

Original Article

Characterization and flexibility properties of UV LED cured acrylic pressure-sensitive adhesives for flexible displays



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ABSTRACT

The era of the Internet of Things (IoT) is becoming a reality, and the display equipment is at its center. The display device is the window that connects the IoT and humans, and is evolving into various form factors convenient to human use, such as foldable mobile phone, flexible wearable device, paper-like display, and rollable TV. Even though transparent flexible electrodes and substrates have been developed, their assembly remains a challenge. Therefore, the importance of optically clear acrylic pressure-sensitive adhesives (PSAs) that ensure device integrity by easily stretched and mitigating the stress during repeated stretching and bending process is being emphasized. In this study, elongation and recovery features were identifiable as key flexible properties of PSAs. These properties were estimated by measuring shear strain and strain recovery using a universal testing machine and by dynamic mechanical analytics respectively. Compared to conventional UV metal halide curing, a newly introduced ultraviolet light-emitting diode (UV LED) curing process resulted in different PSA property variables, namely UV exposure intensity and UV exposure time. Fully cured acrylic PSA realized under UV LED exposure level 30 for 8 s demonstrated the highest shear strain with excellent strain recovery of ~23,500% and 94% respectively. Under these conditions, effective material adhesion performance was achieved while retaining optical transparency and thus fulfilling the dual property requirement for non-opaque material adhesiveness necessary for use in flexible displays. © 2020 The Authors. Published by Elsevier B.V. This is an open access article under the CC

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1. Introduction

Pressure-sensitive adhesives (PSAs) are defined by the ease of 'low pressure' attachment to a variety substrates and can be easily detached from partner substrates without any adhesion failure [1,2]. PSAs have been used in a wide range of industrial fields such as medical products, electronics, automobiles and displays [3–9]. PSAs are classified according to monomer type which include acrylic PSAs, rubber PSAs and silicone PSAs. Among these PSAs, acrylic PSAs have been broadly used because of the availability of a wide range of acrylic monomers, their optically clear properties and their compatibility with high-performance ultraviolet (UV) curing systems [9,10].

In particular, acrylic PSAs have been used as adhesives to assemble the layered structures of display devices [11–14]. As display technology has changed from rigid-type to flexible displays, flexibility has become significantly important for all display components [13,15-18]. PSAs are also essential display elements as they are functionally important for combining the component layers. For this reason, PSAs used in flexible displays should have sufficient flexibility tolerance under stress during repeated folding to prevent fracture or delamination of the display products. The flexibility characteristics of PSAs include both elongation and recovery. When the display is repeatedly bent and relaxed, tensile stress and compressive stress occur within inter-layers of PSAs. The high elongation capability of PSAs enables them to endure these stresses and maintain their structural integrity without adhesion failure during bending. Furthermore, the high recovery property of PSAs enables the restoration of stretched PSAs without buckling during reverse bending cycles.

UV curing systems have been widely utilized in the industry with key advantages ranging from rapid curing reactions, solvent-free formulation, low cost, low energy consumption and ambient-temperature processing [19-21]. For display applications, UV curability and the excellent clarity properties of acrylic PSAs favorably support their use and suitability for UV curing systems [9,11]. Our research group has consistently studied UV-curable adhesives using conveyor-type UV curing devices equipped with conventional UV mercury lamps [11,22-24] and more recently as UV metal halide lamps [12]. Using this device, UV exposure time can be controlled by conveyor speed with a fixed UV exposure intensity; thus, total UV exposure is determined as the product of fixed intensity and exposure time. However, this type of UV curing device has several shortcomings including heat emission causing thermal deformation or unwanted thermal reactions and difficulty in constant precise control of UV exposure by the conveyor. Therefore, a UV light-emitting diode (LED) curing device (Fig. S1) was newly introduced for UV-curable PSAs; it has advantages such as a more compact design, selective UV wavelength, almost constant temperature due to the lack of heat emission, accurate and uniform curing due to the densely arranged lamps, and easily controllable UV exposure by exposure level and time [25-30]. Despite its strengths, research studies on UV LED curing of PSAs are scarce. However, UV LED lamps are available for constant UV irradiation with small deviation and extremely high UV exposure above 10,000 mJ/cm², as shown in Fig. S2.

