of PEG400 by LBPP. The adhesive strength was obtained from T-peel of aspen/polyurethane adhesive joints, and the maximum lap shear strength (4.40 MPa) was obtained when 16.70% of LBPP was added to the LBPP & PEG400 system.

Key words: liquefaction; banana pseudo-stem; polyhydric alcohols; polyurethane adhesive

1 Introduction

Biomass materials include agricultural and forestry residues and they are mainly composed of cellulose, hemicellulose and lignin, which contain two or more hydroxyl groups per molecule basically. The hydroxyl groups have some special chemical properties. Liquefaction of biomass materials is considered as an effective method for the practical utilization of renewable resource in polymer chemistry. Recently, considerable attentions have been given to the preparation of environmentally friendly polymeric products from liquefied biomass materials and their derivatives. Such a liquefaction process is usually carried out in polyhydric alcohols or phenol, sulfuric acid which is used as catalyst. Due to the liquefied products by phenol as liquefied solvent, which can be mostly used to produce phenolic resins and epoxy resins[1,2], researchers prefer to take polyhydric alcohols as liquefied solvent to produce bio-polyol, polyurethane foam and polyurethane films[3-5]. Polyurethane adhesives from renewable sources are greatly appreciated by the researchers since they are cheap and biodegradable as compared to those produced from petrochemical sources[6]. Several publications[7,8] have reported that polyurethane adhesive and poly (vinyl acetate) (PVAC) adhesive are produced from liquefied biomass and the mechanical properties of adhesive meet the actual requirements. Different polymers have been used as adhesives (silicone, polyacrylates, and rubbers), but polyurethanes are the most widely employed adhesives due to their chemical versatility, excellent adhesion properties, weathering resistance, and formulation flexibility.

Banana pseudo-stem is a renewable and abundant biomass material in Hainan province, China. Liquefaction of banana pseudo-stem is one of the processes to utilize agricultural waste and liquefied products could be used as raw materials for polyurethane adhesive. This paper focused on the development of bio-based polyhydric alcohols, and polyurethane adhesive produced from liquefied products and PEG400. To analyze in depth the creation of urethane linkage among LBPP, PEG400 and 4, 4'-MDI, factors which had effects on the residue contents were all investigated. Liquefied product-based polyurethane (LBPP-PU) adhesive was obtained by adding different percentages of LBPP to

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the LBPP & PEG400 system and they were characterized by FT-IR, TG, and T-peel tests.

2 Experimental

2.1 Materials

Banana pseudo-stem (obtained from Haikou, Hainan, China) was dried in oven at 105 °C for 24 h, and polyethylene glycol (PEG400, M_w is 400, chemical purity) and glycerol (Analytical reagent) were from Guangzhou Chemical Reagent Factory. Sulfuric acid (98%) and 1, 4-dioxane were supplied by XiLong Chemical Co., Ltd, China. 4, 4'- diphenylmethane diisocyanate was from Aladdin Industrial Corporation Shanghai, and distilled water was made in laboratory.

Table 1 Components of banana pseudo-stem

Composition	Lignin	Hemicellulose	α-cellulose	Ash
Content/%	23.13	20.20	22.29	16.18

Components of banana pseudo-stem are shown in Table 1. Analysis of the raw materials was conducted according to NREL standard procedures (National Renewable Energy Laboratory, standard Biomass Analytical Procedures).

2.2 Liquefaction of banana pseudo-stem (LBP)

A mixed solvent of PEG400 and glycerol (80/20, *w/w*) was used as the liquefied solvent and sulfuric acid (mmol/g, *n/m*) was used as the catalyst. Banana pseudo-stem, liquefied solvent and sulfuric acid were placed in a three-necked glass flask. The flask was immersed in an oil bath and the working temperature range of the oil bath was from 130 ℃ to 170 ℃. Then the flask was preheated to liquefaction temperature to start the reaction with stirring at 300 rpm. After a preset time, the flask was immersed in a mixture of ice and water to quench the reaction. The resultant products were diluted with 1, 4-dioxane and distilled water (80/20, *v/v*). The insoluble residue was filtered using Buchner funnel, and washed by distilled water thoroughly until the filtrate was colorless. The resultant solid was dried at 105 °C for 24 h in an oven and weighed. Then apparent pH of the filtered solution was adjusted to 5.5 with 1 M potassium hydroxide solution and the potassium sulfates precipitated were removed using the membrane filters. 1, 4-dioxane and distilled water were evaporated under reduced pressure until the moisture content was approximately 1.0%. And the remained solution was called liquefied product (LBPP).

