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Optical properties and UV-curing behaviors of optically clear semi-interpenetrated structured acrylic pressure sensitive adhesives

Seung-Woo Lee [a], Ji-Won Park [a], Young-Eun Kwon [a], Soyon Kim [a], Hyun-Joong Kim [a,*], Eon-Ah Kim [b], Hang-Soo Woo [b], Jolanta Swiderska [c]

[a] *Laboratory of Adhesion & Bio-Composites, Program in Environmental Materials Science, Research Institute for Agriculture & Life Sciences, Seoul National University, Seoul 151-921, Republic of Korea*
[b] *Ulsan Fine Chemical Industry Center, Ulsan 681-802, Republic of Korea*
[c] *Non-public Health Center, Boleslawa S mialego 28, 70-348 Szczecin, Poland*

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ABSTRACT

Acrylic pressure-sensitive adhesives (PSAs) with different side-chain lengths in the co-monomer were synthesized. This study employed semi-interpenetrated structured polymer networks using UV-curing with a hexafunctional monomer, dipentaerythritol hexacrylate (DPHA). The optical properties of the acrylic PSAs were examined by UV-visible spectroscopy and a prism coupler. ARES was used to characterize the viscoelastic properties of the acrylic PSAs. Adhesion performance was conducted by the peel strength and probe tack tests. Also UV-curing behavior of the acrylic PSAs was investigated by FTIR.

1. Introduction

Pressure-sensitive adhesive (PSAs) tapes composed of acrylic copolymers have been extensively utilized in various applications including packaging, printing, electrical insulation and automobile parts. In general, the PSA properties (tack, peel adhesion, holding power) of acrylic adhesives have been controlled by blending tackifiers or dissimilar polymers, by adjusting molecular weight and its distribution, and also by copolymerization with a polar monomer and by varying the curing system. In recent years, the PSA properties of acrylic adhesive copolymers have been interpreted by considering various factors, such as dynamic mechanical properties, surface tension, and miscibility [1–5]. It is of interest to design acrylic PSAs in such a way that their adhesion can be adjusted according to the chemical structure [6–8] as well as external stimuli such as light [9] and heat [10–12].

It is well known that acrylic polymers lacking crosslinks cannot be used as PSAs since crosslinking provides the thermal and mechanical properties required for thermomechanical stability [13]. Recently, UV-curing technology has been used in crosslinking PSAs owing to its economical and environmental advantages, coupled with none of the disadvantages associated with using a crosslinking agent. UV-curing technology has many benefits because of its high performance, cost savings and low emission of VOCs [14].

Because of this high performance, UV-curable PSAs have optical applications. Optical PSAs are being used increasingly in areas such as LCDs, PDPs and OLEDs due to the increase in optical device production [15]. Optical PSAs should have high contrast, clarity, high refractive index and good reliability [16]. Generally, acrylic PSAs often include 3–10 wt% acrylic acid and 90–97 wt% alkyl soft acrylate composed of butyl acrylate, hexyl acrylate, 2-ethylhexyl acrylate, isooctyl acrylate or decyl acrylate [17].

In this study, optically clear acrylic PSAs with a high refractive index material (i.e., 2-phenoxy ethyl acrylate) were prepared by solvent polymerization. Emphasis was placed on the effect of side-chain length in the co-monomer including ethyl, butyl, hexyl and lauryl acrylate groups. The optical properties and UV-curing behaviors of UV-curable acrylic PSAs were investigated. Finally, adhesion performance and viscoelastic properties were examined.

2. Experimental

2.1. Materials

Experiments were conducted using solvent-based acrylic PSAs containing 15 wt% 2-ethylhexyl acrylate (2-EHA, 99.0% purity, Samchun Pure Chemical Co., Ltd, Republic of Korea), 5 wt% acrylic acid

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[*] Corresponding author. Tel.: +82 2 880 4784; fax: +82 2 873 2318.
E-mail address: hjokim@snu.ac.kr (H.-J. Kim).

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Table 1
Monomer compositions of the acrylic PSAs.

	2-EHA	AA	2-PEA	EA	BA	HA	LA
PSA-1	15	5	45	35	0	0	0
PSA-2	15	5	45	0	35	0	0
PSA-3	15	5	45	0	0	35	0
PSA-4	15	5	45	0	0	0	35

*All the monomers were based on weight percent.