These differences in curing methods are expected to lead to improved properties and better curing reactions.

In this study, acrylic PSA pre-polymers were synthesized from reactive acrylate monomers with a radical initiator and crosslinking agent and analyzed to measure their basic properties such as molecular weight and glass transition temperature. Acrylic PSA films were prepared under several curing conditions using the UV LED curing device. The UV curing behaviors, optical properties, adhesion performance, and flexibility of the acrylic PSAs were investigated through gel fraction and Fourier transform infrared spectroscopy (FTIR) conversion, UV—vis transmittance, 180° peel strength, and shear strain and strain recovery. The purpose of this study is to develop an acrylic PSA with both excellent shear deformation and high recovery properties and to provide data for application in flexible displays.

2. Materials and methods

2.1. Materials

2-Ethylhexyl acrylate (2-EHA, 99.0% purity), isobutyl acrylate (IBA, 99.0% purity), acrylic acid (AA, 99.0% purity), methyl methacrylate (MMA, 99.0% purity), and 2-hydroxyethyl acrylate (2-HEA, 99.0% purity) were purchased from Samchun Pure Chemical (Republic of Korea). Additionally, 2-Hydroxy-2-methylpropiophenone (Irgacure 1173) was purchased from BASF (Germany) and used as a photo radical initiator. Poly (ethylene glycol 200 dimethacrylate) (PEG200DMA, Sartomer SR210) was purchased from Sartomer (USA) and used as a crosslinking agent.

2.2. Synthesis of acrylic PSA pre-polymer

The pre-polymer was prepared using 2-EHA (64 wt%), IBA (19 wt%), AA (3 wt%), MMA (4 wt%), and 2-HEA (10 wt%) by bulk radical polymerization with Irgacure 1173 (0.1 phr). Polymerization was performed using a UV spot curing device in a 500-mL four-necked round-bottomed flask with a stirring rate of 150 rpm at 80 $^{\circ}$ C under N₂ purging.

2.3. UV LED curing of acrylic PSA film

After purging for 1 h, PEG200DMA (1 phr) as a crosslinking agent and Irgacure 1173 (1 phr) as a photo radical initiator were added to each 58-mL container. The mixture was blended using a planetary centrifugal mixer ARE-310 (Thinky, Japan) at 600 rpm for 1 min. The acrylic PSA was coated onto the 75-µm-thick corona-treated polyethylene terephthalate (PET) film and $50-\mu$ m-thick release film with a thickness of 50 μ m, and then UV-cured with a wavelength of 365 nm with several UV intensity levels and irradiation times using our customized UV LED curing device (OnUV, Republic of Korea). The photo initiators absorb UV light energy, and became radical species by photocleavage reaction. Initiated radical species attack the monomers, crosslinking agent, and crosslinking group in pre-polymers, inducing free radical polymerization resulting crosslinking and curing.

2.4. Gel permeation chromatography

The molecular weights of acrylic PSA were measured using a YL9100 gel permeation chromatography (GPC) system (Young Lin Instruments, Republic of Korea) equipped with a YL9170 refractive index detector. GPC columns were eluted with tetrahydrofuran at 35 °C and a flow rate of 1 mL/min. Sample was diluted with tetrahydrofuran to 0.5 per cent concentration by weight. The number-average (Mn) and weight-average (Mw) molecular weights were calculated using a calibration curve from polystyrene standards as 630,000 and 1,477,000, respectively, and the polydispersity index (Mw/Mn) thus equaled 2.34.

2.5. Differential scanning calorimetry

Differential scanning calorimetry (DSC) analysis was performed using a DSC Q200 (TA Instruments, USA) apparatus with a refrigerator cooling system (RCS 90) to determine the glass transition temperature (Tg) of acrylic PSA. Samples were loaded in Al pans with 50.0-mL/min high-purity N₂ used as the purge gas. The sample was scanned from -60 to 140 °C at a heating rate of 3 °C/min, and Tg was determined as -33 °C.

