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Preparation and adhesion performance of UV-crosslinkable acrylic pressure sensitive adhesives

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Abstract—UV-crosslinkable acrylic pressure sensitive adhesives (PSAs) were synthesized by copolymerization of 2-ethylhexyl acrylate (2-EHA), vinyl acetate (VAc), acrylic acid (AA), 2-hydroxyethyl methacrylate (2-HEMA) and 4-acryloyloxydiethoxy-4'-chlorobenzophenone (unsaturated photoinitiator), with varying contents of 2-HEMA and photoinitiator, by solution polymerization. The UV-crosslinking behavior of the PSAs was studied by ATR-FT-IR spectroscopy, and PSA performance was characterized by probe tack, peel resistance and shear adhesion failure temperature (SAFT). As 2-HEMA acts as a good hydrogen donor to benzophenone, the efficiency of the photo-reaction was enhanced; thus with increasing contents of 2-HEMA and photoinitiator in the PSAs the incorporation of benzophenone groups in the PSAs, even at low UV doses, was quite fast. In addition, as the crosslinking reaction proceeds *via* photo-reaction mainly between 2-HEMA and photoinitiator, the probe tack and peel resistance of the PSAs having high concentrations of 2-HEMA and photoinitiator rapidly decreased in the early stage of UV irradiation due to increased crosslink density. These phenomena were also observed in the SAFT test, with the PSAs containing high levels of 2-HEMA and photoinitiator showing high SAFT values at low UV doses.

Keywords: Pressure-sensitive adhesive (PSA); UV-induced crosslinking; radiation; SAFT; adhesion performance.

1. INTRODUCTION

Pressure sensitive adhesives (PSAs) are commonly used in various industrial fields such as tapes, labels and medical products [1–4]. As PSAs are composed of viscoelastic materials, they show semi-solid properties that can be evaluated by tack, peel resistance and shear strength measurements.

Among the raw materials for the PSAs, an acrylic ester monomer is widely used in both solution and emulsion polymerization methods to synthesize PSAs.

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However, PSAs made from these methods are of linear structure and are crosslinked only by physical crosslinking, van der Waals forces or hydrogen bonds, and, thus, they exhibit limited mechanical and thermal properties [5]. In order to avoid these problems, chemical crosslinking is needed. Typically, as the crosslinking reaction of PSAs proceeds, PSAs turn into relatively non-tacky materials with decreased tack and peel resistance [6]. Therefore, the degree of crosslinking should be carefully controlled. On the other hand, crosslinking is achieved by using additional reagents, so these systems suffer from the disadvantage of being composed of two components, PSA and the crosslinking agent, and this reduces their pot life [4]. Among the crosslinking methods recently used for PSAs, radiation curing is adapted due to its economical (fast curing) and environmental (low VOC) advantages [6–13].

UV-curing techniques used in the PSA crosslinking process can be divided into UV-polymerization and UV-crosslinking methods. UV-polymerizable PSAs are formulated from oligomers, monomers, tackifiers and photoinitiators by blending in a reactor above the glass transition temperature (T_g) of the tackifier, and are then polymerized by UV irradiation after being coated on a carrier. The main parameters to be considered for UV-polymerizable PSAs are the type, molecular weight, polarity, as well as functionalities and blending ratio of oligomers and monomers. Photoinitiator type and the kind of tackifier are also important in the manufacturing of PSAs. UV-crosslinkable PSA systems are similar to UV-polymerizable PSAs for which the crosslinking reaction proceeds by UV irradiation, but the molecular weight of the UV-crosslinkable PSAs is higher than that of the oligomers or monomers used in UV-polymerizable PSAs and as they are also copolymerized with unsaturated photoinitiator, they have UV-reactive sites in the PSA polymer backbone. The UV-crosslinkable systems have been investigated in the fields of photoresists, industrial coatings and inks for 40 years [13, 14].

UV-crosslinkable PSAs are commercially available and their adhesion properties have been studied [9, 15, 16]. However, these investigations have been limited to variations only in curing conditions, UV dose, curing temperature and film thickness. Consequently, more studies about UV-crosslinkable PSA polymers are required.

The use of unsaturated photoinitiators, such as 4-acryloyloxy benzophenone (ABP) and O-acryloyl acetophenone oxime (AAPO), which are copolymerizable, have been studied by many researchers [16–20] and the ABP has been adapted in UV-crosslinkable PSA investigations [7, 8]. As the ABP consists of an unsaturated group and benzophenone [7], it can be copolymerized in an acrylic PSA, and can give a PSA with structure having pendant benzophenone groups in the polymer side chain. Benzophenone groups in the PSA side chain are hydrogen abstractors; they abstract hydrogen from the hydrogen donor group in the acrylic PSA polymer when irradiated by UV.

In particular, if the hydrogen donor groups are alcohol, ether or amine, the excited benzophenone group can abstract more hydrogen from these groups than from the hydrocarbon groups [21].

The objective of this study was to prepare UV-crosslinkable acrylic PSAs which were able to cure at low UV doses and to elucidate the effect of crosslinking on PSA performance with varying UV dose. The PSAs investigated in this study were synthesized by solution polymerization method using 2-ethylhexyl acrylate (2-EHA), vinyl acetate (VAc) and acrylic acid (AA), and with varying contents of 2-hydroxyethyl methacrylate (2-HEMA) and 4-acryloyloxydiethoxy-4'-chlorobenzophenone (P-36). The UV-crosslinking of the PSAs was monitored by ATR-FT-IR spectroscopy, and PSA performance was evaluated by probe tack, peel resistance and shear adhesion failure temperature (SAFT) measurements at various UV doses.

2. EXPERIMENTAL

2.1. Preparation of UV-crosslinkable acrylic PSAs

2-EHA, VAc, AA and 2-HEMA used for preparing UV-crosslinkable acrylic PSAs were obtained from Junsei Chemicals (Japan) and used as received. The double bond containing photoinitiator (P-36, Fig. 1) was obtained from SK CYTEC (South Korea). Ethyl acetate used as solvent was purchased from Junsei Chemicals and used without further purification.