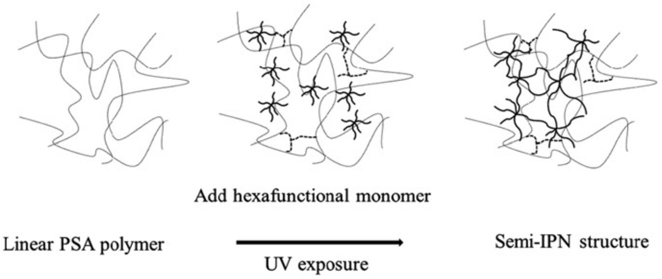

Fig. 1. Process of producing a semi-IPN structure in UV-cured acrylic PSAs [22].

(AA, 99.0% purity, Samchun Pure Chemical Co., Ltd, Republic of Korea), and 45 wt% 2-phenoxy ethyl acrylate (2-PEA, 90.0% purity, Tokyo Chemical Industry, Japan). Ethyl acrylate (EA, 99.0% purity, Tokyo Chemical Industry, Japan), butyl acrylate (BA, 99.0% purity, Samchun Pure Chemical Co., Ltd, Republic of Korea), hexyl acrylate (HA, 99.0% purity, Tokyo Chemical Industry, Japan) and lauryl acrylate (LA, 99.0% purity, Samchun Pure Chemical Co., Ltd, Republic of Korea) were employed as 35 wt% co-monomers, as listed in Table 1. Ethyl acetate was used as the organic solvent at its boiling point temperature of about 77 °C. A solution of 0.1 wt% 2,2′-azobisisobutyronitrile (AIBN, Junsei Chemical, Japan) was used as the thermal initiator to start radical polymerization, and 2-hydroxy-2-methyl-1-phenyl-propane-1-one (Miwon Specialty Chemical, Republic of Korea) was used as a photoinitiator. Dipentaerythritol hexacrylate (DPHA, Miwon Specialty Chemical, Republic of Korea) was used as the diluent monomer.

2.2. Methods

2.2.1. Synthesis of acrylic PSAs

Acrylic PSAs were synthesized by solution polymerization. The solid content in solution was 50%. The mixture was placed into a 500 ml four-neck flask equipped with a stirrer, condenser and thermometer and was heated to 78 °C with constant stirring. At the end of the exothermic reaction, the temperature was maintained for 30 min, and a blend of ethyl acetate and AIBN was added. The reaction was allowed to proceed for 0.5 h or 2.5 h. Finally, polymerization was terminated by cooling the mixture to room temperature.

2.2.2. Preparation of cured acrylic PSAs

UV-curable PSAs were prepared by blending polymerized binders, a photoinitiator and hexafunctional monomer. The amount of hexafunctional monomer was 30 phr of the binder, and the amount of photoinitiator added was 1, 2 or 5 phr of hexafunctional monomer. The mixture was stirred with a mechanical stirrer at room temperature for about 30 min. UV-curable polymers were coated onto polyester films (PET, 50-μm-thick, SKC, Republic of Korea) using a no. 18 bar coater (wet thickness 41.1 μm) and were then dried at 70 °C for 5 min. Its thickness was approximately 25 μm. The UV-curable PSA films were cured using a conveyor belt-type UV-curing apparatus with a 100-W high-pressure mercury lamp (main wavelength: 340 nm). UV doses used in this study were 0, 200, 400, 800 and 1600 mJ/cm^2. UV doses were measured with an IL 390C Light Bug UV radiometer (International Light, USA). Despite the low molecular weights, the hexafunctional monomers in the PSAs were photopolymerized after a UV dose to form semi-IPN structures as shown in Fig. 1.

2.2.3. Gel permeation chromatography (GPC) measurements

Acrylic PSAs with different side-chain lengths of co-monomers were eluted using tetrahydrofuran (THF) at room temperature and were filtered through a 0.2 μm polytetrafluoroethylene (PTFE) syringe filter. Molecular weights were measured by GPC at 35 °C using a YL-CLARITY system (Young-Lin Instrument Co., LTD, Republic of Korea) equipped with refractive index (RI) detectors and a PL-gel 10 μm column (two mixed-B). THF was used as an eluent solvent at a flow rate of 1 mL/min. The number-average (M_n) and weight-average (M_w) molecular weights were calculated using a calibration curve of polystyrene standards.

