

Synthesis of Polyurethane Acrylates by Hydrogenated Castor Oil and Dimer-Based Polyester Diol and Study on Pressure-Sensitive Adhesive

Yaobin Ren,¹ Huiming Pan,¹ Longsi Li,² Jianming Xia,² Yongqiang Yang²

¹Institute of Materials Science and Engineering, South China University of Technology, Guangzhou 510640, People's Republic of China

²Zhongshan New Asia Adhesive Product Co. Ltd., Lianfeng Road, Xiaolan Town, Zhongshan, Guangdong 528415, People's Republic of China

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ABSTRACT: Synthesis of polyurethane acrylate (PUA) and preparation of the UV-cured pressure-sensitive adhesives (PSA) are reported. Molecular weight (M_w) (by gel permeation chromatography) and viscosity (η^*) of PUA were measured. Characterization of PUA and PSA before and after UV-curing was made by FTIR. Increase of the hydroxyls from hydrogenated castor oil/hydroxyls from dimer-based polyester diol ($\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$) ratio decreased the M_w and η^* value of PUA. Dynamic viscoelastic properties (by dynamic rheological spectrometer) and performance of the UV-cured PSA were also studied. Increase of the $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ ratio increased the storage modulus (G'), the loss modulus (G''), and complex viscosity (Eta^*) of the UV-cured PSA, which, in turn, enhanced holding power

and shear adhesion failure temperature (SAFT) and yet decreased peeling strength. Substitution of OB for DBTDL depressed the M_w and η^* value of PUA, while the G' and Eta^* values of the UV-cured PSA were elevated, which, in turn, increased the holding power and SAFT and yet depressed the peeling strength. Elevation of the tackifying resin content depressed the G' , G'' , and Eta^* values of the cured PSA and yet increased glass transition temperatures (T_g) of PSA, measured by differential scanning calorimetry. Peeling strength of PSA elevated as increasing the tackifying resin, while the holding power and SAFT fell. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 98: 1814–1821, 2005

Key words: adhesives; viscoelastic properties; polyurethane

INTRODUCTION

Ultraviolet-curable pressure-sensitive adhesives (PSA) gained wide industrial attraction because of their reduction in volatile organic compounds emissions, faster curing, versatility, energy efficiency, cost benefits, and high properties such as heat resistance, shear resistance, and chemical resistance. Therefore, UV-curable PSAs would be expected to erode the PSA's conventional market share and develop a unique market position.^{1–3}

Dimer acid was obtained by the conversion of unsaturated fatty acids by a combination of pressure, temperature, and catalyst. The characteristic of dimer acid, highly branched and saturated hydrocarbon, provided polyester polyols with properties such as hydrophobicity, hydrolytic resistance, and good thermo-oxidative stability. This allowed the design of a unique class of polyurethane acrylate (PUA): low glass transition temperature (T_g), flexibility, hydro-

lytic and thermo-oxidative stability, high adhesion both to low-surface-energy adherends and to high-surface-energy adherends, and excellent flow properties with efficient substrate wetting.⁴ Hydrogenated castor oil (HCO) was used for PUA for its flexibility, good flow properties, and lower cost.⁵

This study aimed at synthesizing PUA with dimer-based polyester diol and HCO and preparing UV-curable PSA. Measurements of molecular weight (M_w) and viscosity of PUA, dynamic viscoelastic properties, and thermal properties of UV-PSAs were made. The performance of UV-cured PSA, i.e., peel strength, holding power, and SAFT was also tested. The effect of the $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ ratio on dynamic viscoelastic properties and performance of PSA, i.e., the peel strength, HP, and SAFT, were studied. The effect of the concentration of A-100 resin to PUA on the dynamic viscoelastic properties and performance of PSA was also investigated.

The structure of dimer acid, dimer-based polyester diol, and HCO is shown in Figure 1.

EXPERIMENT

Raw materials

Isophorone diisocyanate (IPDI, Aldrich ChemicalsA), organobismuth (OB) (COSCAT 83, produced by Cas-

Correspondence to: Y. Ren (rybhn@163.com).