The UV-crosslinkable acrylic PSAs were prepared by solution polymerization in ethyl acetate as 50 wt% solids. The polymerization was performed in a 500-ml, four-necked, round-bottom flask equipped with a thermometer, condenser, dropping

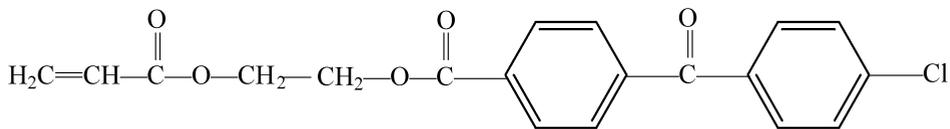


Figure 1. Structure of 4-acryloyloxydiethoxy-4'-chlorobenzophenone (P-36).

Table 1.
PSA compositions

Code	2-EHA (wt%)	VAc (wt%)	AA (wt%)	2-HEMA (phr)	P-36 (phr)
SH0P1	80	15	5	0	1
SH3P1	80	15	5	3	1
SH6P1	80	15	5	6	1
SH9P1	80	15	5	9	1
SH3P05	80	15	5	3	0.5
SH3P2	80	15	5	3	2

The solid content of all synthesized PSAs was 50 wt%. phr, parts per hundred resin.

funnel and mechanical stirrer. The typical synthesis method was as follows (see Table 1, using SH3P2 as an example). The flask was charged with 50% of the monomer and solvent mixture of 105 g ethyl acetate, 80 g 2-EHA, 15 g VAc, 5 g AA, 3 g 2-HEMA and 2 g P-36. The mixture was heated at 80°C in a heating mantle for 2 h and after the initiation of reaction, the remaining 50% of the monomer and solvent mixture was added during 1 h. Then the flask was kept at 80°C for 3 h. All syntheses were carried out in a flask wrapped with an aluminum foil to minimize the light effect.

2.2. Methods

2.2.1. UV curing of acrylic PSAs. All UV-crosslinkable acrylic PSAs were coated onto a corona-treated poly(ethylene terephthalate) (PET, SK Chemical, South Korea) film of 25 μm thickness using a bar coater (No. 26), kept at room temperature for 1 h and then dried in an oven at 70°C for 20 min. These dried films were kept at 22 ± 2 and $60 \pm 5\%$ RH for 24 h before testing. All UV-curable films were cured in a conveyer-belt-type UV-curing machine equipped with a high-pressure mercury lamp (100 W, main wavelength 340 nm). In order to avoid the heat effect from the mercury lamp, a cold mirror was used as a reflector. The UV doses used were 0, 210, 630, 1050, 1470, 1890, 2310 and 2730 mJ/cm^2 . The UV doses were measured with an IL 390C Light Bug UV radiometer (International Light, USA).

2.2.2. ATR-FT-IR spectroscopy. The IR spectra of UV-cured PSAs were obtained using a Nicolet Magna 550 Series II FT-IR (Midac, USA) equipped with a 45° zinc selenide (ZnSe, $n = 2.4$) attenuated total reflectance (ATR) accessory. 32 scans were collected at a resolution of 8 cm^{-1} between 650 and 4000 cm^{-1} . The spectrometer was linked to a PC equipped with Omnic E.S.P 5.2 software to collect the IR spectra and to integrate the peak area.

2.2.3. DSC measurements. Differential scanning calorimetry (DSC, TA Instruments, Q-1000) measurements were carried out using a constant sample weight of 20 mg in aluminum pans under a flowing 50 ml/min atmosphere of nitrogen at a heating rate of $10^\circ\text{C}/\text{min}$ from -70°C to 150°C .

2.2.4. Gel content. The cured PSA samples were weighed and immersed in toluene for 4 days at 40°C and then dried until the weight was constant. The gel content of the samples was calculated by the following equation.

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

where W_t is the weight after immersion and W_0 is the weight before immersion.

2.2.5. PSA performance. PSA performance was evaluated by probe tack, peel resistance and SAFT measurements. The probe tack and peel resistance tests were conducted using a Stable Microsystem (USA) instrument (model TA-XT2i). The probe tack test with a polished stainless steel cylindrical probe of diameter 5 mm was carried out at a separation rate of 0.5 mm/s under a constant pressure of 100 g/cm² and a dwell time of 1 s. The peel resistance tests were performed after applying the PSA samples (25 mm × 300 mm, width × length) to stainless steel (304 type) and keeping the test sample at room temperature for 24 h, at an angle of 180° with a crosshead speed of 300 mm/min. For SAFT measurements, the PSA samples were pressed onto stainless steel (304 type) (bonding area 25 mm × 25 mm) by two passes of a 2 kg rubber roller. The samples were hung in the oven, and then a weight of 1 kg was hung at the end of each sample. The oven was heated from 25°C to 200°C at a heating rate of 0.4°C/min.

3. RESULTS AND DISCUSSION

3.1. ATR-FT-IR measurements

Benzophenone, type II, is the most widely used photoinitiator in photochemistry. When exposed to UV, the carbonyl group in the benzophenone is excited to a triplet state through a singlet state. These triplets abstract hydrogens from suitable hydrogen donor groups such as alcohol, alkyl benzene, hydrocarbon and tributyl hydride [21, 22]. Therefore, if a benzophenone derivative containing vinyl double bond is copolymerized *via* radical polymerization in the polymer backbone, it can work as a crosslinker when exposed to UV (Fig. 2) [8, 23].