The residue content *R* expressed by percentage is defined as follows:

$$R(\%) = \frac{W_i}{W_o} \times 100 \qquad (1)$$

where, W_i is the dry weight of the resultant solid, and W_o is the dry weight of banana pseudo-stem.

2.3 Measurement of hydroxyl number and acid number

The hydroxyl number and acid number of LBPP were determined according to Hassan and Shukry[9] as follows: a mixture of 1g LBPP and 25 mL of a phthalation reagent was heated for 20 min at 110 ℃. This was followed by the addition of 50 mL of 100% 1,4-dioxane and 25 mL of distilled water, and the mixture was titrated with a 1 M potassium hydroxide solution to the equivalent point using a pH meter. The phthalation reagent consisted of a mixture of 150 g phthalic anhydride, 24.2 g imidzaol and 1000 g dioxane. Hydroxyl number of LBPP was calculated by using the following equation:

$$\text{Hydroxyl number (mg KOH/g)} = \frac{(C-B)\times N \times 56.1}{W} + \text{acid number} \qquad (2)$$

where, *A* is the volume of the potassium hydroxide solution, required for titration of LBPP sample (mL), *B* is the volume of blank solution (mL), *N* is the normality of the potassium hydroxide solution and *W* is the weight of LBPP (g).

Acid number of LBPP was calculated by using the following equation:

$$\text{Acid number (mg KOH/g)} = \frac{(C-B)\times N \times 56.1}{W} \qquad (3)$$

where, *C* is the titration volume of the potassium hydroxide solution at the equivalent point (mL), *B* is the volume of blank solution (mL), *N* is the normality of the potassium hydroxide solution and *W* is the weight of LBPP.

2.4 Synthesis of polyurethane adhesive

LBPP and PEG400 were dehydrated under vacuum at 120 ℃ until bubble disappeared. Polyurethane adhesive was prepared through the reactions among LBPP, PEG400 and 4, 4'-MDI (Fig.1 and Fig.2). The substitution of PEG400 by LBPP was at different levels (16.70%, 33.30%, 50.00%, 66.70%, 83.30%, and 100.00%). The formulations were performed in a four-neck glass reactor equipped with a PTFE stirrer, thermometer, oil bath, and nitrogen gas inlet system. LBPP and PEG400 were poured into the reactor in the atmosphere of nitrogen and heated to 50 ℃. An over-