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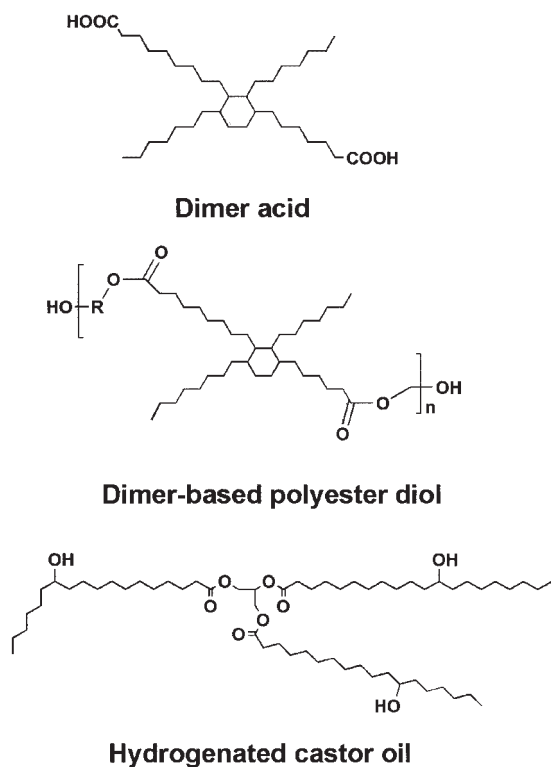


Figure 1 Structure of dimer acid, dimer-based polyester diol, and HCO.

chem), and dibutyltin dilaurate (DBTDL, Junsei Chemical) were used as received. Dimer-based polyester diol (Priplast 1838, Uniqema, The Netherlands) and HCO (Tongliao Tonghua Castor Chemical) were degassed at 120 °C for 2 h under vacuum. 2-Hydroxyethyl acrylate (HEA, International Specialty), 2-phenoxy ethyl acrylate (EM210, Eternal Chemical), and 2(2-ethoxy) ethyl acrylate (EM211) were kept dry with a 4-Å molecular sieve before use for 1 week. Norsolene A-100 resin (C5/C9; melting point, 100 °C), 2,4,6-trimethyl-benzoyl diphenylphosphine oxide (Lucirine TPO, BASF) and 1-hydroxycyclohexylphenyl ketone (Irgacure 184, Ciba-Geigy) were used without further purification.

Synthesis proceeding of PUA

The synthesis of PUA was carried out in a four-neck glass reactor equipped with a stirrer, a reflux con-

denser, thermometer, addition funnel, and nitrogen gas let system. In the first step, dimer-based polyester diol dissolved in the mixed diluents from EM210 and EM211 (weight ratio, 1 : 1) was poured into the glass reactor after nitrogen gas was aspirated for 15 min to eliminate the residual moisture. IPDI was dropped into the reactor slowly at 50 °C. The mixture was stirred quickly for 10 min. Several drops of catalyst were dropped into the reactor slowly. The reaction temperature was raised and controlled below 70 °C for 2 h. The change of NCO value during reaction was determined using the dibutylamine back titration method to find out the end point of the reaction.

In the second step, after the temperature was raised to 80 °C, HCO was added into the reactor and reacted for 3 h using the same method as the first step. In the last step, HEA was dropped into the reactor and reacted with the residual NCO groups below 80 °C for 4 h, capping both molecular ends with vinyl groups. When the amount of remaining isocyanates in this reactive system was less than 0.1 wt %, the temperature was raised to 90–95 °C for 0.5 h to eliminate the residual NCO group. A stream of dry nitrogen gas was led into the flask and maintained throughout the reaction. The formulations of PUA are shown in Table I.

Formulations and curing of UV-PSA

Preparation of UV-cured PSA samples for performance testing

PUA was blended with A-100 resin at 80 °C. After the solution became uniform and transparent, others ingredients were added. Films (24–26 μm thick) of the solution were cast on a polyethylene terephthalate (PET) film and covered with silicone-containing release paper. The adhesive films were then exposed to a 1000 W/cm² medium-pressure mercury vapor lamp of UV-2KW-2–35 curing system (Lantian Special Lamp, China) at 10 m/min for two passes, which ensured complete cure of the PUA evidenced by the lack of any syrupy liquid on the adhesive surface. The samples were kept at 110 °C for 24 h in an air-circulation oven to remove the uncured diluents from the samples. The formulations of UV-curable compositions are shown in Table II.