Because the conversion of the C=O group in benzophenone leads to a loss of conjugation between the carbonyl group and the aromatic ring on UV exposure, the kinetics of UV curing of the acrylic PSAs containing benzophenone group can

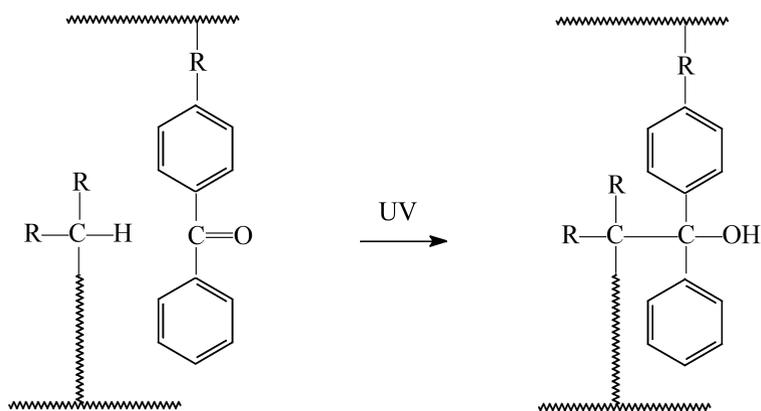


Figure 2. UV-crosslinking reaction of PSA *via* the hydrogen abstraction process [8].

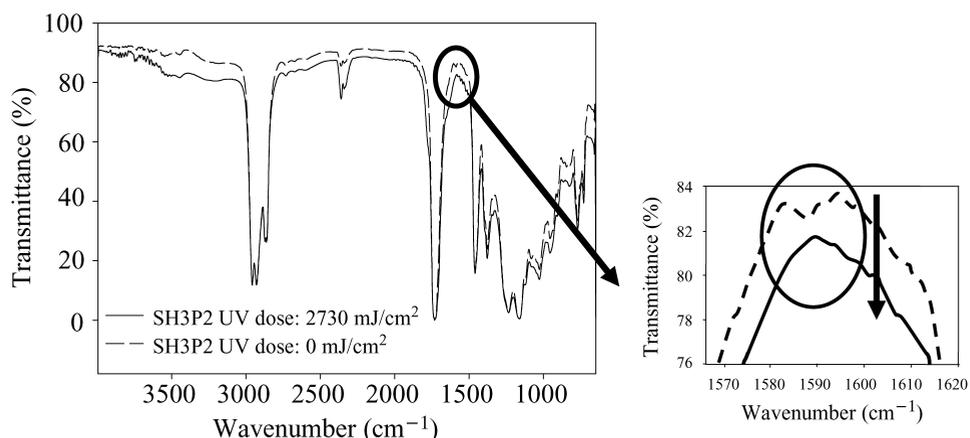


Figure 3. ATR-FT-IR spectra of benzophenone groups in SH3P2 at UV doses of 0 and 2730 mJ/cm².

be evaluated by observing their characteristic absorption band at about 1600 cm⁻¹ (C=C in C₆H₅ of benzophenone group) with increasing UV dose [24].

Figure 3 shows the ATR-FT-IR spectra of synthesized SH3P2 PSA at UV doses of 0 and 2730 mJ/cm². The peak of the C=C at about 1600 cm⁻¹ in the aromatic ring almost vanished at the dose of 2730 mJ/cm². The C=C in benzene ring shows strong stretching vibrations at about 1500 cm⁻¹, and in the case of this group is conjugated with the C=O, a band at about 1600 cm⁻¹ can be observed. Therefore, the loss of conjugation of C=C and C=O in bezophenone can be estimated by observing the band at about 1600 cm⁻¹. As the loss of conjugation C=C and C=O in benzophenone is due to hydrogen abstraction of C=O from the neighboring hydrogen donor groups with increasing UV dose, and, furthermore, if the neighboring hydrogen-donor groups contain hydroxyl group (like 2-HEMA), the rate of the loss of conjugation C=C and C=O will increase. And also by observing the band at about 1600 cm⁻¹, the effect of benzophenone content on UV curing of PSA can be evaluated.

So in order to evaluate the effects of 2-HEMA as a good hydrogen donor and content of photoinitiator on the kinetics of crosslinking of UV-crosslinkable acrylic PSAs, the relative concentration of C=C in the benzophenone was calculated by integrating the peak area between 1577 and 1604 cm⁻¹.

Figure 4 shows the decrease in the relative concentration of the C=C bond in the benzophenone of SH0P1 and SH3P1 with increasing UV dose. As shown in Fig. 4, the relative concentration of the C=C bond in the benzophenone of SH3P1 decreased faster than that of SH0P1 at 210 mJ/cm². However, at UV doses above 630 mJ/cm², there was no difference in the relative concentrations of the C=C in the benzophenone of SH3P1 and SH0P1. These effects are due to the higher hydrogen-donating ability of 2-HEMA which has a hydroxyl group.

In Fig. 5, the influence of the benzophenone content is shown. The relative concentration of the C=C bond in the benzophenone of SH3P2 is lower than that

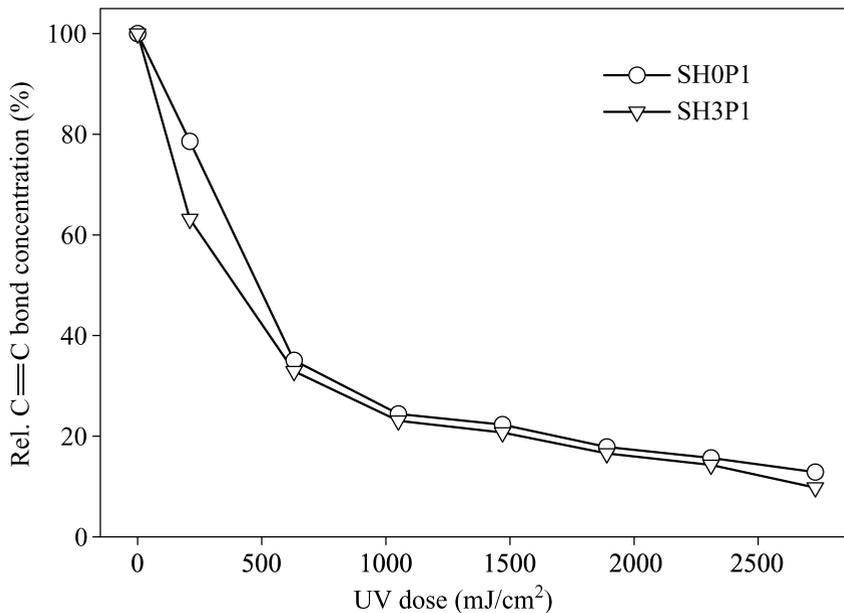


Figure 4. Change in relative concentration of C=C bond in SH0P1 and SH3P1 with varying UV dose.