2.2.4. Optical properties

2.2.4.1. UV-visible spectroscopy. UV-visible spectroscopy (UV-1650PC Shimadzu, Japan) was used to examine the transmittance of the UV-cured acrylic PSAs. Both the bare PET as a control and the acrylic PSA sample coated onto the PET film were set on the instrument. The transmittance was determined in the visible range of 380–700 nm.

2.2.4.2. Prism coupler. The refractive index of the acrylic PSAs coated on a PET film was detected at 404, 532, 632.8 and 829 wavelengths in the visible range using a prism coupler 2010/M (Metricon, USA). Acrylic PSAs were UV-cured after coating the pre-polymers onto the PET film to produce PSA films without cohesive failure. A bare PET film was used as the reference. The prism and film were joined, and the incidence angle of the laser beam was varied so the refractive index in both the thickness and planar directions could be determined.

2.2.5. Adhesion performance

2.2.5.1. Peel strength. Peel strength was measured using a Texture Analyzer (Micro Stable Systems, TA-XT2i). The specimens were prepared as 25-mm-wide samples. The specimens were pressed onto the stainless steel substrate by two passes of a 2 kg rubber and were stored at room temperature for 12 h. Peel strength was determined at an angle of 180° with a crosshead speed of 300 mm/min at 20 °C based on ASTM D3330. The peel strength is the average force on the deboning process. The force was recorded in g units for five different runs, and the average force was reported in g/25 mm.

2.2.5.2. Probe tack. Probe tack was measured using a Texture Analyzer (Micro Stable Systems, TA-XT2i) with a 5 mm diameter stainless steel cylinder probe at 20 °C. The standard probe tack test was divided into three stages: approaching the surface of the PSAs and contact and detachment from the surface of the PSAs. The speed of the probe was 0.5 mm/s, the contact time on the PSAs surface was 1 s under a 100 g/cm^2 constant force and the separation rate was 10 mm/s. In the debonding process, probe tack was obtained as the maximum debonding force.

2.2.6. 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 $5\times0.5\ \mathrm{cm}^2$ pieces. The thickness of the PSAs was not important in the ATR method, so it was not measured. The ATR crystal was zinc selenide (ZnSe) and its refractive index at $1000\ \mathrm{cm}^{-1}$ was 2.4. A transmission range from 400 to $4000\ \mathrm{cm}^{-1}$ was used with a spectrum resolution of $4\ \mathrm{cm}^{-1}$.

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

2.2.7. *Advanced Rheometric expansion system (ARES) analysis*

The viscoelastic properties of the acrylic PSAs were determined using an advanced rheometric expansion system (ARES, Rheometric Scientific, UK) equipped with an 8 mm parallel plate mode. A typical temperature scan range was −40 °C to 80 °C, and the heating rate was 5 °C/min. The frequency was 1 Hz and the gap between the plates was 1 mm. Also, the tan δ curves from the temperature sweep tests suggested glass transition temperature (T_g) data.

3. Results and discussion

3.1. *Characterization of synthesized acrylic PSAs*

For acrylic PSAs synthesis, three basic monomers are used: 2-ethylhexylacrylate (2-EHA), acrylic acid (AA) and 2-phenoxy ethyl acrylate (2-PEA), which provide tack, flexibility and optical properties of the adhesive. The aim of the present study is to investigate the effects of co-monomers which have different side-chain lengths on both the structure and physical properties of the pressure-sensitive adhesive (PSA) coatings. The examined structural properties are molecular weight (M_w) and its distribution (M_wD) of produced acrylic PSAs. Inherent properties, such as copolymer composition and microstructure, M_w and M_wD, are among the most influential parameters affecting the PSA properties [18]. Also, the relative amounts of the molar mass distribution and the cross-linking density of the gel fraction are among the most important factors that influence the adhesive properties [19]. In this study, M_w and M_wD of the PSAs are shown in Fig. 2. It can be seen that the T_g decreases with increasing side-chain length, as expected. In fact, the M_w values of the respective polymers linearly decrease with increasing side-chain length of the co-monomer, which indicates that statistical copolymers were synthesized. Despite the unknown reactivity ratios for the co-monomer, we may speculate from the reported k_p values [20] for the polymerization process of both monomers that the monomers may be interchangeable. This is because the k_p of both monomers are the same order of magnitude, and therefore statistical copolymers may be synthesized. Minor differences were determined in the M_wD of the PSAs, except PSA-4. One may assume that this is due to the effect of lauryl acrylate as a co-monomer that results in an inhomogeneous distribution in polymers.