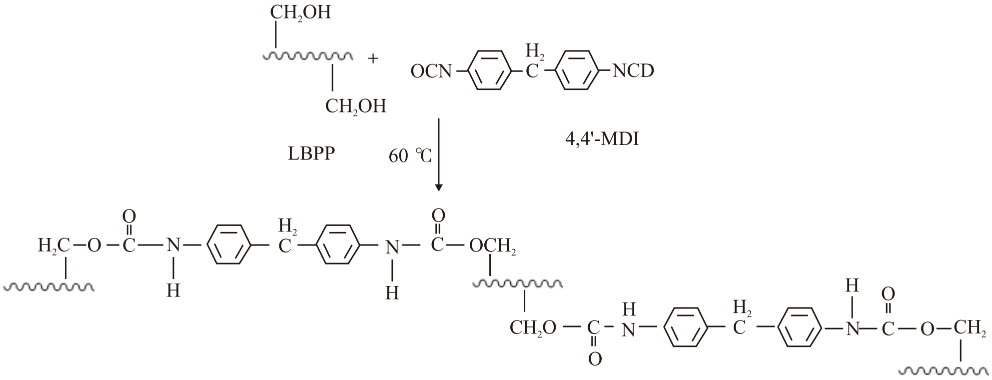


Fig.1 LBPP and prepolymer adhesive reaction with 4,4’-MDI

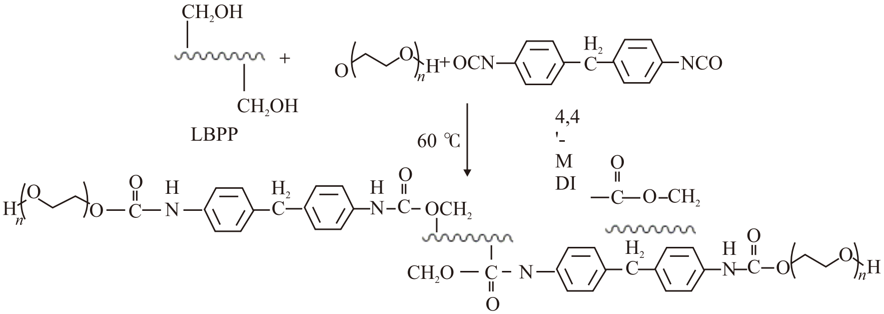


Fig.2 LBPP with PEG400 and prepolymer adhesive reaction with 4,4’-MDI

dose of 4, 4’-MDI was introduced with continuous stirring for 3 h at 60 ℃. With the increase of viscosity of the system during synthesis of polyurethane, certain amounts of acetone were needed to add. The reaction was stopped by cooling the bath and the prepolymer was stored in a sealed glass bottle in nitrogen when the preset time arrived.

2.5 Wood sample preparation and wood bonding testing

The aspen wood pieces were cut into strips (100 mm×20 mm×5 mm) according to the ASTM 906 requirements. They were polished with sandpaper of grit, while the wood was conditioned and stored at room temperature (25 ± 2 ℃) and air humidity (70%-80%).

Wood bonding was tested on aspen wood specimens and urethane prepolymer was put on one piece of the wood strip with a knife. Area of the gum on top of one piece of the wood strip was 20 mm × 30 mm. A load (7.5 kg/cm^2) was placed over the joint sand left for 24 h at room temperature (25 ± 2 ℃) and air humidity (70%-80%) to allow the prepolymer to cure. Each wood joint specimen was tested for lap shear strength using a single arm electronic universal tensile testing machine (Beijing guance jingdian electric equipment Co., Ltd) after 7 days at room temperature and air humidity. Across head speed of 50 mm/min was used in this test, five replicates were tested and the standard deviation was lower than 10%.

2.6 Characterization

2.6.1 FTIR analysis

Fourier transform infrared spectroscopy was carried out on a FTIR spectrophotometer (TENSOR 27, Bruker, Germany) using the KBr disk method. Spectra were acquired in a range of 500-4 000 cm^{-1} performing 16 scans at 4 cm^{-1} resolution.

2.6.2 Thermogravimetry

Thermogravimetric analysis of sample was conducted in a temperature interval of 40-800 ℃ in nitrogen atmospheres with a flow rate of 20 mL/min using an alumina crucible with a pinhole. A constant heating rate of 15 ℃/min was maintained. A NETZSCH5 TG 209F3 thermal analyzer was used for this study.

3 Results and discussion

3.1 Effect of liquefaction conditions on the residue content

3.1.1 Effect of temperature and time on the residue content

Banana pseudo-stem was liquefied by the reaction between PEG400 and glycerol under acidic condition. The black, highly viscous liquefied products were considered as polyhydric alcohols. Because they contained lots of hydroxyl groups, they could originate from the banana pseudo-stem and co-solvent. In this work researchers first examined the effect of liquefaction time on residue content, and then focused on the liquefaction temperature. The rapid liquefaction stage was mainly dependent on the components of lignin and hemicelluloses, whereas cellulose mainly affected the slow liquefaction stage in which the residue decreased slowly. Although banana pseudo-stem has more lignin, the residue content had not changed obviously from 30 min to 150 min at the temperature of 130 ℃ to 170 ℃ (Fig.3). High residue contents (46.50%, 45.60%, 45.20%, 40.00%, and 40.40%, respectively) were obtained at 130 to 170 ℃ for 30 min. As temperature rose, the liquefaction proceeded rapidly and liquefaction rate obtained at the maximum time also was increased. However, as liquefaction time was prolonged, the change of residue content was gently slow. That could be the contribution of easy liquefied compositions degraded completely, so as the time was prolonged, the curve became flat[10]. Under the condition of 170 ℃ and 120 min, the residue content was 34.50%, while extending liquefaction time for another 30 min, the residue content was 35.20%, which was higher than previous residue content of 120 min, implying that recondensation of components from degraded banana pseudo-stem (polysaccharide and lignin) had occurred[11]. With liquefaction time going on, the residue content became lower and had gentle changes during liquefaction process. Therefore, 90 min was considered to be the best liquefaction time for this study.