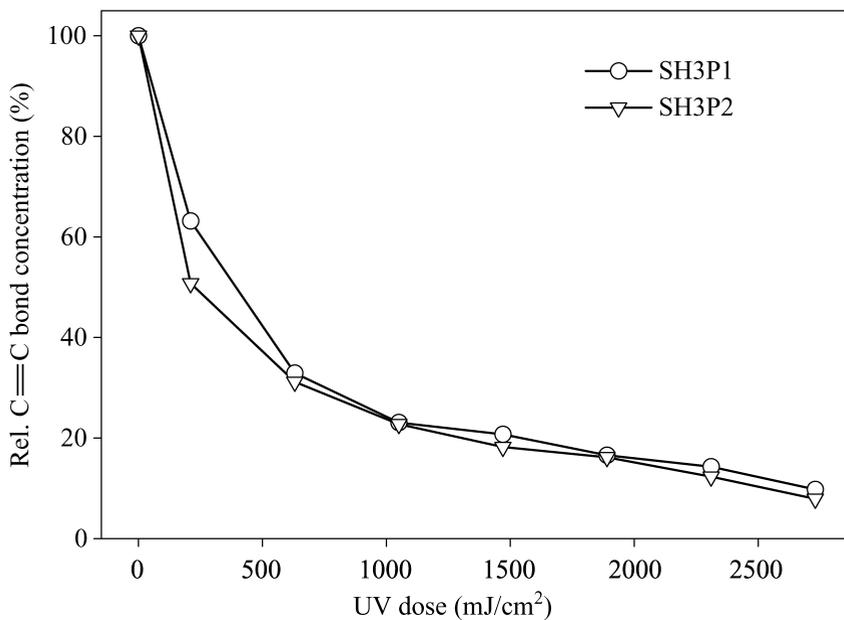


Figure 5. Change in relative concentration of C=C bond in SH3P1 and SH3P2 with varying UV dose.

of SH3P1 at a UV dose of 210 mJ/cm². This effect may be because the higher content of benzophenone in the PSA increased the efficiency of UV curing at low UV dose. But at UV doses above 630 mJ/cm², the relative concentrations of the C=C bond were almost the same; this may be because the increased crosslink density reduced the mobility of the PSAs. This phenomenon was also found in previous investigations in that the maximum UV-curing rate was achieved at an earlier stage of UV irradiation with increasing photoinitiator content but after this point, the increased crosslink density reduced the molecular mobility, so the curing rate decreased [23–25]. This result can be verified by using equation (2) in which the polymerization rate (R_p) is given as follows [23].

$$R_p = \frac{k_p}{k_t^{0.5}} [M] \{ \phi_i I_0 (1 - \exp(-2.303 \varepsilon [PI] d)) \}^{0.5}, \quad (2)$$

where k_p and k_t are rate constants of propagation and termination, respectively, $[M]$ is monomer concentration, ϕ_i is initiation quantum yield of photoinitiator, $[PI]$ is the photoinitiator concentration, d is the film thickness and ε is the molar extinction coefficient. According to equation (2), the polymerization rate should increase as the photoinitiator content increases.

3.2. Gel content

The synthesized PSA polymers have UV-crosslinkable pendant groups in the backbone at which crosslinking reaction occurs when irradiated by UV. The increased gel content means that the crosslinking reaction has occurred, so the PSA gel content was calculated before and after irradiation at various UV doses. In Fig. 6, the changes in gel content of SH0P1, SH3P1 and SH9P1 with varying UV dose (0, 210, 630, 1470, 2310 mJ/cm²) are shown. Before irradiation, the gel contents of all samples were 0%, but after irradiation they increased up to about 60% at 2310 mJ/cm². Especially SH9P1 had nearly 3 times the gel content of SH0P1 at a UV dose of 210 mJ/cm². This means that the rate of crosslinking reaction in SH9P1 was faster than that in SH0P1, and these results correspond exactly to results from ATR-FT-IR. The effect of photoinitiator concentration on gel content is shown in Fig. 7. As previously mentioned, the rate of photopolymerization increased with increasing photoinitiator content. This could be verified by calculating the gel content of SH3P1 and SH3P2 with varying UV dose. SH3P2 showed higher gel content than SH3P1 at higher photoinitiator contents. The gel content of SH9P1 was higher than that of SH3P2 at 210 mJ/cm², indicating that 2-HEMA contributed more active sites for the crosslinking than the photoinitiator did.

3.3. PSA performance

3.3.1. Probe tack. Figure 8 shows the change in probe tack with varying 2-HEMA content at various UV doses. The probe tack of SH3P1, SH6P1 and SH9P1 decreased considerably at a UV dose of 210 mJ/cm², but above 630 mJ/cm²,