3.2. *Optical properties*

3.2.1. *UV-visible spectrometer*

There are many properties that PSAs should possess before they can be employed in optical films. First, the PSAs should be optically clear and have a high refractive index that can prohibit light reflection as a result of the difference in refractive index between the film and adhesives. This property can be obtained using a transmittable acrylic polymer and a particular curing process [21]. The transmittance of the PSAs was measured by UV-visible spectroscopy. Generally, acrylic PSAs should show high transmittance ($>95\%$) to be used in optical films. In this study, the synthesized PSAs were coated onto a PET film, and bare PET film was employed as the reference. While minor differences were determined, the transmittance values of all PSAs were $>95\%$, indicating high transparency in the visible wavelength, as shown in Fig. 3.

3.2.2. *Prism coupler*

As mentioned above, the refractive index of optical acrylic PSAs can be controlled using UV curing. Fig. 4 shows the refractive index of optical acrylic PSAs. In general, the refractive index of a polymer containing a high refractive index functional group is higher than that of the monomer. This means that the PEA in all PSAs, which had a high refractive index of 1.518, shows a higher polymer refractive index due to the presence of a phenyl group [16]. The refractive indexes of the PSAs decreased with increasing side-chain length of the co-monomer. In particular, the refractive index of PSA-4 was much lower than that of any other PSA due to an inhomogeneous distribution in the polymer between 532–829 nm. Hence, the other aim of this study was to increase the refractive index of PSA-4 using UV-curing. As shown in Fig. 5, the refractive index of UV-cured polymers composed of PSA-4, DPHA (30 phr in a binder) and photoinitiator (2 phr in hexafunctional monomer) was

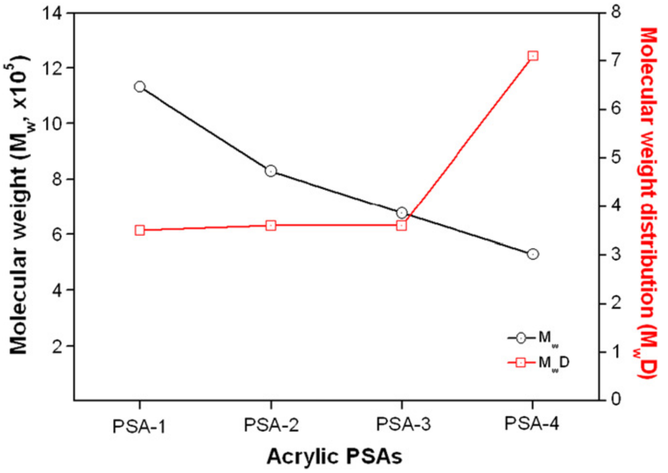

Fig. 2. Molecular weight and molecular weight distribution of the acrylic PSAs.

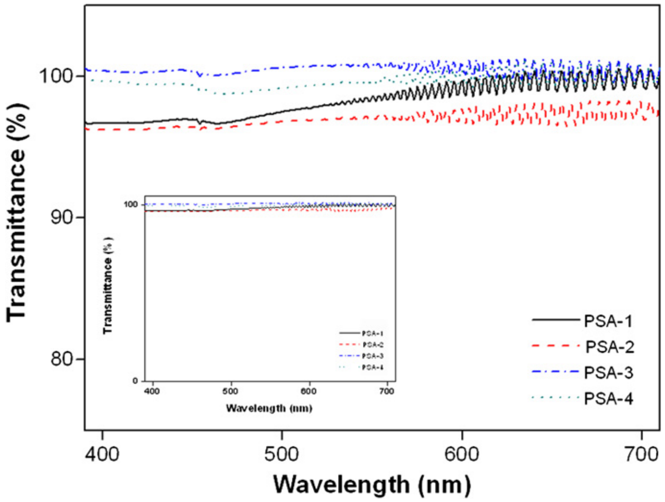

Fig. 3. Transmittance of acrylic PSAs in the visible region, 380–700 nm.

increased. In particular, the value was higher at a UV dose of 1600 mJ/cm^2 due to the highly crosslinked semi-IPN structure.

