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The curing performance of UV-curable semi-interpenetrating polymer network structured acrylic pressure-sensitive adhesives

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Abstract—This article reports on the curing performance of UV-curable acrylic binders prepared with trifunctional monomers and a photoinitiator. The curing reaction was achieved by direct excitation of pressure-sensitive adhesives (PSAs) by irradiation with a 100-W high-pressure mercury lamp with different UV doses. The curing performance of PSAs was studied by photo-differential scanning calorimetry (photo-DSC), gel-fraction determination and Fourier transform infrared–attenuated total reflection (FTIR–ATR) spectroscopy. The reaction rate and extent of UV curing were found to be strongly dependent on the curing rates of the trifunctional monomers, trimethylolpropane triacrylate (TMPTA) and trimethylolpropane ethoxylated (6) triacrylate (TMPEOTA), which have different molecular weights. Exothermic areas increased with increasing acrylic acid concentration. Moreover, gel fractions sharply increased after UV irradiation and then remained constant with prolonged UV exposure. TMPTA blends had higher gel fractions than TMPEOTA blends because of TMPTA's fast curing rate. Also, the gel fractions of TMPTA blends showed no variation with acrylic acid concentration. However, the FTIR–ATR absorption peak areas representing the relative concentration of C=C bonds showed more conspicuous trends for the curing reaction. Although the gel fractions of TMPTA blends showed no differences, the relative concentrations of C=C bonds increased with increasing acrylic acid concentration. In addition, TMPTA blends showed higher relative concentrations of C=C bonds because of the faster curing rate of TMPTA.

Keywords: PSAs; photo-DSC; gel fraction; TMPTA; TMPEOTA.

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1. INTRODUCTION

Photo-induced polymerization includes polymerization by UV light, visible light, electron beam, or laser. Photocuring represents polymerization which includes unsaturated inks, coatings and pressure-sensitive adhesives (PSAs) [1]. UV-curing systems, involving photocuring, are widely used because of their rapid production rate in small working place and solvent-free curing at ambient temperatures.

In many industries, acrylic monomers and oligomers are widely used because they possess superior properties, such as transparency, colorlessness, resistance to yellowing under sunlight and resistance to oxidation due to their saturated structures. However, acrylic compounds have poor thermomechanical stability because they generally have a linear structure. Therefore, cross-linking of multifunctional acrylates is needed in order to increase their thermomechanical stability. Kaczmarek and Decker [2] reported that when an adhesive is weakly cross-linked, it shows a fluid-like behavior; however, for a more cross-linked adhesive, creep resistance greatly increases [3]. Moreover, multifunctional acrylates cross-link quickly by radical and cationic polymerizations, and their kinetics and properties have been investigated [4]. In general, cross-linking of trifunctional acrylate monomers, such as trimethylolpropane triacrylate (TMPTA) and tetraethylene glycol diacrylate (TEGDA), can lead to removability [5]. Also, the advantages of trifunctional acrylates include good adhesion to plastics, and high cross-link density, reactivity, chemical resistance, hardness and scratch resistance [6]. Cross-linked multifunctional acrylates produce semi-interpenetrating polymer networks (IPNs). The semi-IPN structure gives PSAs the advantages, such as improved heat resistance as well as creep resistance [3, 7]. Also, Athawale *et al.* [8] reported that photopolymerization was effective in obtaining networks with a high degree of interpenetration.

The UV dose, the type of photoinitiator, the T_g of the binder, and the functionality, concentration and structure of the reactive monomers all affect the photopolymerization reaction and conversion. The relationship between the chain length of the monomer and the degree of polymerization reaction has been investigated. For example, poly(ethylene glycol) diacrylate polymerizes much faster than di-, tri- and tetraethylene glycol diacrylates. Also, poly(ethylene glycol) diacrylate achieves almost a complete conversion because the longer chain length of the monomers increases the diffusion of unreacted C=C bonds during the final stage of the polymerization reaction. Enhanced molecular mobility favors higher conversion rates [9]. Therefore, long-chain monomers react faster although they have higher viscosity than short-chain monomers when a photoinitiator is not added, and also the conversion of long-chain monomers is higher than that of short-chain monomers. Scherzer [10] reported that penetration of UV light strongly increases from short-chain monomers to long-chain monomers.

In this study, semi-IPN structured PSAs were prepared using two types of trifunctional acrylate monomers and a UV-curing system. The trifunctional acrylate monomers used were TMPTA and TMPEOTA. These monomers have different

chain lengths. Finally, conversion and reaction rates were investigated using photo-DSC, gel fraction determination and FTIR-ATR spectroscopy.