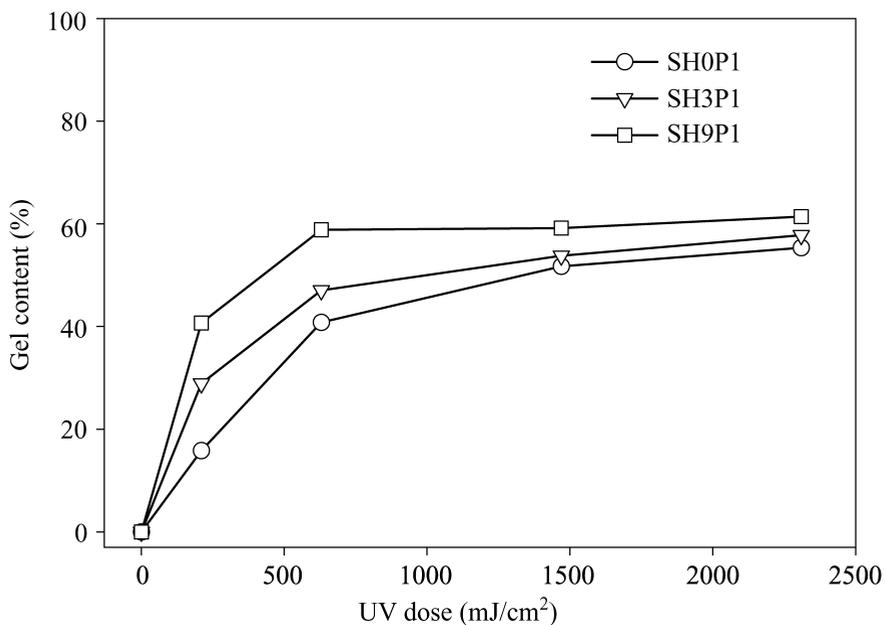


Figure 6. Change in gel content in SH0P1, SH3P1 and SH9P1 with varying UV dose.

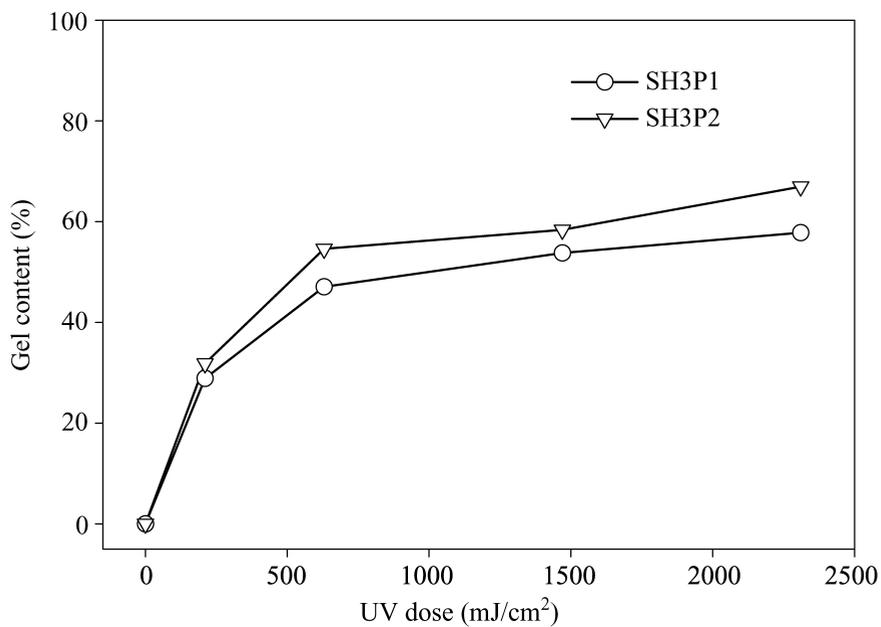


Figure 7. Change in gel content in SH3P1 and SH3P2 with varying UV dose.

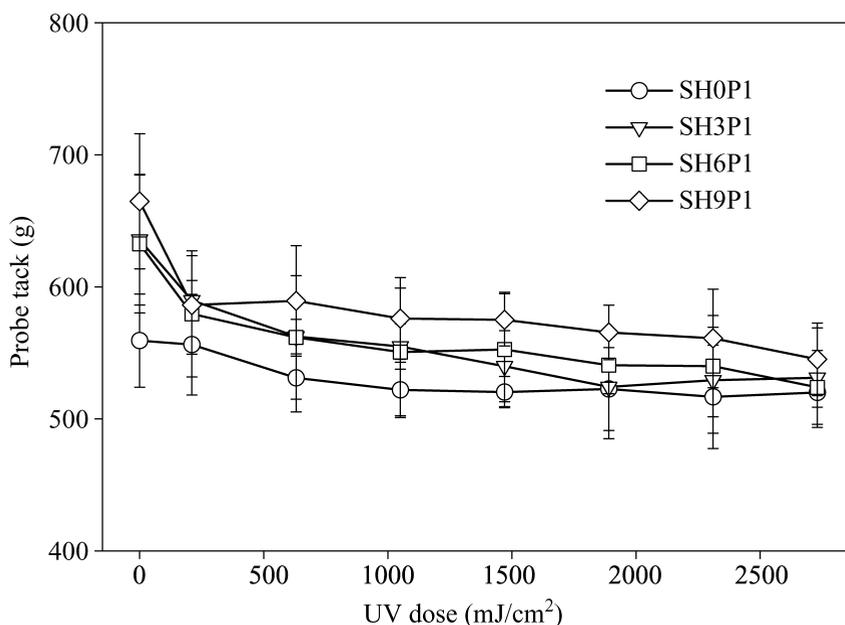


Figure 8. Change in probe tack of SH0P1, SH3P1, SH6P1 and SH9P1 with varying UV dose.

the probe tack decreased only slightly. The relative decrease in the probe tack (D) at 210 mJ/cm² compared with probe tack before UV exposure was calculated by following equation:

$$D(\%) = \left(1 - \frac{\text{tack}_{210\text{mJ/cm}^2}}{\text{tack}_{0\text{mJ/cm}^2}} \right) \times 100. \quad (3)$$

The decreased values for SH0P1, SH3P1, SH6P1 and SH9P1 were 0.6, 7.3, 8.4 and 11.8%, respectively.

These results can be explained by the fact that as the increased 2-HEMA acted as a more efficient hydrogen donor than any other hydrocarbon in the UV-crosslinking system, crosslink density increased at low UV doses, thereby reducing the molecular mobility and decreasing the probe tack. The effect of photoinitiator concentration on the probe tack is shown in Fig. 9. As at 210 mJ/cm² the rate of crosslinking reaction of SH3P2, having a higher content of photoinitiator, was faster than that of SH3P05, the increased crosslink density hindered the PSA from wetting the probe, which consequently reduced the probe tack more in SH3P2. The decrease in probe tack values for SH3P2 and SH3P05 were 15.7% and 1.2%, respectively.

