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Preparation of acrylic pressure-sensitive adhesives by UV/UV step curing as a way of lifting the limitations of conventional dual curing techniques



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ABSTRACT

Pressure-sensitive adhesives (PSAs) and UV-curable acrylic systems are widely utilized in different industries, with particular attention currently directed at their use in the production of re-useable modules for smart devices. Herein, we developed a two-step UV/UV curing technique to overcome the limitations of dual curing methods conventionally used for PSA preparation and characterized the thus obtained acrylic adhesives by a range of techniques to determine the uncured marginal portion of the pre-polymer, viscoelastic properties, and adhesive properties (peel and tack). Insufficient primary curing resulted in marked physical property deterioration, while the expression of secondary curing was weak when excessive irradiation energy was supplied. Thus, it was concluded that the physical properties of acrylic PSAs could be controlled by adjustment of primary curing energy.

1. Introduction

Pressure-sensitive adhesives (PSAs) and UV-curable acrylic resins have found diverse applications in present-day industrial processes [1–3]. Generally, the properties of acrylic PSAs can be controlled using both mechanical and chemical methods, e.g., by adjusting the amount/ type of blended tackifiers, the extent of main chain cross-linking [4], or type of cross-linking system [5].

PSAs produced by irradiation of UV-curable systems with UV light in the presence of a suitable photoinitiator, which induces radical photopolymerization and promotes the monomer to oligomer conversion, exhibit numerous advantages (e.g., solvent-free nature and low content of volatile organic compounds) that are important in industries such as coating, painting, and adhesive production [6–8].

The formation of post-adhesion defects during smart device assembly often results in the replacement of the whole affected module. However, such modules are rather expensive, which necessitates the development of alternative strategies such as those based on step curing. Previously, stepwise curing has been implemented as UV/ thermal dual-curing [9], which, however, can result in thermal damage of the module and is therefore of limited applicability to smart device fabrication. In addition, step curing can allow one to choose the curing time between primary and secondary curing. PSA has relatively high softness before the secondary curing, and hence increase rework properties. In addition, in the event of a process error or human error, rework can be performed before secondary curing. After the assembly is complete, the worker can proceed with secondary curing to improve the adhesion properties of PSA. For this reason, there is a problem in applying to smart devices. To solve this problem, we chose UV/UV step curing.

To reduce the uncured marginal portion of the pre-polymer used in step curing, acrylic PSAs are commonly formulated to contain acrylic acid (3–10 wt%) and a soft acrylate component (90–97 wt%) with chains bearing different functional groups and having various lengths [10]. Herein, we show that the problem of thermal damage can be circumvented, adopting a UV/UV step curing approach and polymerizing solvent-free. When MMA is used as the additive, crosslink density is increased [11].

It is possible to observe a phenomenon by which the reaction becomes slower when MMA is added, especially due to UV polymerization. Because of steric effects and the stability of the radical, methacrylates participate slowly in the polymerization [12,13]. In addition,

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Fig. 1. Schematic diagram for UV/UV step curing.

UV/UV step curing was implemented using black light (BL; long-time curing using a low-pressure light source) and a UV conveyor belt (fast curing using a high-pressure light source). Fig. 1 shows a schematic for stepwise UV/UV step curing; (a) no reaction occurs in the monomer state, (b) reaction proceeds partially in the pre-polymer state, (c) partial reaction occurs due to primary curing, (d) secondary curing proceeds and the reaction is complete. UV curing behavior was evaluated using photo-differential scanning calorimetry (photo-DSC) to measure the reaction rate and calculate conversion, while viscoelastic properties were probed by dynamic mechanical analysis (DMA) in film tension mode. An extensive network structure was formed even for low concentration of primary curing.

2. Experimental

2.1. Materials

All reagents were used without further purification. 2-Ethylhexyl acrylate (2-EHA, 99.0%), acrylic acid (AA, cohesion enhancer, 99.0%), 2-hydroxyethyl acrylate (2-HEA, cohesion enhancer, 99%), and methyl methacrylate (MMA, delayed reaction, 99.0%) were sourced from Samchun Pure Chemical (Republic of Korea), and isobornyl acrylate (IBA, 99%) was sourced from Sigma Aldrich (USA). Hydroxydimethyl acetophenone (HA, Micure HP-8, Miwon Specialty Chemical, Republic of Korea) used as a photoinitiator featured two absorption ranges (at 265–280 and 320–335 nm). Table 1 shows the compositions of the acrylic PSAs.