3.3. Adhesion performance

The most important factors of the UV crosslinked acrylic PSAs, peel strength and tack, can be controlled by the UV dose. In a production plant, the UV dose can be controlled by adjusting the power of the lamps and/or the speed at which the substrate is passed under lamps [9]. Fig. 6 shows the effect of the UV dose on the peel strength at different photoinitiator contents in PSA-4. Soluble linear PSAs turned into UV-cured insoluble semi-IPN structured polymers by blending the hexafunctional acrylic monomer with the photoinitiator [23]. Photoinitiator contents were 1, 2 or 5 phr (in hexafunctional monomer) in PSA-4. The peel strength decreased with increasing photoinitiator content at a UV dose of <400 mJ/cm^2. In particular, at a UV dose <400 mJ/cm^2, cohesive failure occurred when the acrylic PSA was detached from the SUS surface. However, at a UV dose 400$>$ mJ/cm^2, the acrylic PSA did not indicate cohesive failure, and the standard deviation was constant. After UV exposure, the hexafunctional monomer formed a tightly crosslinked semi-IPN structure in the PSAs because of its short chain length. In addition, PSA molecular mobility could be decreased because of the over-cured hexafunctional monomer, which increased the cohesion of the PSAs and decreased the peel strength. Also, the changes in the glass transition temperature of acrylic PSAs and the crosslinking density due to UV-curing are important factors for controlling the adhesion performance [22]. UV light is absorbed mainly by the photoinitiator, and it is attenuated progressively as it passes through the sample. The penetration of the incident photons is directly related to the photoinitiator content. Therefore, photoinitiated radical polymerization is an effective process for readily crosslinking functionalized polymers [24]. The probe tack slightly decreased with increasing UV dose, as shown in Fig. 7. When the UV dose was less than 200 mJ/cm^2, fibrillation occurred between acrylic PSAs and the stainless steel probe. This is because acrylic PSAs showed greater cohesion force to withstand the peeling and probe tack force. This suggests that a greater UV dose results in increased attachment to the SUS surface, and the T_g increases with increasing UV dose. Acrylic PSA with a high T_g indicates a more rigid structure with a higher peel strength.

3.4. FTIR-ATR spectroscopy

The kinetics of the photoinduced cross-linking were investigated using FTIR-ATR. 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 [25]. FTIR spectra of the UV-curable mixture composed of the PSA-4, DPHA and photoinitiator (2 and 5 phr in hexafunctional monomer) were investigated, as shown in Fig. 8. The conversion of C=C bonds as a function of UV dose was calculated according to the following equation:

$A^t_{N,C=C} = A^t_{810}/A^t_{int}$; $\quad Conversion = 1 - A^t_{N,C=C}/A^o_{N,C=C}$where A^t_{int} is absorbance of the internal standard during measurements.

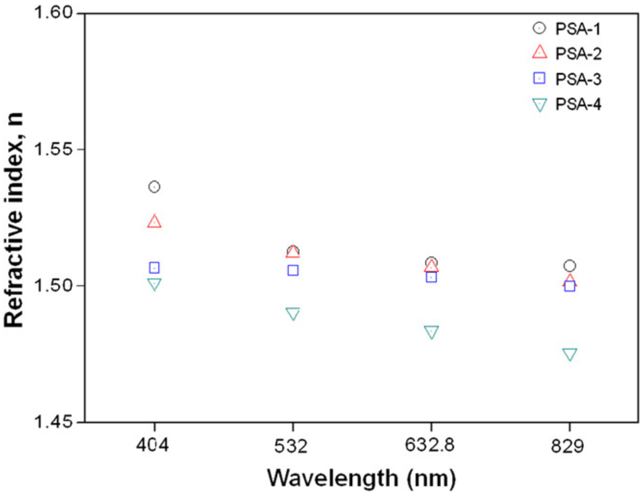

Fig. 4. Refractive index of the acrylic PSAs.