TABLE I
Compositions and Viscosity (cp) of PUA (mol)

Symbol	OH _{Diol} ^a	OH _{HCO} ^b	HEA	IPDI	Catalyst	Diluent (g)	OH _{Diol} /OH _{HCO}	M _w (g/mol)	Viscosity (cp)
P-1	0.08	0.05	0.03	0.08	DBTDL	20	8/5	113709	8050
P-2	0.09	0.04	0.03	0.08	DBTDL	20	9/4	92428	7300
P-3	0.1	0.03	0.03	0.08	DBTDL	20	10/3	80018	6800
P-4	0.1	0.03	0.03	0.08	OB	20	10/3	21161	5500

^a The hydroxyls mole value from dimer-based polyester diol.

^b The hydroxyls mole value from hydrogenated castor oil.

TABLE II
Compositions of UV-Curable PSA for Performance Test (g)

Sample	PUA		A-100	211	184	Addition
	Symbol	Weight				
A-1a	P-1	10	1.0	1	0.3	0.5
A-2a	P-2	10	1.0	1	0.3	0.5
A-3a	P-3	10	1.0	1	0.3	0.5
A-4a	P-4	10	1.0	1	0.3	0.5
A-5a	P-3	10	0	1	0.3	0.5
A-6a	P-3	10	1.5	1	0.3	0.5
A-7a	P-3	10	2.0	1	0.3	0.5

Preparation of UV-cured PSA samples for dynamic viscoelastic properties measurement

Films ($\approx 100 \mu\text{m}$ in thickness) of the UV-curable compositions, shown in Table III, were cast on a silicone-containing release paper and then were exposed to UV radiation for four passes at a speed of 10 m/min. The samples were kept at 110 °C for 24 h in an air-circulation oven to remove the uncured diluents from the samples. Several pieces of samples (25 × 25 mm) were carefully overlapped to avoid air bubble between samples and form a sheet (0.5–0.7 mm in thickness).

Measurement of viscosity

The viscosity of PUA was measured using a Brookfield synchroelectric viscometer, Model RVRH, at 50 °C and 1 s^{-1} , and are listed in Table I.

FTIR measurements

FTIR spectra of PUA after being degassed under vacuum and PSA before UV irradiation and FTIR-ATR spectra of PSA after UV irradiation were collected on a Nicolet Magna-IR 860 spectrometer (Thermo Nicolet Japan, Kanagawa, Japan) using a potassium bromide pellet (Fig. 3). FTIR-ATR spectra of PSA after UV curing were also collected on the above-mentioned spectrometer. An ATR accessory (Spectra-Tech) with a

TABLE III
Compositions of UV-Curable PSA for Dynamic Viscoelastic Properties Measurements (g)

Sample	PUA		A-100	211	184	TPO	Addition
	Symbol	Weight					
A-1	P-1	10	1.0	1	0.1	0.1	0.5
A-2	P-2	10	1.0	1	0.1	0.1	0.5
A-3	P-3	10	1.0	1	0.1	0.1	0.5
A-4	P-4	10	1.0	1	0.1	0.1	0.5
A-5	P-3	10	0	1	0.1	0.1	0.5
A-6	P-3	10	1.5	1	0.1	0.1	0.5
A-7	P-3	10	2.0	1	0.1	0.1	0.5

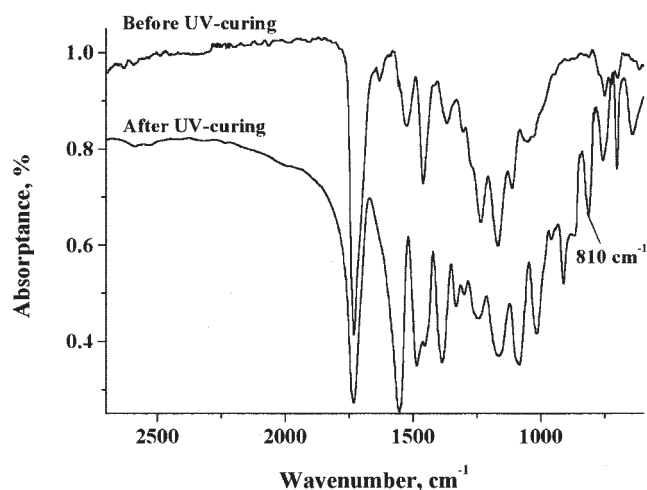


Figure 2 FTIR spectra for PSA before and after UV-curing (A-3).

zinc selenium crystal for a 45° angle of incidence was used. Dry air was purged continuously in these instruments.