Table 1

Compositions	of	the	acry	vlic	PSA	s
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	Composition (%)	
Reactive monomer	monomer 2-EHA (2-ethylhexyl acrylate)	
	AA (Acrylic acid)	3
	IBA (Isobornyl acrylate)	19
	MMA (Methyl methacrylate)	4
	2-HEA (2-hydroxyethyl acrylate)	10
Photo initiator	Hydroxydimethyl acetophenone	0.3

2.2. Pre-polymer synthesis

The pre-polymer was synthesized from a mixture of 2-EHA (64 wt %), AA (3 wt%), IBA (19 wt%), MMA (4 wt%), 2-HEA (10 wt%), and HA (0.3 wt%) by bulk radical polymerization, which was performed in a 500-mL four-necked flask equipped with a mechanical stirrer, N₂ inlet, thermometer, and a light-emitting diode UV lamp. The polymerization was carried out on a 200 g scale. The temperature and stirring speed were maintained at 296 K and 100 rpm, respectively. After 30-min N₂ purging upon constant stirring, the temperature was increased by 15 K, and the monomer mixture was exposed to UV light (20 mW/cm², main wavelength: 365 nm) upon continued N₂ purging [14]. After increasing the temperature, air blowing was carried out for 30 min.

2.3. Adhesive film preparation

UV-curable syrups were prepared by blending the pre-polymer with ethoxylated trimethylolpropane triacrylate (EO6TMPTA, Sigma-Aldrich), commonly used a d multi-functional acrylates (Fig. 2(a)) [15].

EO6TMPTA was used to evaluate the delay in primary UV curing and to make uncured marginal portion of the pre-polymer, and lauryl acrylate (LA; Fig. 2(b)) was used to delay the UV curing reaction by reaction with the chain transfer agent [16]. All samples contained 2 phr LA.

2.4. Step curing

Corona-treated polyethylene terephthalate films (SKC Co. Ltd., Republic of Korea) were coated with a 100- μ m-thick syrup layer and passed through a BL curing machine equipped with a low-pressure mercury UV lamp (20 mW, main wavelength = 365 nm) for primary curing. Secondary curing was performed by passing the primary-cured film through a conveyor-type UV-curing machine equipped with medium-pressure mercury UV lamps (154 mW/cm², main wavelength = 365 nm). The UV irradiation dose was fixed at 3000 mJ/cm². Stepwise-curing follows the following Fig. 3.

2.5. Photo-DSC

The UV-curing behavior of adhesives was evaluated by photo-DSC (DSC Q200, TA Instruments, USA) using a spot-cure light source



Fig. 2. Structures of (a) EO6TMPTA (n + m + o = 6) and (b) lauryl acrylate (LA).



Fig. 3. UV/UV step curing process for the PSAs.

(Omnicure-s2000, Excelitas, Waltham, MA, USA) comprising a 100W mercury vapor lamp. Light intensity was determined by placing an empty open aluminum DSC pan on the sample cell. The UV light level of the UV accessory equipped with a 90% UV filter was determined as 10%. The sample weight was 1–2 mg. All measurements were performed at 25 °C [17].

2.6. Adhesion properties

2.6.1. Peel strength

The 180° peel strength was measured based on ASTM D3330 (TA-XT2i texture analyzer, Micro Stable Systems, UK) at a crosshead speed of 300 mm/min at 293 K for bonded specimens (width = 25 mm) that were left to stand at room temperature for 24 h. The debonding force was recorded for five different runs, and the average force was reported as N/25 mm. SUS 304 was used as the substrate. Curing progresses in the film at the time of primary curing when measuring the peel strength, and curing proceeds after adhering to SUS304 at the time of secondary curing. This is to confirm changes in physical properties in actual applications.

2.6.2. Probe tack

Probe tack was measured at 293 K using the abovementioned texture analyzer and a 5-mm-diameter stainless steel cylinder probe. The cylinder approached the adhesive sample at a speed of 0.5 mm/s, stayed in contact with the PSA surface for 1 s at a constant pressure of 100 g/cm^2 , and was then debonded at a speed of 0.5 mm/s. Probe tack was measured as the maximum debonding force (ASTM D3330) [18].