2.6. Gel fraction

Gel fraction was measured to determine the degree of acrylic PSA crosslinking. The release films with coated PSA were cut into pieces. After the release films were removed, each piece was enveloped by a piece of stainless-steel sieve with an opening size of 200 mesh. The sieves were soaked in ethyl acetate and shaken at 200 rpm for 24 h. After filtering, the sieves were dried at 80 °C for 24 h and the gel fraction was calculated by

Gel fraction(%) = $(W_1 / W_0) \times 100(\%)$

where W_0 and W_1 are the sample weights before and after the filtering in ethyl acetate, respectively.



Fig. 1 – Effects of UV exposure time on the gel fraction of acrylic PSA samples for UV exposure intensities from Level 10 to Level 99. The lines are drawn for easier figure reading.

2.7. Fourier transform infrared spectroscopy

FTIR analysis was carried out to determine the UV LED curing conversion of acrylic PSA. The spectra were obtained using an FT/IR-6100 (JASCO, Japan) equipped with an attenuated total reflectance (ATR) accessory composed of a diamond crystal. Spectra were collected from 4000 to 400 cm⁻¹ 32 times at a resolution of 4 cm⁻¹. All spectra were corrected through CO₂ reduction, H₂O reduction, noise elimination, and baseline correction. The conversion of acrylic PSA was calculated by

Conversion (%) =
$$\frac{(A_{810})_0/(A_{1720})_0 - (A_{810})/(A_{1720})}{(A_{810})_0/(A_{1720})_0} \times 100(\%)$$

where $(A_{810})_0$ and (A_{810}) are the intensity for 810 cm⁻¹ (C=C bond) at the initial time and after curing, respectively, and $(A_{1720})_0$ and (A_{1720}) are the intensity for 1720 cm⁻¹ (C=O bond) at the initial time and after curing, respectively.

2.8. Transmittance

Visible-light transmittance was measured to confirm the optical clarity of acrylic PSA. Spectra were collected from 800 to 400 nm using a Cary 100 UV—vis spectrometer (Agilent Technologies, USA). Transparent 75-µm-thick PET film was used as a reference for the samples. The PSA sample was measured while attached to the PET film.

2.9. Peel strength testing

Peel strength was measured using a texture analyzer TA.XT-Plus (Micro Stable Systems, UK) according to ASTM D3330. The PET films with coated PSA were cut into 25-mm-width samples. Each specimen was pressed onto a stainless-steel substrate (type 304) by two passes of a 2-kg rubber roller and left at 30 °C for 24 h. The peel strength was measured at a 180° angle with a crosshead speed of 300 mm/min and obtained as the average strength between 5 and 8 s.

2.10. Lap shear strength testing

Lap shear strength testing was conducted to measure the shear strain using an AllroundLine Z010 universal testing machine (Zwick, Germany). Each specimen was pressed onto 75- μ m-thick PET film by two passes of a 2-kg rubber roller with an adhesion area of 25 \times 25 mm². The tests were performed at a crosshead rate of 1 mm/s. The shear strain was calculated by

Shear strain (%)
$$= \frac{\Delta L}{t} \times 100$$

where ΔL is the moving distance, and t is the thickness of the PSA film.

2.11. Strain recovery testing

Strain recovery testing was conducted using a DMA Q800 (TA Instruments, USA) equipped with a tension clamp under the dynamic mechanical analysis (DMA) stress relaxation mode. Each specimen consisted of ~50- μ m acrylic PSA film between two polycarbonate substrates with an adhesion area of 20 \times 6 mm². The specimen was stabilized at 25 °C for 5 min under a



Fig. 2 – Effects of UV exposure time on the gel fraction of acrylic PSA samples for UV exposure intensities of Level 10, 15, 20, and 30. The lines are drawn for easier figure reading.

stress of 0.001 N, and then 300% shear strain was applied for 10 min followed by a recovery under 0.001 N for 5 min. The strain recovery was calculated by

Strain recovery (%) = $\frac{S_i - S_f}{S_i}$

where S_i and S_f are the initial strain at the beginning of recovery and the final strain after recovery for 5 min, respectively.