Gel permeation chromatography (GPC)

A Model 410 GPC equipped with Styragel HR 1–4 columns (Water Associates) measured molecular weight distributions at 25 °C. The flow rate of the carrier solvent, THF, was 0.5 mL/min. The molecular weight measured for PUA samples is listed in Table I.

Differential scanning calorimetry (DSC)

DSC measurement was done on a NETZSCH DSC 204. Scans were run at 10 °C/min heating rates, using approximately 10-mg samples in crimped aluminum pans. The T_g was calculated both at half height and at the inflection point.

Dynamic viscoelastic properties

Dynamic viscoelastic properties of the blends were determined by a dynamic rheological spectrometer RDA-III (Rheometrics) in the parallel plate mode (25-mm diameter plates with a 0.5–0.7). From angle frequency (ω) scans from 0.1 to 500 rad/s at a fixed temperature of 30 °C, the G' , G'' , and Eta^* were evaluated as a function of ω .

UV-cured PSA performance

The 180° peel strength was evaluated following Pressure Sensitive Tape Council Test Method Number 1 (PSTC-1)⁶ test procedure in which a strip of 25-mm-width adhesive tape was pasted onto a stainless-steel panel (SUS-430BA, 50 × 100 mm²) and peeling off at a

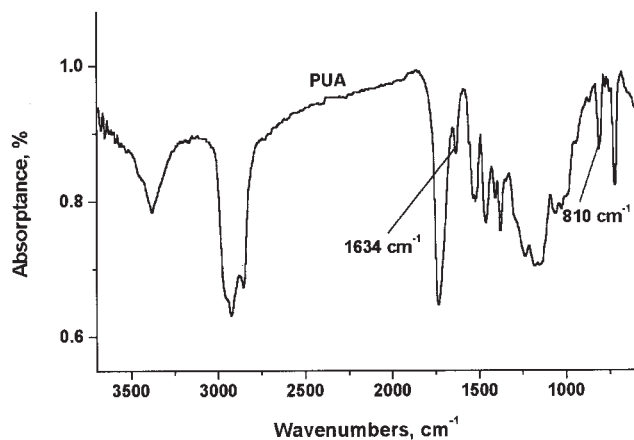


Figure 3 FTIR spectra for and PUA (P-1).

crosshead speed of 300 mm/min by using a Stograph-RI (Toyoseiki) after pasting and pressing with a 2.0-kg rubber roller. All specimens were placed at 25 °C and 65% relative humidity for 20 min before testing. Three replicates were tested for each condition.

The holding power measurements were conducted via the Pressure Sensitive Tape Council Test Method Number 7 (PSTC-7)⁶ test procedure in which a strip of 25 × 25 mm² adhesive tape was applied to a stainless-steel surface mounted in a vertical position after which a 500-g weight was attached to the tape. The time to slip completely (holding time, hours) was recorded as the failure time. The panels were cleaned with acetone and then with trichloroethylene. Before testing the cured PSA tapes were pressed onto those substrates by a 2-kg rubber roller passing over twice after being seasoned at 25 °C and 65% relative humidity. Three replicates were tested for each condition.

Shear adhesion failure temperature (SAFT)

The 20 × 20 mm² adhesive sample on 25 μm PET was placed on a solvent-cleaned stainless-steel panel and rolled down with a standard 2-kg roller. The samples sat for 30 min at 25 °C and 65% relative humidity and placed in a 70 °C oven. A 500-g weight was suspended from each sample. After 20-min equilibration, the timer was started and the oven temperature was increased at a rate of 0.4 °C/min to a final temperature of 175 °C. The failure temperature of each sample and mode of failure was noted if it failed. Three replicates were tested for each condition.

RESULTS AND DISCUSSION

FTIR spectra

FTIR spectra of PUA (P-1) are shown in Figure 2. The PUA exhibited the characteristic absorption peaks at about 810 cm⁻¹ (C=C band), 1633 cm⁻¹ (C=C bond),

1523 cm⁻¹ (N-H bending and C-N stretch), and 1723 cm⁻¹ (C=O stretch). No absorption was observed at 2270 cm⁻¹ (NCO bond), indicating that the NCO group was completely reacted. The FTIR spectra of PSA before and after UV curing, shown in Ref. ³ showed that the band at 810 cm⁻¹ disappeared completely, implying that the vinyl polymerization reaction occurred successfully.