2.7. Viscoelastic properties

The temperature dependence of dynamic storage modulus and tan δ were evaluated by a dynamic mechanical analyzer (DMA Q800, TA Instruments, USA) in film tension mode for specimens with length, width, and thickness of approximately 11–13, 12, and 1 mm, respectively. Testing (strain rate = 0.1%, frequency = 1 Hz, heating rate = 5 K/min) was conducted between 240 and 370 K.

2.8. Gel fraction

The contents of insoluble compounds such as cross-linked or network polymers were determined by measurements of the (solvent solubility parameter-dependent) gel fraction [19] using toluene as the solvent. Cross-linked adhesives were soaked in toluene for 24 h at 323 K, and the insoluble residue was removed by filtration and dried to constant weight at 323 K [20]. The gel fraction was calculated as gel fraction (%) = $(W_1/W_0) \times 100$, where W_0 is the weight of the original sample, and W_1 is the weight of the dried insoluble residue.

3. Results and discussion

3.1. Curing behavior determined by photo-DSC

The C=C double bond is broken by radical polymerization of the acrylic resin increases [21]. Fig. 4 (a) shows the results of photo-DSC analysis of primary-cured samples, revealing clear peaks for samples with exposure energies of 2.5–3.5 J. As seen in Fig. 4 (b), the trend for 4-5 J is the same as the heat flow that occurs in the overall energy during secondary curing. However, weak peaks were observed for samples with exposure energies of 4-5 J, i.e., curing was almost complete in these samples. Fig. 4 (b) shows the real-time heat flows pertaining to the secondary curing step, revealing that only weak peaks were observed for all exposure energies.

Notably, a similar phenomenon was observed in Fig. 5, which shows areas under the exothermic curves of primary/secondary curing The area under the photo-DSC curve reflects the extent to which the polymerization reaction takes place during secondary curing.

Fig. 5(a) and (b) show the area under the exothermic curve. The observation of a plateau indicates completion of the reaction. Comparison with Fig. 4 shows that the PSAs subjected to primary curing at 4J–5J are similar to those subjected to secondary curing. Such a result can be expected that if secondary curing proceeds at 4J–5J, and there is no margin for secondary curing progress. It is an important factor that margin is left to proceed with step curing It can be confirmed from Fig. 5 (c) that the primary curing energy is 4–5J and that the flat area is reached within 10 s, unlike the case of other PSAs. Such results can also be confirmed from Fig. 5 (d), which reveals that all the PSAs reach a flat area within a few seconds. These results can confirm the margin that



Fig. 4. Real-time heat flows after (a) primary and (b) secondary curing steps.



Fig. 5. Area under the Exothermic Curve (a) Primary Curing, (b) Secondary Curing and. Area under the Exothermic Curve up to 30 s (c) Primary Curing, (d) Secondary Curing.



Fig. 6. Conversion profiles: (a) primary curing step and (b) secondary curing steps.



Fig. 7. Schematic effect of primary curing energy variation on cross-linking density.



Fig. 8. Gel fractions of cross-linked acrylic PSAs prepared at different exposure energies.

can facilitate step curing. On the other hand, in Fig. 5 (c), the PSAs with a primary curing energy of 2.5 J tend to be similar to those cured at 4-5 J. However, the overall trend implies the progress of the reaction. This phenomenon will be explained again from the back.

Photo-DSC measurements provide kinetic data that can be used to directly determine the final conversion and polymerization extent for a given formulation, while heat flow data reflect the full curing reaction of the sample [22,23]. For curing induced by photo-DSC, heat flow was assumed to be proportional to the measured conversion rate. This assumption would be valid for substances that do not lose solvents or other volatile components via evaporation or feature large changes in heat capacity caused by phase changes, etc.

Therefore, the conversion or polymerization rate $(d\alpha/dt)$ can be defined as [24–26].

$$d\alpha/dt = (dH/dt)/\Delta H_{\text{total}},\tag{1}$$

where dH/dt is the measured heat flow, and ΔH_{total} is the total exothermic heat of the reaction. Integration of Eq. (1) allows conversion at time t (α_t) to be calculated as

$$\alpha_t = \Delta H_t / \Delta H_{\text{total}},\tag{2}$$

where ΔH_t is the cumulative heat of reaction up to curing time t [27].