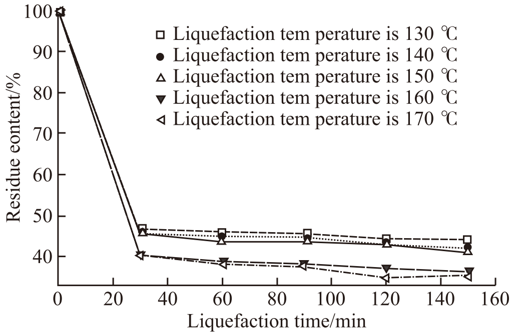


Fig.3 Effect of temperature and time on the residue content (liquid-solid ratio: 6; the amount of catalyst: 1 mmol/g)

3.1.2 Effect of the amount of catalyst on the residue content

Fig.4 shows the changing trend of residue contents in liquefaction of banana pseudo-stem with different amounts of catalyst. It is apparent that increasing the amounts of catalyst in liquefaction will increase the liquefaction yield. The residue content was 68.80% without catalyst, while the catalyst was 1 mmol/g (sulfuric acid/banana pseudo-stem, *n/m*), the residue content was 44.00%. Using sulfuric acid as the catalyst could make liquefaction faster compared with that without catalyst. Comparing with residue content remained in 90 min, it is indicated that residue content reflected the speed of liquefaction. When the amount of catalyst was 3.0 mmol/g, the residue content was only 19.00%, which could be attributed to the ash content in banana pseudo-stem. Moreover, the ash content of biomass affects the overall biomass conversion[12]. Becuase the used sulfuric acid must be removed after the liquefaction process by a neutralization process and a high acid concentration accelerated the condensation of lignin as well as substrate liquefaction, the amount of catalyst of 1 mmol/g was selected.

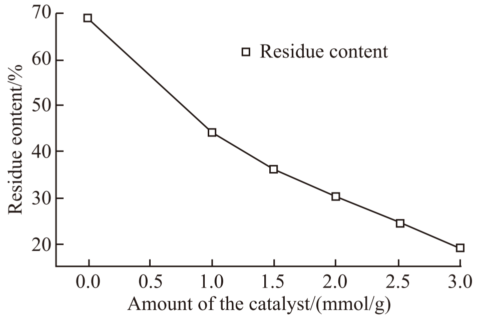


Fig.4 Effect of the amount of catalyst on the residue content (temperature: 150 ℃; time: 90 min; liquid-solid ratio: 6)

3.2 Changes of hydroxyl number and acid value in liquefied products

In this work, the -OH group content was one of the most important factors in the preparation of PU[13]. Hydroxyl number of liquefied product ranged from 294.8 to 370.2 mg KOH/g, which was suitable for the preparation of polyurethane adhesive. The hydroxyl number and acid value of LBPP were influenced by many factors, including liquefaction temperature, reaction time, acid content and liquid-solid ratio[14]. Under the same conditions of liquid-solid ratio, liquefaction temperature and amount of the catalyst used, hydroxyl number and acid number were discussed.