2. EXPERIMENTAL

2.1. Materials

2-Ethylhexyl acrylate (2-EHA, Junsei Chemical, Japan), acrylic acid (Junsei Chemical, Japan), and vinyl acetate (VAc, Kanto Chemical, Japan) were used as received. Methanol (Samchun Pure Chemical, South Korea) and ethyl acetate (EAc, Duksan Chemical, South Korea) were used as solvents. Without further purification, 2,2'-azobisisobutyronitrile (AIBN, Daejung Chemical, South Korea) was used as the initiator. Trimethylolpropane triacrylate (TMPTA, Miwon Commercial, South Korea) and trimethylolpropane ethoxylated (6) triacrylate (TMPEOTA, Miwon Commercial, South Korea) were used as the trifunctional monomers. Figure 1 illustrates the structures of the trifunctional monomers. 2,2-Dimethoxy-1,2-diphenylethanone (Miwon Commercial, South Korea) was the photoinitiator.

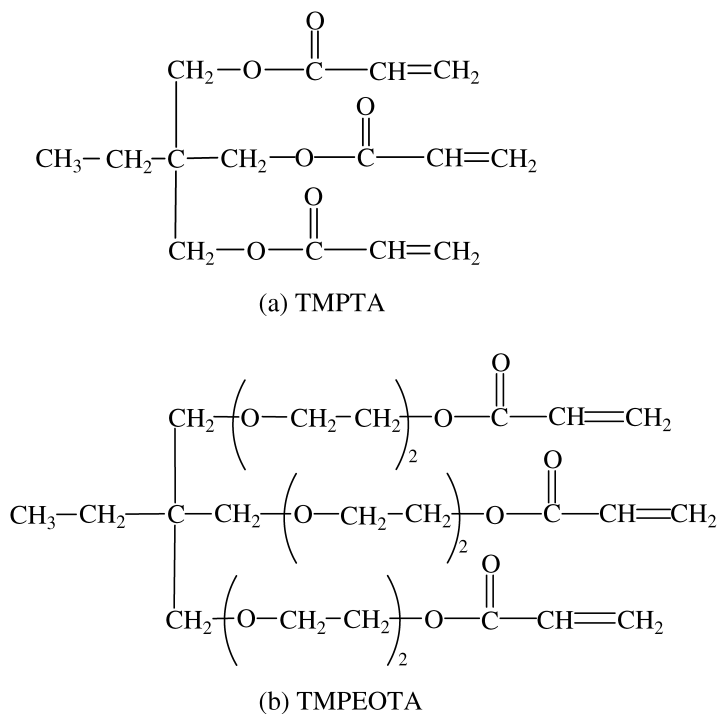


Figure 1. Chemical structures of trifunctional monomers.

Table 1.

Blend compositions for polymerization and characteristics of binders used

	2-Ethylhexyl acrylate (wt%)	Vinyl acetate (wt%)	Acrylic acid (wt%)	T_g (°C) ¹	Viscosity (cP)
Binder 1	88	7	5	−49.8	16 000
Binder 2	83	7	10	−46.9	30 000
Binder 3	78	7	15	−43.5	65 000

¹ Glass transition temperatures (T_g) measured by DSC.

2.2. Synthesis of binders

Binders were prepared as 40 wt% solids by solution polymerization. Various amounts of 2-EHA and acrylic acid, 10.5 g VAc, 25 g methanol, 0.15 g AIBN and 50 g ethyl EAc were mixed in a 500-ml four-neck flask equipped with a stirrer, a dropping funnel and a thermometer. This mixture was heated to 70°C with stirring. After the exothermic reaction was complete, the temperature was maintained for 30 min. A mixture of 100 g EAc and 0.3 g AIBN was added. After 1 h, another mixture of 100 g EAc and 0.3 g AIBN was added. After 3 h, a third mixture of 100 g EAc and 0.3 g AIBN was added. After 1 h, polymerization was stopped. The prepared pre-polymers were used as PSAs. The compositions of the PSAs are listed in Table 1.

2.3. Preparation of UV-curable acrylic PSAs

UV-curable PSAs were prepared by blending polymerized binders, a photoinitiator and trifunctional monomers. The amount of trifunctional monomer was 30 wt% of the binder and the photoinitiator added was 5 phr of trifunctional monomer. The mixtures were stirred with a mechanical stirrer at room temperature for about 30 min. The UV-curable polymers were coated onto polyester films (PET, 50 μm thick, SKC, South Korea) using a bar coater No. 18 (wet thickness 41.1 μm) and then dried at 70°C for 5 min. The UV-curable PSA films were cured using a conveyor belt-type UV-curing equipment with a 100-W high-pressure mercury lamp (main wavelength: 340 nm). UV doses used in this study were 0, 200, 600, 1000, 1400 and 1800 mJ/cm^2 . UV doses were measured with an IL 390C Light Bug UV radiometer (International Light, USA).