In order to exhibit good performance as a PSA, the PSA polymer must have a T_g far below the room temperature, depending on its applications [3]. The T_g of a PSA can be adjusted by formulating with a low T_g material (soft polymer) and high T_g additives (hard polymers or oligomers). As the T_g of a PSA is lowered, both tack

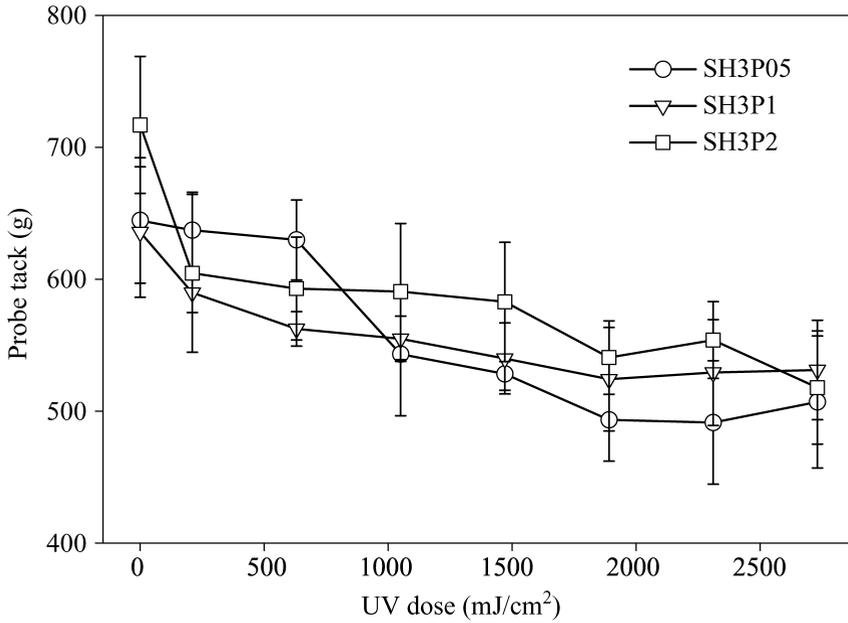


Figure 9. Change in probe tack of SH3P05, SH3P1 and SH3P2 with varying UV dose.

Table 2.

Glass transition temperature (T_g , °C) of PSAs with varying UV dose

PSA	UV dose (mJ/cm ²)			
	0	210	1470	2310
SH0P1	-61.1	-45.6	-42.7	-42.2
SH3P1	-61.0	-45.4	-42.6	-42.8
SH6P1	-62.3	-44.5	-42.6	-43.6
SH9P1	-61.9	-49.6	-47.8	-46.0
SH3P05	-62.1	-48.7	-46.3	-46.6
SH3P2	-61.3	-40.0	-40.8	-40.3

and elongation at break increase, but hardness and tensile strength decrease [26]. Table 2 shows the T_g results for all PSA samples with varying UV dose. Before UV irradiation of PSA samples, they exhibit a T_g of about -60°C , and with increasing UV dose, the T_g of all samples increased. These results correspond to probe tack results in that before UV irradiation, PSA samples (lower T_g) showed high probe tack, and after UV irradiation (higher T_g), probe tack was lowered. However, with increasing UV dose, the increment of T_g was negligible and also probe tack for all samples did not show much difference at high UV dose. The T_g and probe tack results showed similar tendency.

3.3.2. Peel resistance. The peel resistance results of SH0P1, SH3P1, SH6P1 and SH9P1 are shown in Fig. 10. At UV doses above 630 mJ/cm^2 , all of the PSAs gave equal values, but for low UV doses (210 mJ/cm^2) they showed different behaviors in peel resistance. The peel resistance values of SH6P1 and SH9P1, having a higher 2-HEMA content, were reduced by 53.3% and 52.7%, respectively, at 210 mJ/cm^2 . In contrast, the peel resistance values of SH0P1 and SH3P1 were reduced only by 8.9% and 15.1%, respectively, at the same UV dose. These results compare well to those of the probe tack in which the increased content of 2-HEMA caused the PSA to crosslink in the early stage of UV exposure to produce a rapid decrease in peel resistance. For example, the high crosslink density of PSAs further increases the volume contraction and the storage modulus, thereby decreasing the peel resistance to low values [25, 26]. The effect of photoinitiator content in the PSAs is shown in Fig. 11. As expected, the higher photoinitiator content in SH3P2 produced a decrease of peel resistance by 69.8% at a UV dose of 210 mJ/cm^2 , whereas SH3P05 showed only a 9.1% reduction in peel resistance at the same dose.

Probe tack and peel resistance tests are almost the same in that both tests involve a bonding step and a debonding step [7]. However, there is a difference in measurements between the probe tack and peel resistance; peel resistance is measured after a relatively long contact time after application of a light or medium pressure, whereas probe tack is measured after a short contact time, or by a light pressure [27]. So the peel resistance is also influenced by the T_g in that if the T_g of the PSA is sufficiently low, the wettability will be high after applying to the substrate and as a result the peel resistance will also increase [27, 28].