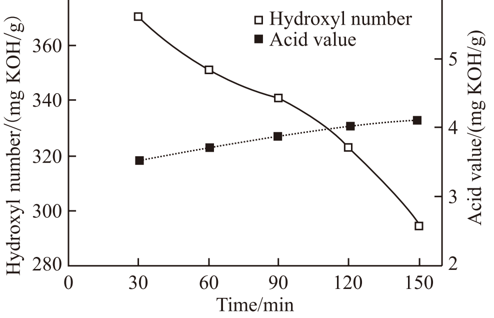


Fig.5 Effect of time on the hydroxyl number and acid value (temperature: 150 ℃; liquid-solid ratio: 6; amount of catalyst: 1 mmol/g)

Fig.5 shows the time dependence of the hydroxyl and acid number curves of LBPP. The hydroxyl number of LBPP decreased as the time was prolonged during liquefaction, but the acid number of LBPP was increased, which indicated that the acidic substance contents of LBPP were increased during liquefaction. It could be attributed to the depolymerized banana pseudo-stem components or the oxidation of the saccharides during the liquefaction[15]. The hydroxyl groups, in both the decomposed banana pseudo-stem fragment and polyhydric alcohols would react with each other to form a derivative with an ether bond and then dissolve in the remained free polyhydric alcohols with time going by.

3.3 Determination of the effect of LBPP on adhesive performance

These polyhydric alcohols were combined with an aromatic adduct based on 4, 4'-diisocyanate to form a PU adhesive. The adhesive performance of the PU adhesive in bonding wood was evaluated by lap shear tests. The ratio of NCO/OH and the hydroxyl number kept constant. The ratio of NCO/OH was 1.3 and hydroxyl number was 311.95 mg KOH/g.

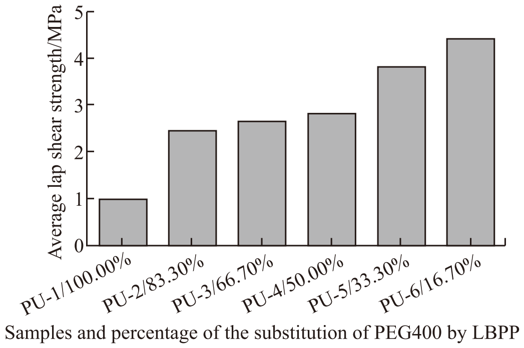


Fig.6 Effect of different formulations on adhesive of average lap shear strength

Fig.6 shows the inverse relationship between the percentage of LBPP in the synthesis of polyurethane adhesive and the average lap shear strength. When the percentage of the substitution of PEG400 by liquefied product was 100.00% in the synthesis of polyurethane adhesive, the average lap shear strength got the lowest value (1.00 MPa). The average lap shear strength value (4.40 MPa) peaked as the percentage of the substitution of PEG400 by liquefied product was 16.70%. That was assigned to liquefied product as the "polyhydric alcohols" mixed with PEG400, containing some small molecules, which made the molecular weight of polyurethane adhesive relatively low and were the main components of segments for polyurethane adhesive.

3.4 FT-IR spectra

There was a strong and broad absorption band at 3 000-3 500 cm^{-1}, which is attributed to the hydroxyl groups in the co-solvent and liquefied product. The absorption bands of 2 912 cm^{-1} and 2 877 cm^{-1} for co-solvent and liquefied product, respectively, were attributed to the C-H and CH_2- vibrations of aliphatic hydrocarbons[16]. The absorption intensity of C-H and CH_2- in liquefied product was increased significantly after liquefaction, suggesting that PEG400 had been introduced into the structure of liquefied product. The absorption bands at 1 954 cm^{-1} and 1 956 cm^{-1} were due to the ether bond of PEG400 and LBPP. The absorption band of 1 732 cm^{-1} was the stretching vibration of carbonyl group that belongs to acetyl groups or carboxyl groups, the appearance of the absorption suggested that some of cellulose, hemicellulose, and lignin were oxidized and formed a new aldehyde group or carboxyl group in LBPP. The bending vibration absorption of 1 651 cm^{-1} and 1645 cm^{-1}, respectively, indicated the absorbed water in co-solvent and LBPP. The peaks observed at 1 457cm^{-1}, 1 352 cm^{-1}, 1 456 cm^{-1}, and 1 350 cm^{-1} in co-solvent and LBPP were assigned to the CH_2- bending and -CH_2 bending, respectively.