2.4. Differential scanning calorimetry (DSC)

Glass transition temperatures (T_g) were measured using a TA Instruments Q-1000 DSC equipment (at NICEM, Seoul National University). Samples were cooled to -80°C and then heated to 150°C at a heating rate of $10^\circ\text{C}/\text{min}$ for the first scan. Next, samples were immediately quenched to -80°C , and then kept at this temperature for 3 min. Samples were then reheated to 200°C at a heating rate of

5°C/min for the second scan. In this study, the T_g was obtained from the second scan to exclude thermal history effects.

2.5. Viscosity

The viscosity of the prepared PSAs was measured using a Brookfield Viscometer (DV-II+) equipped with an RV-7 spindle at 30 rpm. All measurements were performed at 20°C and 50% relative humidity.

2.6. Photo differential scanning calorimetry (photo-DSC)

Photo-DSC experiments were conducted using a TA Instruments Q-1000 DSC equipped with a photocalorimetric accessory (Novacure 2100), which used light from a 100 W middle-pressure mercury lamp. Light intensity was determined by placing an empty DSC pan on the sample cell. UV light intensity at the sample was 50 mW/cm² over a wavelength range of 300–545 nm. The weight of the sample was about 3 mg and the sample was placed in an open aluminum DSC pan. Measurements were carried out at 25°C.

2.7. Gel fraction

Gel fractions of acrylic PSAs blended with trifunctional monomers and UV-cured acrylic PSAs were determined by soaking the samples in toluene for 1 day at 50°C. The sample amount was about 5 g. The soluble part was removed by filtration and dried at 50°C to a constant weight. The gel fraction was calculated by the following equation:

$$\text{Gel fraction (\%)} = (W_1 / W_0) \times 100, \quad (1)$$

where W_0 is the weight before filtration and W_1 is the weight after filtration.

2.8. Fourier transform infrared (FTIR) spectroscopy

IR spectra were recorded using a Nicolet Magna 550 Series II FTIR spectrometer (Midac, USA) equipped with an attenuated total reflectance (ATR) accessory. To obtain the IR spectra of the UV-curable PSAs, each PSA was cut into 5 × 0.5 cm² pieces. The thickness of PSAs is not important in the ATR method, so it was not measured. The ATR crystal was zinc selenide (ZnSe) and its refractive index at 1000 cm⁻¹ was 2.4. It had a transmission range from 400 to 4000 cm⁻¹. The resolution of the spectra recorded was 4 cm⁻¹.

The curing behavior of the UV-curable PSAs was analyzed by observing changes in the deformation of the C=C bond at 810 cm⁻¹. Also, all results were obtained with a baseline correction, which was used to correct the spectra that had sloping or curved baselines.

3. RESULTS AND DISCUSSION

3.1. Performance of binders

The T_g and viscosity of the binders are listed in Table 1. The T_g of the binders increased with increasing acrylic acid concentration because acrylic acid has the highest T_g among the monomers used in this study. Similar results were obtained by Kim and Mizumachi [11] and Shojaei *et al.* [12]. They reported that the T_g of the copolymers of acrylic acid and 2-EHA increased with increasing acrylic acid concentration. Also, the viscosity of the binders increased with increasing acrylic acid concentration because of the formation of hydrogen bonds among the hydrogens in the carboxylic group of the acrylic acid [7]. Both the T_g and viscosity of the binder can affect the reaction rate of trifunctional monomer in the binder. The results showed a relationship between the curing kinetics of trifunctional monomers and acrylic acid concentration in the binder.

3.2. Photo-DSC

Photo-DSC offers a simple method to characterize the kinetics of photopolymerization reaction. Reaction rate can be measured by monitoring the rate at which heat is released from the polymerizing sample because polymerization is generally an exothermic reaction. Therefore, the profiles of heat of reaction versus time, provided by photo-DSC, can be used to characterize reaction kinetics and to evaluate polymerization rate constants. Nelson *et al.* [13] used photo-DSC to characterize free radical photopolymerization.