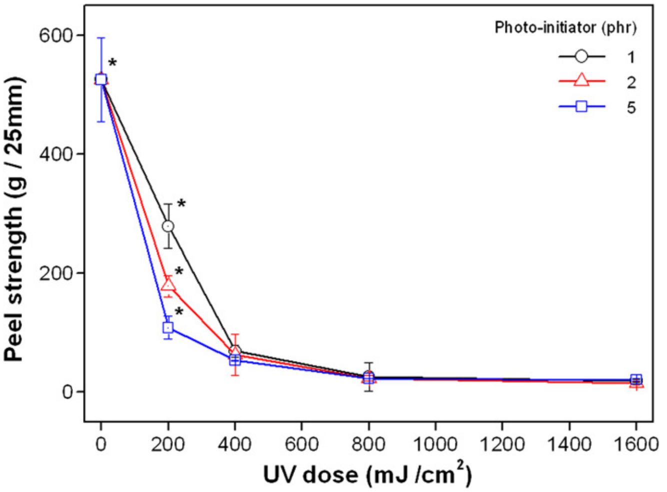

Fig. 6. Peel strength of UV-cured PSA-4 with photoinitiator content (* cohesive failure).

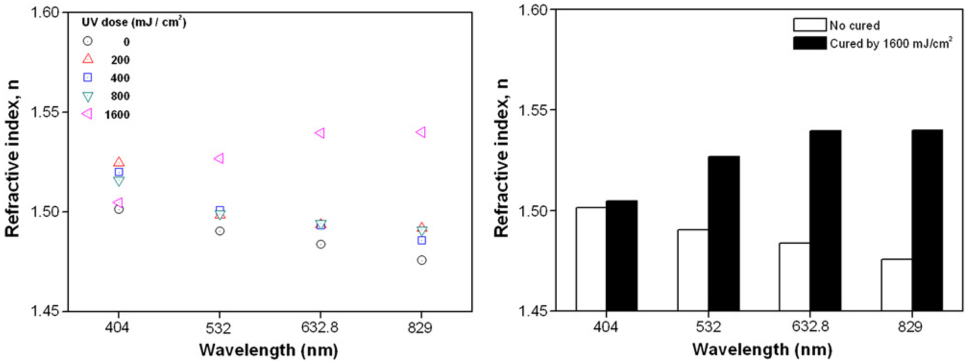

Fig. 5. Refractive index of UV-cured PSA-4 using DPHA (Semi-IPN structured).

$A^t_{N,C=C} = a_{C=C}\ x\ b\ x\ C_{C=C}$ where $a_{C=C}$ and $C_{C=C}$ represent the absorptivity and the concentration of C=C group while *b* represents path length. Providing the conversions and/or absorbance data in this manner makes thickness of the samples on ATR not important. The conversion of C=C bonds in the blend increased sharply as the UV dose was increased to 400 mJ/cm^2 regardless of the photoinitiator content, as shown in Fig. 9. Entanglement and/or orientation of multifunctional monomers around the acrylic copolymer may induce a rapid radical chain reaction, resulting in fast reactivity [7]. Also, the photoinitiator content accelerates the crosslinking reaction, but only a very small organic photoinitiator was present. Thus, the concentration or conversion of C=C bonds was not 100%. The remaining C=C bonds might have remained unreacted after the action of the photoinitiator because they were trapped in the crosslinked polymer network.

3.5. *Viscoelastic properties*

The viscoelastic properties that play a key role in the adhesion performance of acrylic PSAs blended with different UV doses were tested by ARES measurements [26]. The storage modulus (G′) is linked with the hardness of the adhesive, and the loss modulus (G″) is related to energy absorption. The values indicate the balance of the viscoelastic behavior. After UV irradiation, the adhesive strengths of various PSA compositions including DPHA decreased drastically compared with other compositions. Because of network formation via UV irradiation, this composition had a greater volume contraction that might yield microvoids at the interface between the adhesive and SUS surface, resulting in weak adhesion [27]. The effect of the higher T_g of the particular PSA is noticeable in the measured adhesion properties. Polymer T_g exerts influence upon the adhesion properties of the particular the PSA by determining the softness of the PSA, which is needed for flow of the adhesive and bonding of the PSA with the surface. Regarding our previous studies [28], it can be speculated that the adhesion