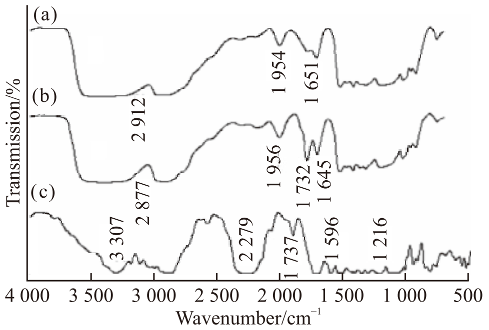
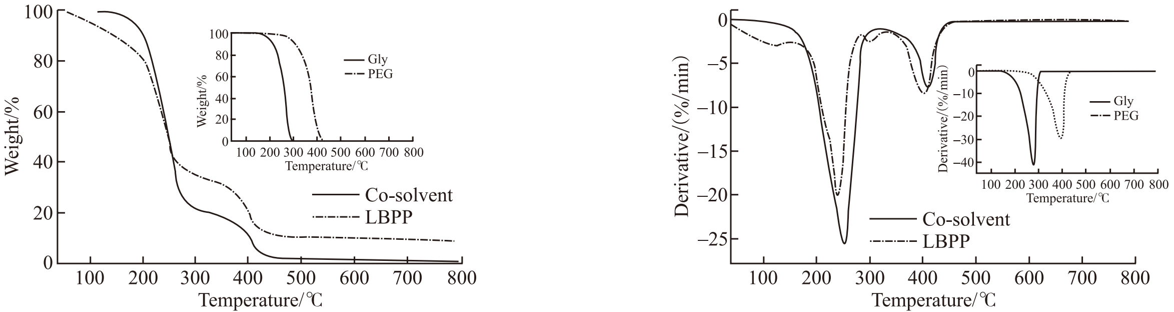
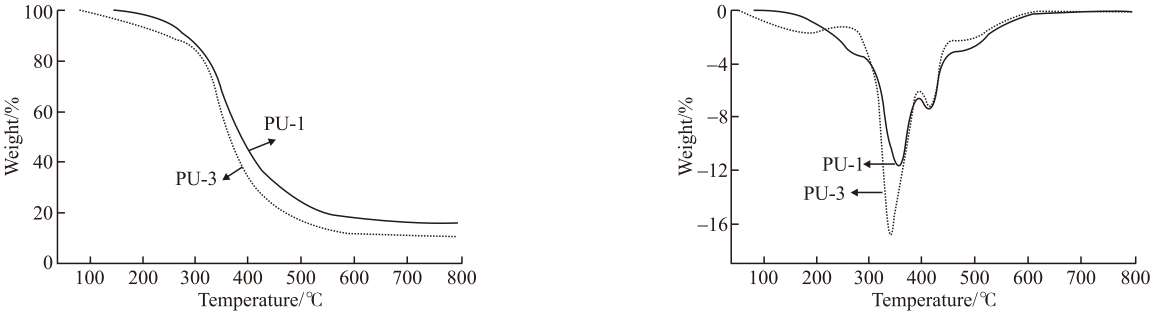


Fig.7 FT-IR spectra of co-solvent (a); LBPP (b); LBPP-PU adhesive (c)

The formation of LBPP-PU adhesive could be seen with the emergence of a new shoulder peak at 3 307 cm^{-1} near the hydroxyl (-OH) band of LBPP (Fig.7(c)). This new peak could be attributed to the – NH stretching of the urethane linkage, indicating that a reaction had occurred in this system. It was studied that infrared band intensities had developed at bands 3 307, 1 737 and 1 216 cm^{-1}, suggesting that some reactions between isocyanate and hydroxyl groups had happened[7] and urethane groups formed. The absorption peaks resulting from -NH stretching and bending vibrations were observed at 3 307 and 1 540 cm^{-1}[17], respectively. The peaks located at 2 919 cm^{-1} were assigned to the -CH symmetric and asymmetric stretching vibra-