Figure 2 shows photo-DSC thermograms of two trifunctional monomers, TMPTA and TMPEOTA, in the presence of the same concentration of photoinitiator. The curing reaction of TMPTA was fast, based on the slope because of its short chain length. Anseth *et al.* [9] showed that the rate of polymerization decreased and the conversion increased with increasing number of ethylene glycol units in the monomer. Enhanced mobility due to short chain length decreases cross-link density and the concentration of double bonds. Therefore, we can expect that TMPTA reacts fast and then quickly cures because it has a shorter chain length than TMPEOTA.

Reaction rates of PSAs blended with TMPTA and TMPEOTA were measured. Photo-DSC thermograms of the PSAs blended with these two trifunctional monomers were calculated because the maximum reaction rates of binders blended with TMPTA and TMPEOTA showed very tiny differences (not shown).

Figure 3 shows the exothermic areas of the binders blended with trifunctional monomers. TMPTA shows a higher exothermic area as well as higher conversion than TMPEOTA. The reaction energy of TMPTA was higher than that of TMPEOTA. TMPTA has a short chain length and can avoid entanglement between binder polymers and TMPTA. TMPEOTA, on the other hand, has a relatively long chain length and can become more entangled with binder polymers; thus, the reaction of TMPEOTA might be hindered by the binder polymers. Therefore, TMPTA

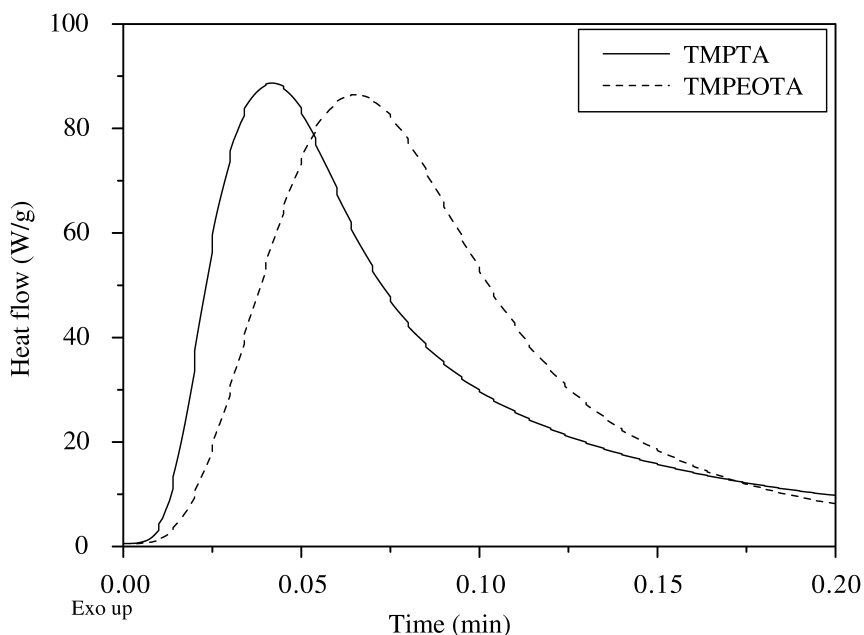


Figure 2. Photo-DSC thermograms of trifunctional monomers blended with a photoinitiator.

achieved higher conversion than TMPEOTA. This result was different from that reported by Anseth *et al.* [9]. The difference between Anseth *et al.*'s research and this study is the presence of a binder, and acrylic binders that affect the conversion of reactive monomers. The type of binder affects the conversion of TMPEOTA blends. For TMPTA blends, there is no such tendency. However, the conversion of TMPEOTA blends increased from Binder 1 to Binder 3. TMPEOTA largely reacted in Binder 3 because in this blend TMPEOTA could be free of entanglement in contrast to other TMPEOTA blends. Binder 3, with the highest T_g among the three binders, can facilitate TMPEOTA's reaction, whereas Binder 1 can obstruct the reaction of TMPEOTA because it has the lowest T_g and viscosity. The chain length of trifunctional monomers influences conversion. Additionally, the type of binder affected the conversion of TMPEOTA.

3.3. Gel fraction

Gel fraction can be determined by measuring the insoluble fractions, such as cross-linked or network polymers. In this study, the soluble linear binder polymers in organic solvents turned into insoluble semi-IPN structures by photopolymerization using trifunctional monomers.