Molecular weight and viscosity of PUA

Molecular weight and viscosity of PUA are shown in Table I. Table I shows that, with increased OH_{HCO}/OH_{Diol}, the viscosity of PUA and M_w of PUA increased (i.e., P-1 > P-2 > P-3). Because one HCO molecule had 2.75 hydroxyls group on average, one HCO molecule could react with the 2.75 NCO group. Therefore, increasing OH_{HCO}/OH_{Diol} enhanced the probability of branching and crosslinking during the reaction between polyols (HCO and dimer-based polyester diol) and IPDI, which, in turn, resulted in an increase in PUA's viscosity and molecular weight. Although both DBTDL and OB catalyzed the reaction between the NCO group and the OH group, the η^* and M_w value of P-3 was higher than that of P-4. Because the OB catalyst had a high selectivity toward the polyol-isocyanate reaction, the moisture-isocyanate reaction was weakened. Therefore, the reaction between NCO-terminated oligomers was weakened. Thus, the η^* and M_w value of P-4 was lower than that of P-1.⁷

Thermal properties of PSA

The DSC thermograms of PSA samples are shown in Figure 4. With weight concentration of A-100 resin to P-3 increasing from 0 to 20%, the T_g of PSA increased from -39.5 to -28.7 °C. In addition, the PSA exhibited

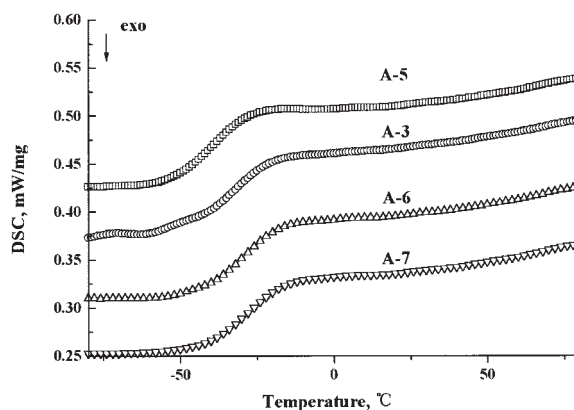


Figure 4 DSC thermograms of PSA-cured samples. Weight concentration of A-100 resin to PUA (P-3): A-3, 10%; A-5, 0; A-6, 15%; A-7, 20%.

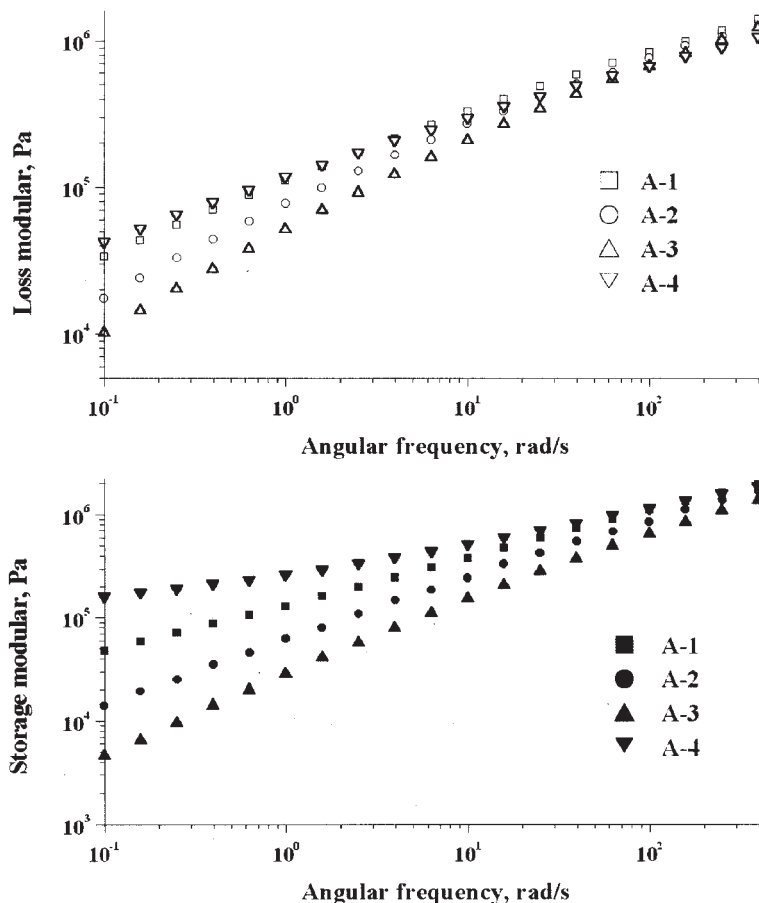


Figure 5 Dynamic storage modulus (G') and dynamic loss modulus (G'') of UV-cured PSA. The $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ ratios of PUA: P-1, 5 : 8; P-2, 4 : 9; P-3, 3 : 10, and A-1, A-2, A-3 and A-4 were formulated with P-1, P-2, P-3, and P-4, respectively.