(a) TG curves of co-solvent and LBPP (b) DTG curves of co-solvent and LBPP



(c) TG curves of LBPP-PU adhesive (d) DTG curves of LBPP-PU adhesive

Fig.8 The inset figures are graphs of pure glycerol, PEG400

tions of -CH_2 groups. The absence of peaks observed at 2 279 cm^{-1} indicated the presence of unreacted NCO in the higher ratio of NCO/OH in sample. The peaks observed at 1 737 and 1 596 cm^{-1} were due to C=O and C=C bonds of -$NHCOO^{-}$ and 4,4'-MDI, respectively. It was indicated clearly that the formation PU existed according to the absence of -OH (Figs.7(a) and 7(b)) absorbance band associated with the LBPP and the emergence of a-NH (Fig.7(c)) of LBPP-PU adhesive.

3.5 Thermogravimetry

Specimens were thermo-gravimetrically analyzed to give the consequences arising from the heating and sequent pyrolytic degradation of LBPP and co-solvent (Figs.8(a) and 8(b)). All the peaks observed were due to the decomposition or depolymerization of LBPP and co-solvent. The peaks ranging from 148 °C to 287 ℃ and from 287 ℃ to 419.5 ℃ observed in the TG curve of co-solvent were caused by the decomposition of glycerol and PEG400, respectively[5]. During the rapidly liquefied stage, like lignin and amorphous region of cellulose reacted with co-solvent[18], explaining the peaks observed compared with co-solvent, a different peak ranging from 237 ℃ to 292 ℃ observed in the TG curve of LBPP, and there are two main losses at 164 ℃ to 280 ℃ and 340 ℃ to 450 ℃.

The thermal properties of LBPP-PU adhesive were investigated by TG analysis (Figs.8(c) and 8(d)). The percentages of the substitution of PEG400 by LBPP were 66.70% of LBPP-PU-3 and 100% of LBPP-PU-1. When the temperature of the thermal decomposition of polyurethane was about 200 ℃[19], urethane molecular backbone and substituted urea first broke at C-O and then broke into the hard and soft segments, namely isocyanate and polyol, and hard and soft segments were further pyrolysised. Fig.8(c) indicates that the thermal stability of PU-1 is higher than that of PU-3. It can be indicated that the thermal stability of LBPP-PU is improved when the percentage of the substitution of PEG400 by LBPP is high. Because the thermal stability of LBPP is higher than that of PEG400, there is degradation of banana pseudo-stem into LBPP, the thermal degradation of wood can be represented as a sum of the thermal degradations of the individual wood constituents[5] and the main constituents of banana pseudo-stem are similar to wood. Therefore, it is assumed that the dissolved constituents of banana pseudo-stem resulted in the increase of thermal stability of LBPP compared with co-solvent. Fig.8(d) shows that different PUs have different initial broken down temperatures, and the initial broken down temperatures of PU-1 and PU-3 are 291.6 ℃ and 293.2 ℃, respectively. The peaks of PU-1 and PU-3 at 357.9 ℃ and 337.1 ℃, respectively, are caused by the decomposition of isocyanate in urethane polymer. This is based on the report that the isocyanate decomposition of urethane elastormer showed peaks ranging from 290 ℃ to 370 ℃ by the analysis of DSC thermo grams[20]. As can be seen in Fig.8(d), the loss rate of mass of PU-3 is higher than that of PU-1, therefore, the thermal stability of PU-1 was higher than that of PU-3, because the percentage of the substitution of PEG400 by LBPP was raised, and the thermal stability of LBPP-PU was increased.

4 Conclusions

Banana pseudo-stem was liquefied by the mixture of PEG400 with glycerol. Temperature, liquid-solid ratio and amount of catalyst all have effects on the residue contents. With increase temperature, liquid-solid ratio and amount of catalyst, the residue contents are decreased. With prolonging time, hydroxyl number is decreased while acid number is raised. Liquefied products were used as the raw material for the partial substitution of PEG400 to produce LBPP-PU adhesive by reacting with 4, 4’- MDI. Lap shear strength of LBPP-PU adhesive is decreased as the percentage of the substitution of PEG400 by LBPP is increased, while the thermal stability is improved.

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