3. Results and discussion

3.1. Gel fraction and FTIR conversion

Gel fraction was physically measured using the weight ratio of insoluble polymers in terms of degree of crosslinking in the cured acrylic PSAs. The acrylic PSA gel fraction was presented using UV exposure times from 0 to 10 s and UV exposure intensities from Level 10 (97 mW/cm²) to Level 99 (1,252 mW/ cm²), as shown in Fig. 1. With increasing UV exposure time, gel fraction was gradually observed to increase. The gel fraction values for Level 10, 20, and 30 begin to increase at 5, 4, and 3 s, respectively. This indicates that higher UV exposure intensity induces a rapid onset of PSA crosslinking. The gel fraction for each UV exposure intensity reaches the saturation point; this point was decreased for a maximum of 57% (Level 10 and Level 20) to 20~30% (Lever 50 and higher). However, the PSA samples appeared to be fully cured (shaped in cured film, reduced tack, no uncured resin flow) even with low gel fraction levels. In UV metal halide curing, high UV exposure intensity is generally regarded as the occurrence of more crosslinking. In previous studies, UV exposure for PSA curing was usually controlled by only the UV exposure time with a fixed UV exposure intensity using UV mercury [11,22-24] or UV metal halide [12] curing systems. In contrast, UV LED curing exhibits an entirely different behavioral tendency of the gel fraction as a function

of UV exposure intensity. At Level 10, gel fraction continued to increase under a UV exposure time of 10 s. The low intensity however, necessitated a 20 s exposure period to reliably measure the gel fraction to completion as shown in Fig. S3. Different UV exposure intensity levels resulted in different gel fraction values even at the same level o UV exposure also shown in Fig. S3. Therefore, both UV exposure intensity and time are the crucial factors in UV LED curing. Four types of UV exposure intensity (Levels 10, 15, 20, and 30) were chosen for the study because their gel fractions showed relatively large and meaningful differences, as shown in Fig. 2 and Fig. S4.

The conversion of acrylic PSAs is shown in Fig. 3 and Fig. S5. The conversion of C=C bonds increases as the UV exposure time increases [11]. Level 10 exhibited a slower conversion to the more rapid conversion rate of Level 30. The bond conversions reached close to totality (almost 100%) in comparison with corresponding gel fraction values. This behavior is attributed to the difference in the measurements between gel fraction and conversion. A nearly 100% conversion indicates that C=C bonds in acrylic PSAs were fully cured by UV LED exposure. Thus, the low saturation point of gel fraction under high UV exposure intensity could be attributed to the simple difference in the curing mechanism, This could be reasoned by the simultaneous random conversion of monomers under intense UV LED light exposure during the initial phase of the reaction resulting in crosslinked polymers comprising an admixture of smaller and more-condensed clusters.

3.2. Transmittance

The transmittance of acrylic PSAs is one of the most important properties for display applications. Acrylic PSAs are generally referred to as optically clear adhesives and this specific usage has much relevance to its importance of optical clarity [9,11,22,23]. Several curing conditions were chosen including low and high UV exposures for each UV exposure



Fig. 3 – Effects of UV exposure time on the monomer conversion of acrylic PSA samples for UV exposure intensity of Level 10, 15, 20, and 30. The lines are drawn for easier figure reading.

level of intensity. The spectra in the visible light region are presented in Fig. 4. All spectra are nearly the same and exhibit above 100% transmittance, which means that the cured acrylic PSAs are similar in terms of optical clarity when fully cured and have better transparency compared to the reference neat 75- μ m thick PET film. These results satisfy the optically clear criterion [9], indicating that the acrylic PSAs are suitable for display applications.