a single glass transition temperature for all PSA specimens. This was a clear indication that A-100 resin was miscible with P-3 on molecular scale, and that A-100 resin increased the T_g of PSA.

Dynamic viscoelastic properties

The performance of PSA was closely associated with the viscoelastic properties of the bulk adhesives, as well as with the surface energies of the adhesives and adherend.^{8,9} Bonding was a low rate process at low deformation when PSA was pressed onto a surface. The PSA deformation during the bonding stage had to be, in large part, viscous. However, debonding in a tack or peeling strength test had a high rate and the adhesive deformation in the debonding stage was associated with the elastic property of PSA. The shear strength or holding power was a low rate process at high deformation and was influenced by the dynamic viscoelastic properties (such as G' and loss modulus, G'') and other factors, e.g., the friction coefficient between the adhesive and the adherend or cohesive strength.^{10,11} Therefore, the viscoelastic behavior of

PSA bulk was an important factor in controlling its performance.

Effect of $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ for PUA and catalysts on the dynamic viscoelastic properties of PSA

Figure 5 shows the variation in storage modulus and loss modulus of PSA specimens at different angular frequency (ω). With the $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ ratio increased, a significant increase of the storage modulus, especially at low frequency, was found, while the corresponding increase of the loss modulus was much lower, indicating that HCO was of critical importance to enhance the elasticity of UV-cured PSA. HCO had lower molecular weight, 938 g/mol and 2.75 hydroxyls per molecular, implying that increase of $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ resulted in the increase of the hard segment content and elevation of molecular weight as mentioned before, which, in turn, increased the hard segment content and molecular weight of polymer in the UV-cured PSA. This caused enhancement of the elasticity of the UV-cured PSA.

Figure 5 also shows that $G'_{\text{A-3}}$ was much lower than $G'_{\text{A-4}}$, especially at low frequency, whereas the differ-

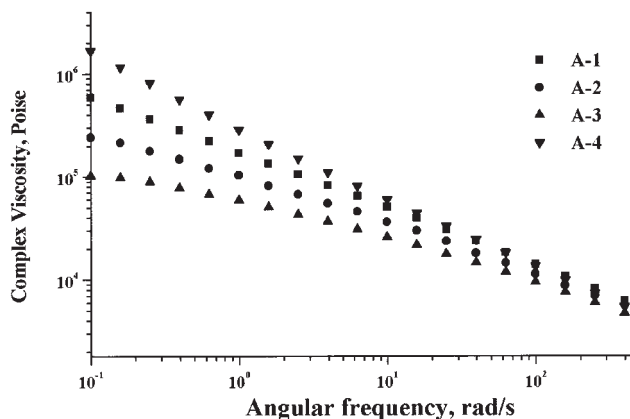


Figure 6 Complex viscosity of UV-cured PSA. The $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ ratios of PUA: P-1, 5 : 8; P-2, 4 : 9; P-3, 3 : 10, and A-1, A-2, A-3, and A-4 were formulated with P-1, P-2, P-3, and P-4, respectively.

ence between G''_{A-3} and G''_{A-4} was not so much and even G''_{A-4} was larger than G''_{A-3} when $\omega > 2.51 \text{ rad/s}$. These phenomena indicated that catalyst for preparation of PUA was another important effect factor. Obviously it was very different when moisture was eliminated completely. With organic bismuth as catalyst,

the NCO group was nonsensitive to moisture during synthesis of PUA, which depressed both the reaction between NCO-terminated oligomer and water and that between NCO-terminated oligomer.⁷ Therefore, more NCO group reacted with HEA and the vinyl group number increased, which, in turn, improved the crosslinking degree of the polymer in the cured PSA and enhanced the elasticity.