Figure 4 shows the gel fraction of the three binders blended with TMPTA and TMPEOTA. Before UV irradiation, the gel fractions of Binder 1, Binder 2 and Binder 3 blended with TMPTA are about 4%, 27% and 65%, respectively. The gel fractions of the three blends increased up to 80–90% after UV irradiation. The

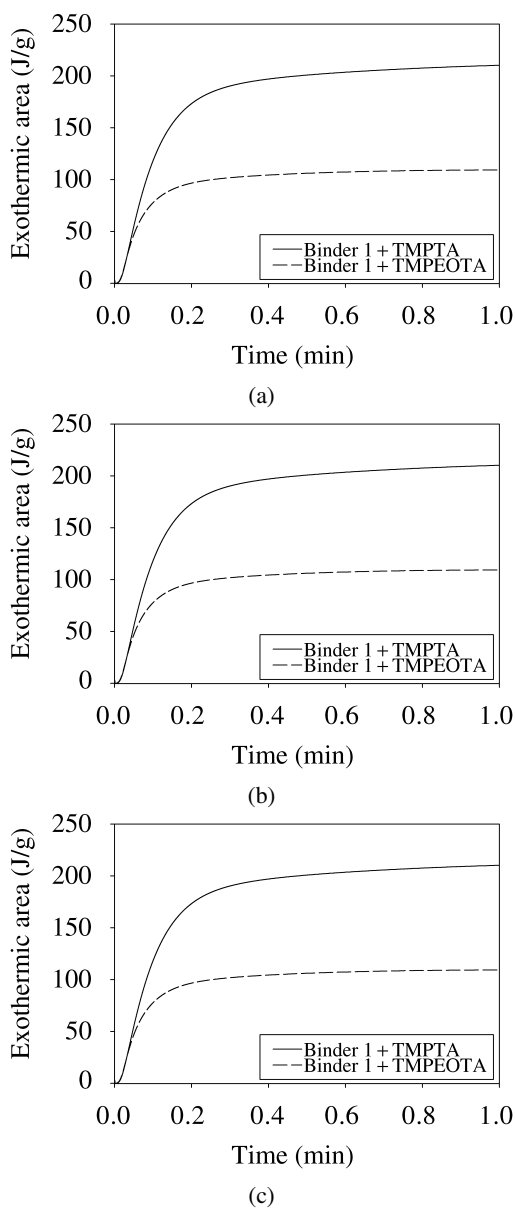
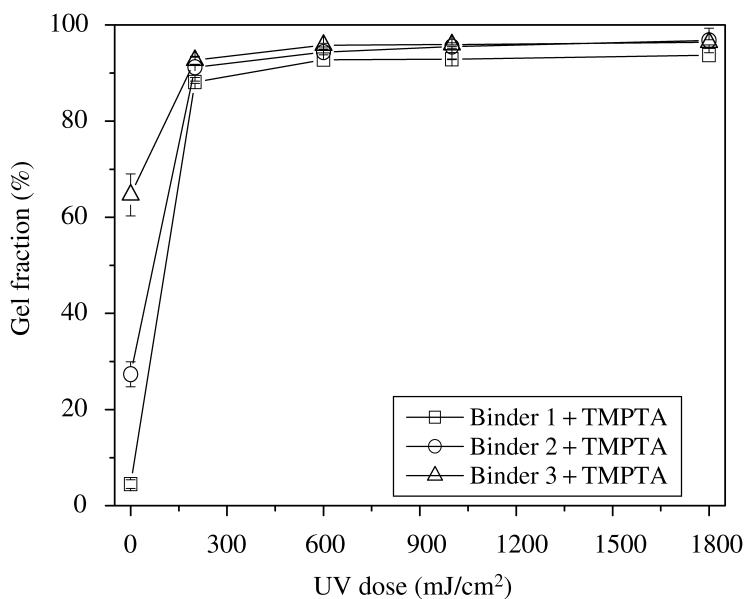
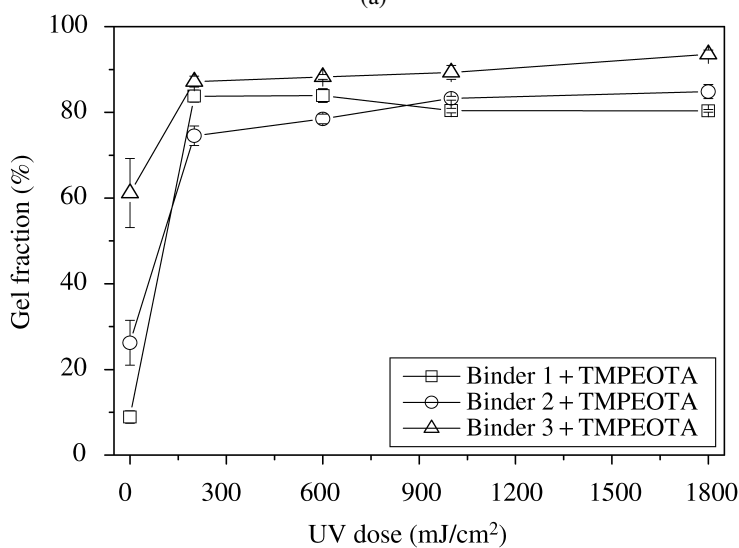


Figure 3. Exothermic areas of three binders blended with trifunctional monomers. (a) Binder 1. (b) Binder 2. (c) Binder 3.