3.3. Adhesion performance and elongation property

The 180° peel strength testing of acrylic PSAs was conducted on testing specimens prepared according to UV exposure time after film formation using the gel fraction results. Peel strength is an indicator of the PSA adhesion and bond performance [11]. The initial peel strength at low UV exposure is almost zero even after film formation, but the strength sharply increases as the degree of UV exposure time increases as shown in Fig. 5. With UV LED curing, the acrylic PSAs exhibit proper adhesion performance and good wettability to the substrates. The lap shear strength testing specimens were also prepared after UV exposure time in the same way as those for the peel strength testing. The lap shear strength values are initially minimal because of the lower degree of curing under low UV exposure but increase as the UV exposure time increases. Higher UV exposure intensity greatly induces lap shear strength as shown in Fig. 6. The tendency of the lap shear strength is in accordance with that of the peel strength as a function of UV exposure. UV LED curing with sufficient UV exposure leads to the strong cohesion of acrylic PSAs, resulting in improved peel strength and lap shear strength.

The shear strain of acrylic PSAs is shown in Fig. 7. The shear strain initially plunges and gradually increases at a certain point as the UV exposure time increases. Minimally cured acrylic PSAs show relatively high shear strain when cured using UV LED. The initial reduction could be attributed to the decline in PSA resin flowability due to UV curing and the



Fig. 4 – Visible-light transmittance of acrylic PSA samples cured at UV exposure intensities of Level 15, 20, and 30.



Fig. 5 – Effects of UV exposure time on the 180° peel strength of acrylic PSA samples cured at UV exposure intensities of Level 10, 15, 20, and 30. The lines are drawn for easier figure reading.

subsequent gradual increase can be assigned to the formation of elastic crosslinking networks. Therefore, the shear strain of the acrylic PSAs can be manipulated by varying elongation properties from broad (~24,000%) to low (~5,000%) elongation achieved by modulating the UV exposure intensity and time period under UV LED curing. Shear strain is an important parameter for flexible display applications in terms of PSA flexibility. Under UV metal halide lamp conditions, low shear strain (%) is observed at low UV exposure and a gradual increase in shear strain values correlates well with increasing UV radiation [12]. In contrast, a decrease in the strain properties of PSA was noticeable with increasing UV energy. The unexpected difference in behavior likely originates from variation in the energy intensity of emitted radiation from both sources with dramatic effects on the crosslinking mechanism alerting PSA cohesion, stiffness and storage



Fig. 6 – Effects of UV exposure time on the lap shear strength of acrylic PSA samples cured at UV exposure intensities of Level 10, 15, 20, and 30. The lines are drawn for easier figure reading.



Fig. 7 – Effects of UV exposure time on the shear strain of acrylic PSA samples cured at UV exposure intensities of Level 10, 15, 20, and 30. The lines are drawn for clarity.

modulus of cured PSA samples under investigation. This result confirms that the properties of acrylic PSAs can be affected by the UV curing method.

Meanwhile, as observed for the gel fraction results, changes in UV exposure intensity cause different peel strength, lap shear strength, and shear strain values even at the same level of UV exposure as shown in Fig. S6, Fig. S7, and Fig. S8, respectively. This finding asserts that both UV exposure intensity and time should be duly considered in UV LED curing process.

To summarize, the gel fraction data improves more slowly and saturates at lower values as the UV light level increases as confirmed by Figs. S3, S4 and S5. Samples for Level 10 reach 80% and Level 30 are around 40% of the gel fraction value. As shown in Fig. S5, during the initial period of UV light irradiation, samples corresponding to Level 20 and 30 showed faster conversion rates than for Level 10 and 15. The conversion rate however is reversed after reaching 1 J. It is interesting that the lower the gel fraction continues to be observed at the same irradiation exposure when the UV energy is intensified. The probable cause may result from an initial rapid reaction from irradiation exposure at high UV intensity levels potentially causing the formation of clusters. Regional proximity effects may favor rapid cross-linking events from the UV light source dismissing the formation the usual universally cross-linked semi-IPN structure. When irradiated with a high amount of light, random polymerization occurs sporadically and abruptly due to rapid cleavage of the initiator. We assert that the assembly and existence of the crosslinked PSA clusters arising from this phenomena lower the overall crosslinking density and rate of conversion. This rationality explains low performance in cohesion and adhesion performance when the light intensity is high as shown in Fig. S5, S6 and S7. This



Fig. 8 – Strain change during stress relaxation for acrylic PSA samples cured at UV exposure intensities of Level (a) 10, (b) 15, (c) 20, and (d) 30.