Master curves (at 30 °C) of complex viscosity (Eta^*) for PSA specimens, i.e., A-1, A-2, A-3, and A-4, are shown in Figure 6. The Eta^* value of UV-cured PSA, especially at low frequency, increased dramatically with increasing $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ ratio. The Eta^*_{A-3} value was higher than the Eta^*_{A-4} value by about 10 times at low frequency, 0.1 rad/s. Therefore, conclusions could be made that both the $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ ratio and the kind of catalyst are important factors affecting the complex viscosity of the UV-cured PSA.

Effect of concentration of A-100 resin to PUA on the dynamic viscoelastic properties of PSA

Master curves (at 30 °C) of dynamic viscoelastic properties for PSA specimens, i.e., A-3, A-5, A-6, and A-7, are shown in Figures 7 and 8. Upon increasing the

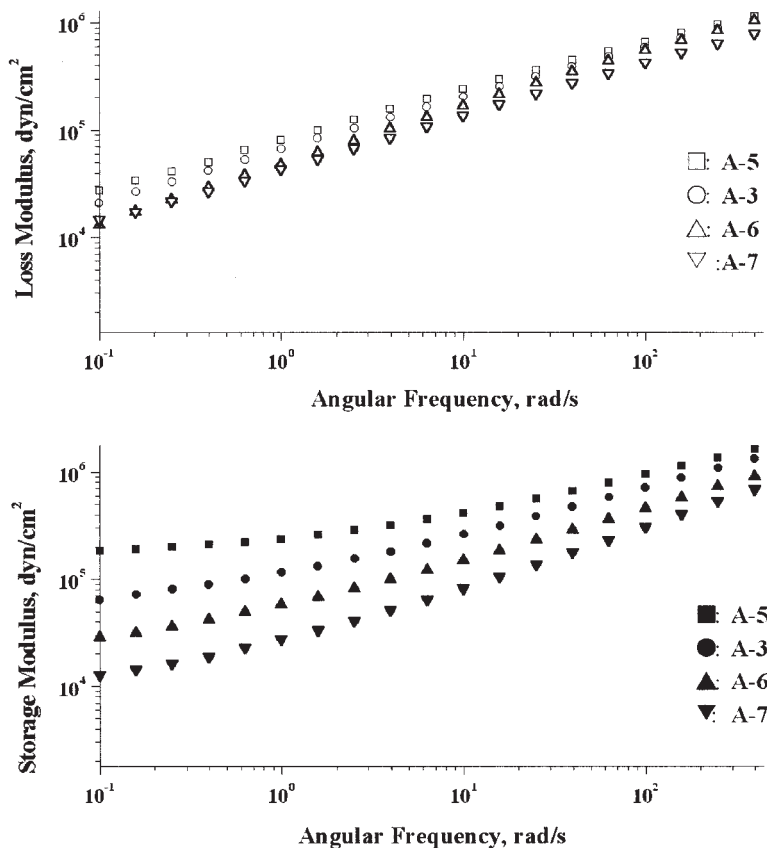


Figure 7 Dynamic storage modulus (G') and dynamic loss modulus (G'') of UV-cured PSA. Weight concentration of A-100 resin to PUA (P-3): A-3, 0.1; A-5, 0; A-6 0.15; A-7, 0.2.

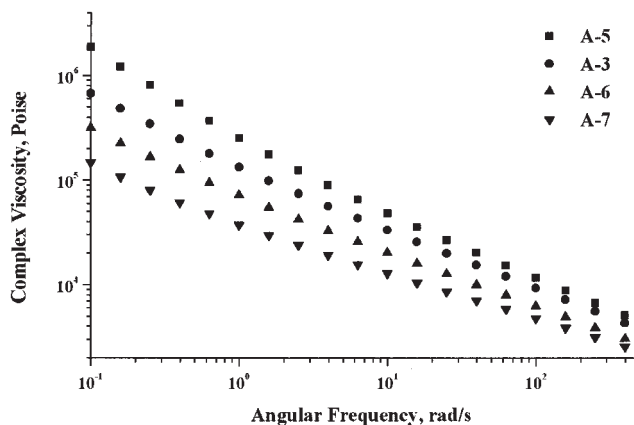


Figure 8 Complex viscosity of UV-cured PSA. Weight concentration of A-100 resin to PUA (P-3): A-3, 0.1; A-5, 0; A-6 0.15; A-7, 0.2.