TMPTA formed tightly cross-linked semi-IPN structures with binder polymers after UV exposure. TMPTA made densely cross-linked structures due to its short chain length. The three blends had different gel fractions because of different binder viscosity before UV exposure. However, they had similar gel fractions after UV exposure.



(a)



(b)

Figure 4. Gel fraction as a function of UV dose for the three binders blended with trifunctional monomers. (a) Blended with TMPTA. (b) Blended with TMPEOTA.

PSAs blended with TMPTA showed slightly higher gel fraction values than TMPEOTA blends. The gel fraction of Binder 1 blended with TMPEOTA steeply increased after UV irradiation. This result is similar to Binder 1 and TMPTA blends. However, the gel fraction of Binder 1 blended with TMPTA after UV irradiation is about 94%, whereas that of Binder 1 blended with TMPEOTA is about 80%. The

long chain length of TMPEOTA disturbed the formation of a tightly cross-linked network.

In general, short chain length monomers react fast, while long-chain length monomers react slowly. When a monomer reacts quickly, there are many unreacted monomers in the polymer network after UV exposure because the monomers form a network before achieving complete conversion. However, when a monomer reacts slowly, monomers achieve complete conversion without leaving unreacted monomers. Therefore, long chain monomers can achieve complete conversion. In this experiment, the reactivity of the two trifunctional monomers was found not to be different. However, the short chain length monomer, TMPTA, had a higher conversion rate than the long-chain length monomer, TMPEOTA. This was due to TMPEOTA's entanglement with the binders because of its long chain length and hindrance by the binders owing to TMPEOTA's low mobility. As a result, TMPTA and TMPEOTA blends had different gel fractions because of different viscosity, mobility and chain length of trifunctional monomers. Also, the binders had a greater effect on gel fractions before UV exposure.

3.4. FTIR-ATR

The kinetics of the photoinduced cross-linking was observed using FTIR-ATR. After photoinitiation by UV irradiation at a specific wavelength, functional monomers proceeded to polymerize. These functional monomers formed a cross-linked semi-IPN structure. The curing behavior of functional monomers can be monitored using FTIR because the C=C twisting vibration in functional monomers participates in the cross-linking reaction [10].

The FTIR spectra in Fig. 5 of Binder 1 blended with TMPEOTA as a function of UV irradiation show absorption bands at 810 cm^{-1} and at 1635 and 1620 cm^{-1} . Before UV irradiation (0 mJ/cm^2), absorption bands of the acrylate group ($\text{H}_2\text{C}=\text{CH}-\text{C}=\text{O}$) are seen at 810 cm^{-1} and absorption bands for $\text{C}=\text{C}-\text{C}=\text{O}$ stretching are seen at 1635 and 1618 cm^{-1} [14]. Several studies have shown that the absorption band at 810 cm^{-1} due to the C=C twisting vibration of acrylate groups decreases with increasing UV exposure time [2, 15]. These double bonds have a plane conformation, but UV irradiation deforms the C=C bonds into an out-of-plane conformation.

In this study, UV-curing behavior was determined by integrating the absorbance peaks around 1635 and 1620 and 810 cm^{-1} from the FTIR spectra. The C=O stretching vibration of an acrylic monomer around 1730 cm^{-1} was adopted as an internal standard band for calculation to compensate for the effects of differences in the thicknesses of the PSA samples. The relative concentration of C=C bond, as a function of UV dose and trifunctional monomer, was calculated according to the following equation:

$$\text{Relative concentration of UV cured group (\%)} = \frac{[A]_{810}^{\text{UV}}/[A]_{1730}^{\text{UV}}}{[A]_{810}^0/[A]_{1730}^0} \times 100, \quad (2)$$