Fig. 9 – Effects of UV exposure time on the strain recovery of acrylic PSA samples cured at UV exposure intensity of Level 10, 15, 20, and 30. The lines are drawn for clarity.

curing mechanism is also shown by the peel strength and lap shear data in Fig. S6 and S7. Both peel strength and lap shear strength show a J-shaped sharp improvement curve before the conversion saturation point at each UV light level followed by a relaxed improvement curve for peel strength and saturation curve for lap shear strength after the conversion saturation point. It is thought that local crosslinking which is not detected by FT-IR proceeds even above the conversion saturation point causing a slight improvement in physical properties. We attribute this to property changes as exposure energy increases despite plateauing of conversion events as it approaches saturation. Hence, a small amount of monomer around the polymer still participates in the reaction making the length of the polymer longer and consequently changing macro properties.

3.4. Recovery property

The strain recovery testing specimens were also prepared according to UV exposure time as mentioned previously. The strain recovery was measured to identify recovery properties after 10 min of heavy-load strain. The change in shear strain is plotted in Fig. 8 by the UV exposure time during stress relaxation testing from Level 10 to Level 30. As the UV exposure time increases, the residual strain gradually decreases for acrylic PSAs cured at Level 10 and Level 20 in Fig. 8(a) and 8(b), respectively. This result is attributed to an increased degree of crosslinking during UV curing which can be explained by gel fraction and conversion. In the cases of Levels 20 and 30 in Fig. 8(c) and 8(d), respectively, the residual strain is almost below 10%, indicating that UV curing with high UV exposure intensity has the advantage of removing the residual strain in a quick curing system with less UV exposure time.

The strain recovery was calculated as the strain difference during the 5-min natural recovery process from the straintime curves, as summarized in Fig. 9 and Fig. S9. Strain recovery is also an important parameter for flexible display applications in terms of PSA flexibility [13,31,32]. Compared to UV metal halide curing [12], most UV LED curing conditions exhibit good strain recovery properties above 90%, except for the low-cured Level 10 because of the low UV exposure. Using UV LED curing, quick and precise curing and high recovery of acrylic PSAs are possible for specific applications.

4. Conclusions

Optically clear acrylic PSAs with good transparency were synthesized and prepared by UV LED curing for flexible display applications. The gel fraction and FTIR conversion of C=C bonds were examined and confirmed the UV LED curing behavior. The adhesion performance was also measured using peel strength and lap shear strength testing. The UV exposure can be controlled by two variables namely UV exposure intensity and UV exposure time. The combination of two variables in UV LED curing leads to different properties of acrylic PSAs. From the results of lap shear strength testing and strain recovery testing, acrylic PSAs cured by UV LED exposure can be optimized with both high shear strain and high strain recovery. In the previous study, the optimization of two properties proved difficult because the shear strain increased in proportion to UV exposure, but the obtained strain recovery was relatively low under UV metal halide curing. The curing condition at Level 30 (385 mW/cm²) and 8-s exposure exhibits the highest shear strain (23,469%) and high strain recovery above 90% (94.0%) and demonstrate improved properties compared to UV metal halide curing. This condition also exhibits a saturated gel fraction and saturated complete conversion which signifies that the acrylic PSA was fully cured by UV LED exposure. Among the specimens in this study, the curing conditions applied to acrylic PSA is considered as the optimal for flexible display applications. Furthermore, acrylic PSAs cured by UV LED exposure have clear advantages that favor the removal of thermal and controllability issues as existing barriers to its performance but also provide an enhancement in flexibility properties. We conclude from the improvements in the properties of UV LED that curing is a suitable replacement of UV metal halide curing. However, further research on other monomer formulations is required for a deeper understanding and more efficient utilization of UV LED curing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jmrt.2020.12.034.

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