A-100 resin concentration from 0 to 20%, the storage modulus of the UV-cured PSA at low frequency 0.1 rad/s fell dramatically from about 2.0×10^5 to about 1.1×10^4 dyn/cm², whereas the corresponding decrease in the loss modulus G'' was much lower. In the case of the cured A-6 and A-7, with the A-100 resin concentration 15 and 20%, respectively, the value of G'' was higher than that of G' at 0.1 rad/s, indicating that viscosity dominated elasticity. From these results conclusions can be made that increasing the A-100 resin content reduced effectively the elasticity of the cured PSA and enhanced the viscosity.

Adhesive performance of PSA

The main performance of UV-PSA specimens is listed in Table IV. With the $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ increase in ratio, the peel strength of UV-PSA both to the stainless-steel panel and to the PP panel fell in the order A-3a > A-2a > A-1a, while both the corresponding holding power and the SAFT were increased. The G' value of PSA was related to the diffusion property of the bulk adhesives onto substrates within a short period of time. Increasing the storage modulus of PSA caused an increase in elasticity and depressed polymer chain mobility, resulting in the decrease of the interfacial bond strength of PSA.¹² In this study, increase in the $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ ratio enhanced the storage modulus of the cured PSA dramatically and weakened polymer chain mobility, which caused a smaller adhesive volume to be deformed at any time during peeling and a lower peel strength. The holding power and SAFT value depended primarily upon the steady-flow viscosity of PSA when shearing stresses were small and the rate of creep was very slow.⁸ In this study, increasing of the $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ ratio improved the complex viscosity Eta^* at low frequency, 0.1 rad/s, which resulted in enhancement of holding power.

Comparing A-3a to A-4a, the peel strength of A-4a was lower, and the HP and SAFT of the A-4a were higher. The UV-cured PSA prepared with P-4 had higher storage modulus and complex viscosity, which caused increased HP and SAFT of the cured PSA and reduction in the peeling strength.

Table IV also shows that, with increased A-100 resin, the peeling strength of PSA increased and the holding power and SAFT decreased. The A-100 resin acted as a plasticizer to depress dramatically the storage modulus G' and complex viscosity Eta^* of the cured PSA, especially at low frequency, as shown in Figures 7 and 8, which enhanced the interfacial bond strength of PSA and increased the peeling strength, whereas it depressed the holding power and SAFT.

CONCLUSIONS

1. Polyurethane acrylate was synthesized with dimer-based polyester diol, and UV-cured PSA was prepared with PUA.
2. The characteristics of the PSA in this study were high adhesion both to high-surface-energy and to low-surface-energy adherends, high shear adhesion failure temperature, and relatively long holding power.
3. Increased $\text{OH}_{\text{HCO}}/\text{OH}_{\text{Diol}}$ ratio improved the η^* and M_w values of PUA and enhanced the G' , G'' , and Eta^* values of the UV-cured PSA. This caused increased holding power and SAFT of the UV-cured PSA and yet reduced holding power of PSA.
4. Substitution of OB for DBTDL during the synthesis of PUA decreased the η^* and M_w values of PUA and yet increased the G' and Eta^* values of the UV-cured PSA, which, in turn, caused increased holding power and SAFT and yet reduced peeling strength.
5. A-100 resin was miscible with PUA. Elevation of A-100 resin content depressed the G' , G'' , and Eta^* values of UV-cured PSA, which weakened the holding power and SAFT of UV-cured PSA and yet increased the T_g value and the peeling strength.

TABLE IV
Performance of UV-Cured PSA

Sample	P (N/25mm)		HP (h)	SAFT (°C)
	Steel	PP		
A-1a	10 ± 0.3	6.1 ± 0.4	94 ± 5	108 ± 5
A-2a	11.7 ± 0.3	7.2 ± 0.4	56 ± 6	95 ± 4
A-3a	13.6 ± 0.3	9.4 ± 0.3	22 ± 5	73 ± 3
A-4a	10.3 ± 0.4	6.1 ± 0.4	46 ± 7	102 ± 4
A-5a	12.7 ± 0.3	8.3 ± 0.3	37 ± 5	87 ± 3
A-6a	14.8 ± 0.3	10.5 ± 0.3	13 ± 4	64 ± 3
A-7a	15.2 ± 0.2	11.2 ± 0.3	5 ± 2	56 ± 3

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