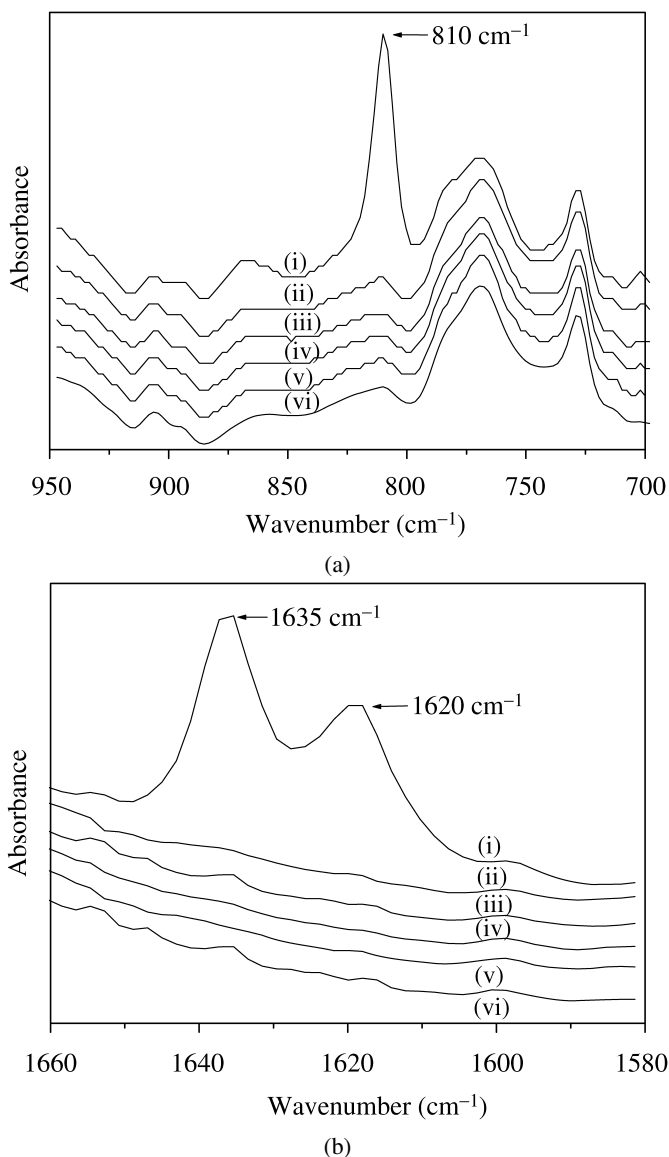
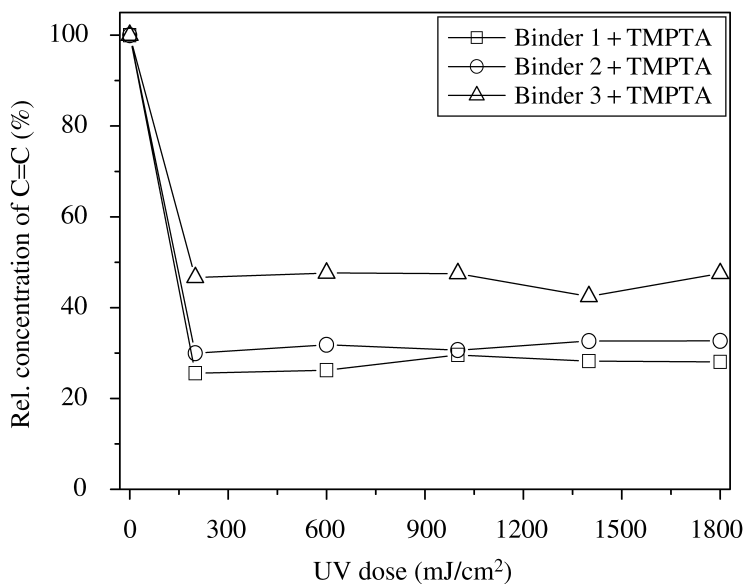