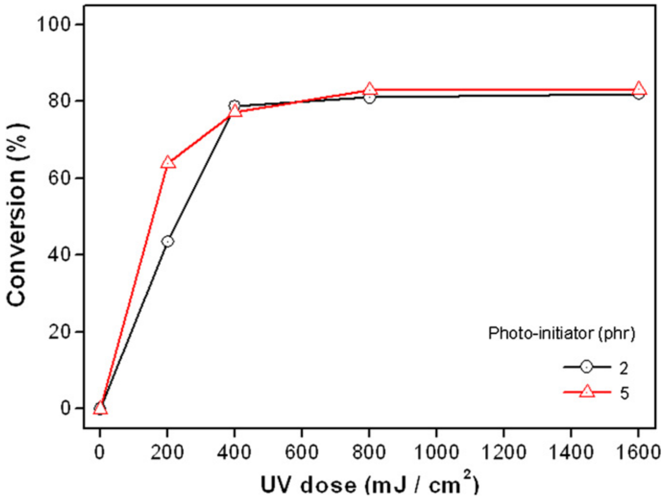

Fig. 9. Conversion of C=C bonds as a function of UV dose at 810 cm^{-1} with two different photoinitiator contents.

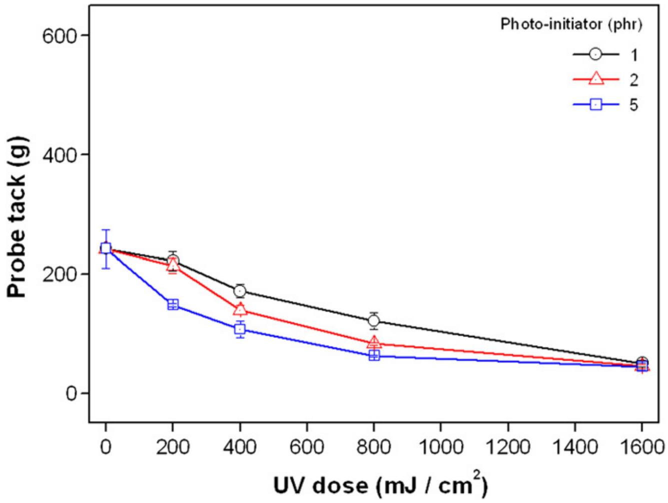

Fig. 7. Probe tack of UV-cured PSA-4 with photoinitiator content.

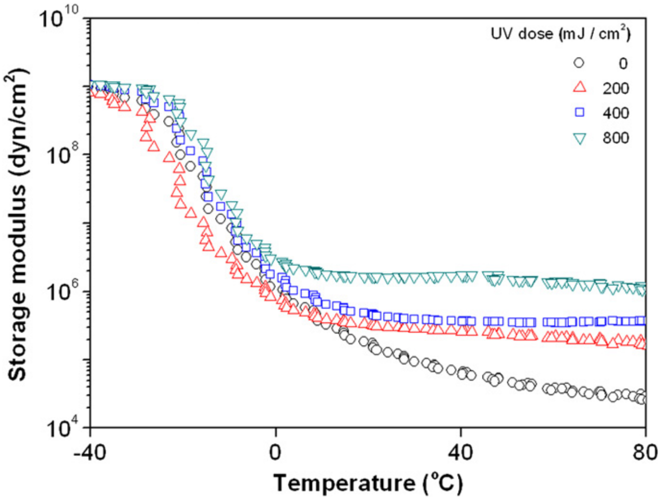

Fig. 10. Change in the storage modulus of UV-cured PSA-4 with UV dose.