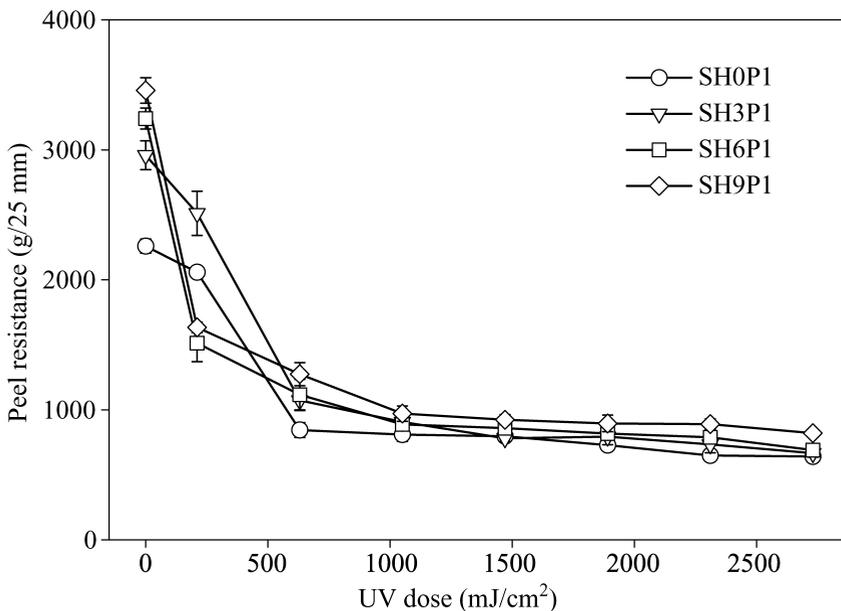


Figure 10. Change in peel resistance of SH0P1, SH3P1, SH6P1 and SH9P1 with varying UV dose.

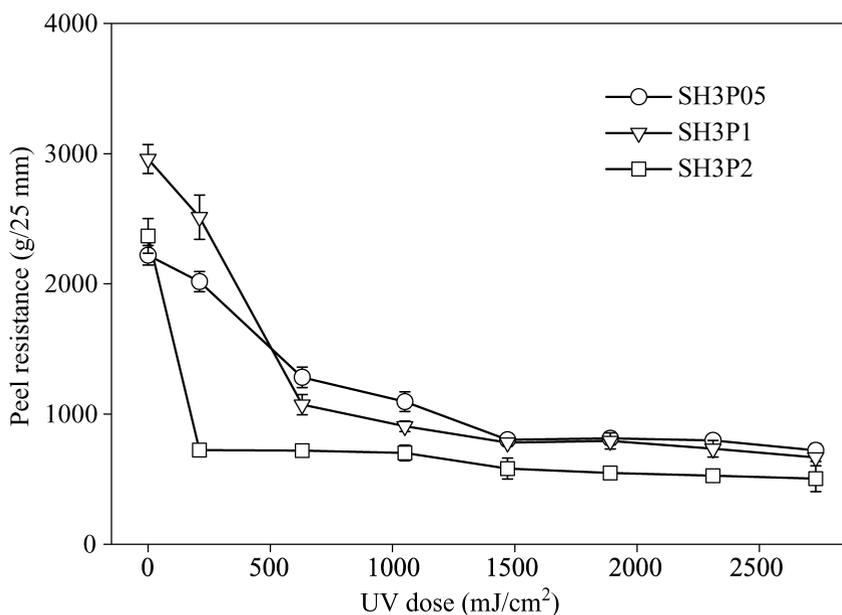


Figure 11. Change in peel resistance of SH3P05, SH3P1 and SH3P2 with varying UV dose.

The T_g results, shown in Table 2, and the peel resistance results showed similar tendency in that before UV irradiation of PSA (lower T_g), all PSAs exhibited higher peel resistance and after UV irradiation (higher T_g), peel resistance values were lowered.

3.3.3. Shear adhesion failure temperature (SAFT). In general, an acrylate copolymer which has no crosslinking, or which is crosslinked only by hydrogen bonding, does not show an adequate PSA performance at an elevated temperature, so chemical crosslinking is required to provide high shear strength [29]. The shear strength of a PSA is measured by dynamic or static method [30]. In the dynamic shear test, SAFT, the failure temperature, is measured by pulling the PSA downward under a constant force from a vertically placed test substrate in a direction parallel to the bonding surface, while the temperature is gradually raised. On the other hand, in the static shear test, the failure time is measured by pulling the PSA downward under a constant load at a fixed temperature [29].

The SAFT results on PSAs with varying contents of 2-HEMA are shown in Fig. 12. With increasing UV dose, the PSAs showed higher SAFT values. Especially, SH9P1, with higher 2-HEMA content, showed high SAFT values at low UV doses and reached a SAFT level of 150°C earlier than SH3P1 and SH0P1. This means that 2-HEMA acted as a good hydrogen donor to the benzophenone group, thereby allowing an increased crosslink density at a lower UV dose to increase the PSA polymer cohesion. Figure 13 presents the effects of photoinitiator concentration on SAFT at various UV doses. As the rate of photopolymerization is

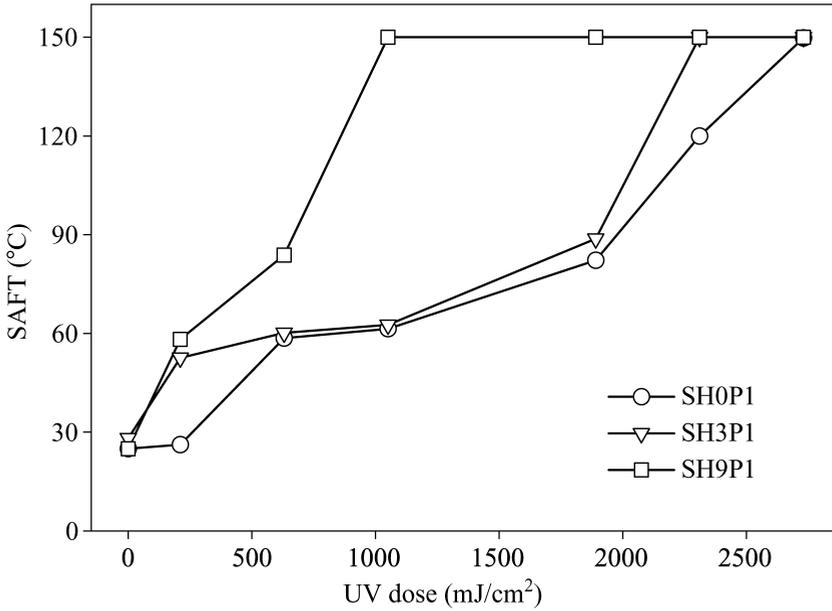


Figure 12. Change in SAFT of SH0P1, SH3P1 and SH9P1 with varying UV dose.