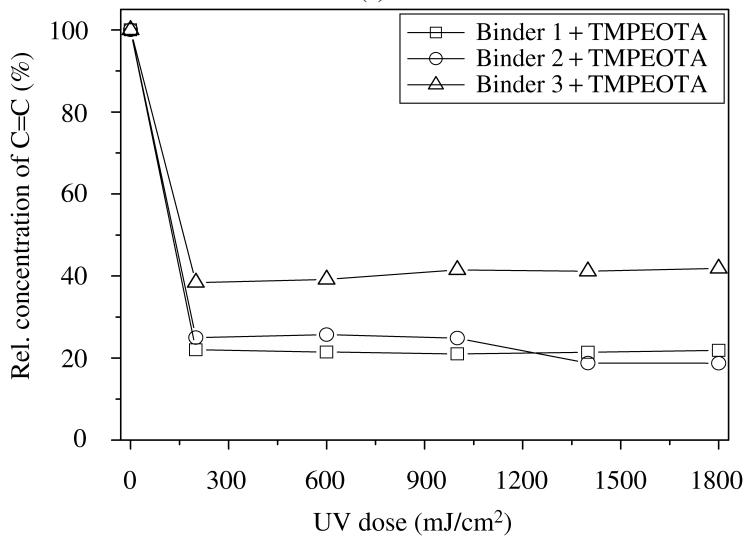
Figure 5. FTIR spectra of Binder 1 blended with TMPEOTA showing absorption bands at 810 cm⁻¹ (a) and 1635 and 1620 cm⁻¹ (b) for different UV doses: (i) 0, (ii) 200, (iii) 600, (iv) 1000, (v) 1400 and (vi) 1800 mJ/cm², respectively.

where $[A]_{810}^0$ is the IR absorbance at 810 cm⁻¹ before UV irradiation, $[A]_{1730}^0$ is the IR absorbance at 1730 cm⁻¹ before UV irradiation, $[A]_{810}^{UV}$ is the IR absorbance at 810 cm⁻¹ after UV irradiation and $[A]_{1730}^{UV}$ is the IR absorbance at 1730 cm⁻¹ after UV irradiation.

Figure 6 shows the relative concentrations of C=C bonds after UV exposure of the PSAs blended with functional monomers. The amount of remaining C=C



(a)



(b)

Figure 6. Relative concentrations of C=C bonds at 810 cm^{-1} as a function of UV dose for UV-curable groups in binders blended with trifunctional monomers. (a) TMPTA and (b) TMPEOTA.

bonds decreased with increasing acrylic acid concentration in the binder because acrylic acid affects the conversion of the blends. This is also shown in the gel fraction results. The relative concentrations of C=C bonds of binders blended with TMPTA were lower than those of TMPEOTA blends because of different reaction rates of these trifunctional monomers. As acrylic acid concentration increased,

interactions between binder molecules increased, and the degree of freedom of TMPTA decreased. Therefore, the relative concentration of C=C bonds decreased with increasing acrylic acid concentration in the binder. Similar to the TMPTA blends, the relative concentrations of C=C bonds of the binders blended with TMPEOTA decreased with increasing acrylic acid concentration in the binders.

As the content of trifunctional monomer increased, the amount of remaining C=C bonds decreased after UV exposure. TMPTA had a faster reaction rate with photoinitiator than TMPEOTA because of TMPTA's short chain length. Therefore, the relative concentrations of C=C bonds of TMPTA were higher than those of TMPEOTA. In addition, the relative concentrations of C=C bonds were not zero, and gel fractions did not reach 100%. The remaining C=C bonds might have remained unreacted by the photoinitiator because they were trapped in the polymer network.

4. CONCLUSIONS

The goal of this study was two-fold: first, to synthesize acrylic binders and obtain semi-IPN structured PSAs, using two trifunctional monomers of different molecular weights, TMPTA and TMPEOTA, by UV curing and second, to investigate their UV curing performance, using photo-DSC, gel fraction and FTIR.

At fixed synthesis conditions, the addition of acrylic acid increased the viscosity of the prepared acrylic binders because of formation of hydrogen bonds in the carboxylic group of acrylic acid. Also, the T_g of the binder increased because of the highest T_g of acrylic acid among the monomers used.

Although the curing rates of the two trifunctional monomers were similar, their reaction rates in the presence of binders were different. The conversion of blends with TMPEOTA increased with increasing acrylic acid concentration in the binders. However, TMPTA did not show this tendency. Moreover, gel fractions of the blends drastically increased with 200 mJ/cm² of UV exposure. After prolonged UV irradiation, the blends showed similar values of gel fraction. Also, the PSAs blended with TMPTA showed higher gel fractions because of their faster reaction rate due to their shorter chain length.

UV irradiation of the functional monomer blended PSA films led to changes in specific absorption bands of the FTIR spectra due to the cross-linking reaction of C=C bond. The peaks due to C=C bonds at 810 cm⁻¹, and at 1635 and 1620 cm⁻¹ were reduced with increasing UV dose. The relative concentration of C=C bonds sharply decreased with 200 mJ/cm² of UV irradiation and then showed only small changes with higher UV exposure. In addition, in the blends with trifunctional monomers, the relative concentrations of C=C bonds increased with increasing acrylic acid concentration. TMPTA blends showed higher values because of their faster photocuring reaction with the photoinitiator.

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