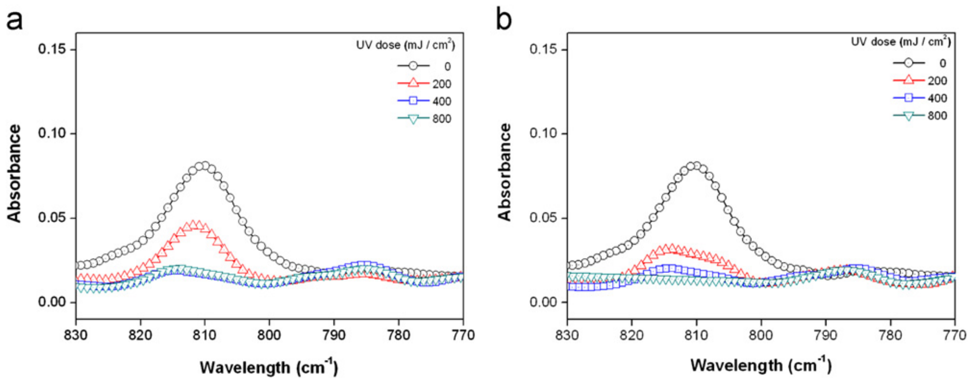

Fig. 8. FTIR spectra of UV-curable PSA-4 with (a) 2 phr and (b) 5 phr photoinitiator in DPHA.

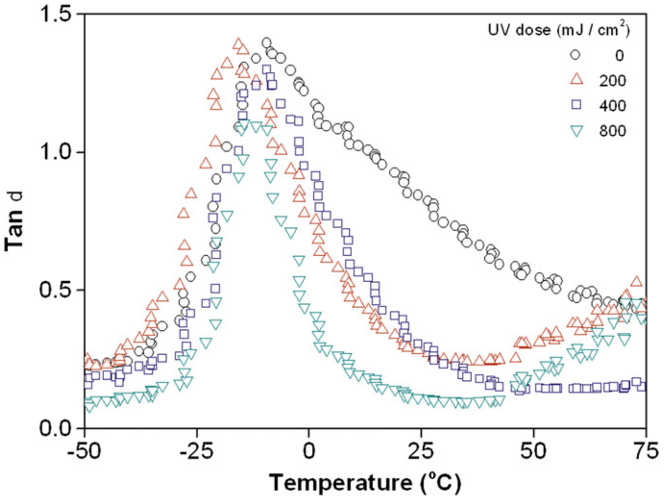

Fig. 11. Change in tan δ of UV-cured PSA-4 with UV dose.

properties decrease with increasing UV dose (and hence the T_g) as a consequence of poor wetting of the adhesive with the substrate. In Fig. 10, the plateau area of the storage modulus at the high temperature region suggests that acrylic PSA forms an entanglement structure. Further, a rubbery plateau at high temperatures is comparable to that in the acrylic PSA without a UV dose. Because the storage modulus at high temperature is directly related to the thermal resistance of acrylic PSAs, acrylic PSAs become more elastic at high temperature. Fig. 11 shows the tan δ curve of the acrylic PSA. The T_g of the acrylic PSAs (temperature at tan δ peak) slightly increased and values of the tan δ decreased with increasing UV dose. This suggests that the storage modulus of the acrylic PSAs is strongly related to UV dose at high temperatures.

4. Conclusion

Optically clear acrylic PSAs with different co-monomer side-chain lengths were synthesized and UV-cured. According to the afore-mentioned results, the following conclusions were adopted. Minor differences were determined among PSA-1, 2 and 3 in terms of the optical properties of transmittance and refractive index. But PSA-4, having lauryl acrylate as a co-monomer, showed lower values due to an inhomogeneous distribution in polymers. Also, the UV curing behavior of the acrylic PSAs was monitored by the change in FTIR. Using UV-curing, the refractive index was increased due to a crosslinked semi-IPN structured. The conversion of the remaining monomer into polymers may not have occurred after the action of the photoinitiator due to its being trapped in the crosslinked polymer network. The adhesion performance and viscoelastic properties of PSA-4 were quantified by measurements of a peel strength, probe tack and rheological performance. The peel strength of the acrylic PSAs decreased with increasing UV dose, and probe tack values followed the same trend as peel strength. However, slight decreases in measured peel and tack value values were noticeable as the amount of photoinitiator was increased from 1 to 2 and 5 phr (in DPHA). The storage modulus increased with increasing UV dose. Because polymer T_g exerts influence upon the adhesion properties by affecting the softness of the PSAs, UV dose, which is needed for curing, was related to the flow of the adhesive and bonding with the surface.

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