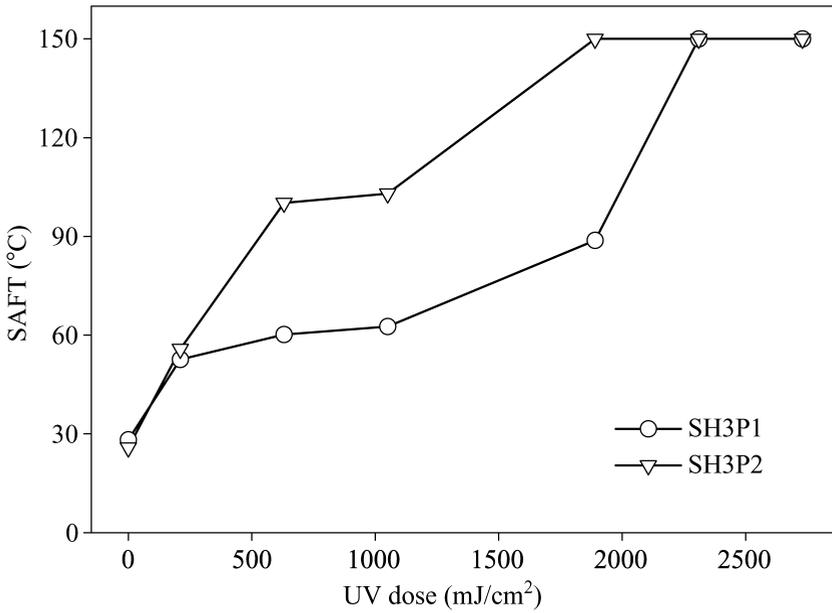


Figure 13. Change in SAFT of SH3P1 and SH3P2 with varying UV dose.

proportional to the photoinitiator concentration, the increased photoinitiator in the PSA produces a high crosslink density at low UV doses. As a result, the SAFT values of SH3P2 were higher than those of SH3P1.

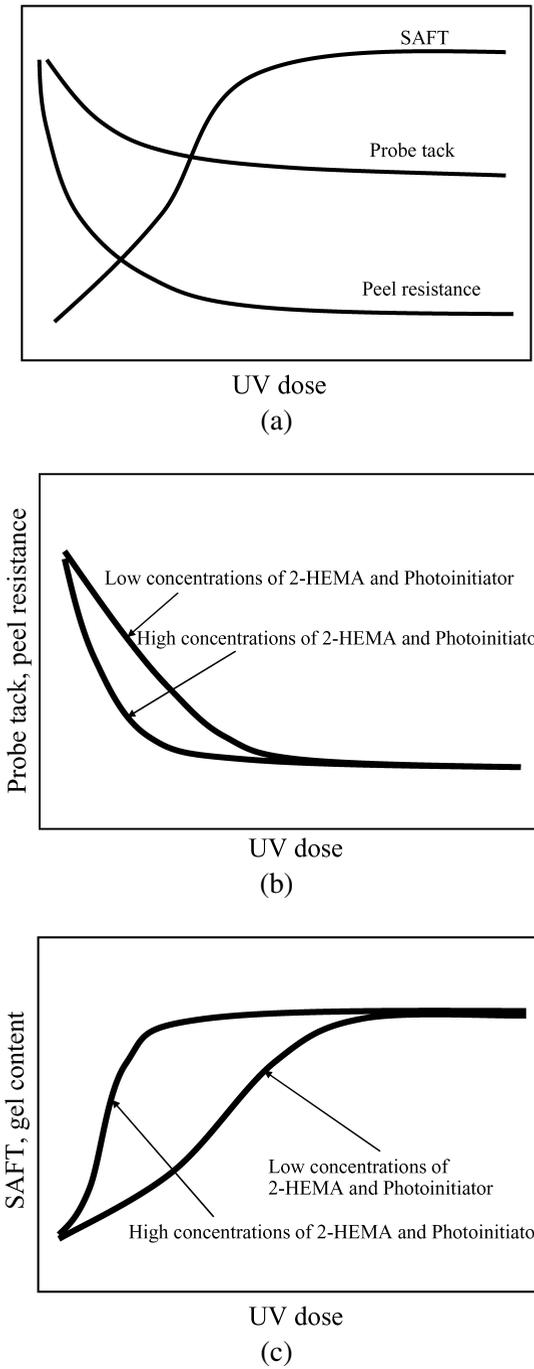


Figure 14. Schematic illustrations of changes in PSA performance with variations in (a) UV dose (b, c) 2-HEMA and photoinitiator concentrations.

3.4. Schematic illustration

Schematic illustrations of the PSA performance are shown in Fig. 14. As shown in Fig. 14, the variables in this study were classified into three groups: UV dose, 2-HEMA concentration and photoinitiator concentration.

With increasing UV dose on PSAs, the values of both probe tack and peel resistance gradually decreased, whereas the SAFT value increased. In addition, the PSA containing higher concentrations of both 2-HEMA and photoinitiator showed a rapid decrease of both probe tack and peel resistance values and a high SAFT value in the early stage of UV exposure.

4. CONCLUSIONS

Of the various methods to prepare UV-curable PSAs, that of preparing a UV-crosslinkable acrylic PSA was adapted to evaluate the UV-curing behavior and adhesion performance at various UV doses. The UV-crosslinkable PSAs were synthesized by copolymerization of acrylate monomers and an unsaturated photoinitiator, P-36, with varying contents of 2-HEMA and photoinitiator.

From the ATR-FT-IR results the rate of crosslinking of the PSAs having higher contents of 2-HEMA and photoinitiator was faster than that of the PSAs with lower contents of 2-HEMA and photoinitiator. The PSA performance, in terms of probe tack, peel resistance and SAFT, was also affected by the curing rate. With increasing concentrations of 2-HEMA and photoinitiator in the PSAs, the probe tack and peel resistance rapidly reduced at the beginning of UV exposure, whereas the SAFT values increased.

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