Fig. 6 (a) shows the conversion profiles for the primary curing step, calculated using the above method, revealing that at identical t, conversions observed the reaction possibility of primary curing. Fig. 6 (b) shows the conversion plots for the secondary curing step, revealing that the conversion steadily increased with time and that no reaction proceeded during secondary curing. As is clear from the above equation, if the calculated value is a straight line and the result is calculated, it can be explained that there is no margin that can be reacted. Therefore, in the primary curing stage, under 5 J irradiation, it can be confirmed that there is no margin to be reacted, and the response is finished even with 4J secondary curing. In addition, it is possible to confirm from Fig. 6 (b) that the reaction after primary curing is delayed.

Similar results were derived as for PSAs with a primary curing energy of Figs. 5 to 2.5 J before with no cure margin. This is expected to



Fig. 9. Temperature dependence of PSA storage modulus determined for different exposure energies results for (a) primary and (b) secondary curing.



Fig. 10. Temperature-dependent $\tan \delta$ of cross-linked acrylic PSAs prepared using different exposure energies: (a) primary and (b) secondary curing. (c) Temperatures of maximum $\tan \delta$ extracted from parts (a) and (b).



Fig. 11. Peel strength and probe tack as functions of UV exposure energy.

be delayed with low energy. These results can be confirmed by the fact that the PSA subjected to a primary curing energy of 2.5 J also shows slow conversion, as seen in Fig. 6 (b). Therefore, it is expected that when using a UV source with low light intensity, some time would elapse before a sufficient level of cross-linking density is achieved. This is the same as Fig. 7. A reaction occurs in the UV source and cross-linking occurs in the bulked pre-polymer, but if low energy (2.5 J) is possessed, a sufficient number of oligomers do not react. Conversely, at high energy (5 J), a sufficient number of oligomers react to improve the crosslinking density, as can be seen by analogy with tan δ in Fig. 10.

3.2. Gel fraction measurements

Cross-linking density is generally measured indirectly, e.g., by calculating the fraction of soluble substances [28].

Fig. 8 shows that the gel content of PSAs was independent of primary curing exposure energy, e.g., a gel content of 90% was obtained even at the lowest energy of 2.5 J. Thus, primary curing afforded highly cross-linked structures. Although polymerization of methacrylate monomers in resin composites is known to afford highly cross-linked structures [29], the thus obtained resinous material is not fully polymerized but contains considerable quantities of pendant double bonds [30].

3.3. Viscoelastic properties

Fig. 9 (a) shows the temperature-dependent storage moduli of crosslinked acrylic PSAs after primary curing, revealing that low moduli were observed, since primary curing did not result in a marked molecular weight increase [31]. Similar behavior was observed for secondary curing (Fig. 9 (b)), with the lowest modulus measured for an exposure energy of 2.5 J. Fig. 10 shows the effect of temperature on $\tan\delta$ of cross-linked acrylic PSAs after primary/secondary curing and the temperatures of maximum tan δ peaks. For primary curing and a low exposure energy of 2.5 J, maximal tan δ was observed at a rather low temperature, while maxima corresponding to exposure energies of 3-5 J were observed at almost identical temperatures. Similar results were also obtained for secondary curing. Fig. 10(a and b) show the temperature of maximum $tan\delta$, which is known to correspond to the glass transition temperature (T_g) [32]. Primary curing was characterized by low exposure energy, which resulted in a slight T_g increase with progressing curing. Moreover, a similar increase was also observed for secondary curing. However, there is no difference between the primary curing exposure energies (4 J and 5 J). In other words, for exposure energies of 4 and 5 J, most of the crosslinking occurred at the primary curing stage.

3.4. Adhesion properties

The adhesion properties of acrylic PSAs can generally be quantified using three test methods, with the most widely used ones corresponding to peel strength and probe tack determination. Moreover, these properties are also greatly affected by the cross-linking mechanism [33]. Fig. 11 shows the results of peel strength and probe tack testing for resins prepared using different UV exposure energies. All adhesion properties were evaluated after primary curing and were rather poor when a low energy of 2.5 J was used. Moreover, although a slight increase of adhesiveness was observed after secondary curing, the large magnitude of the observed deviation implied that the distribution of molecular weight was not balanced. Moreover, peel result slightly increased at an energy of 3.5 J, and samples prepared using exposure energies of 4-5 J showed similar hardness in the cases of both primary and secondary curing. These results seem to imply that curing was almost complete at the primary stage. There was a slight difference between tack result and primary curing and secondary curing, and they showed almost similar values. It can see that curing steps is not affected by the case of probe tack.

4. Conclusions

Herein, we investigated the possibility of preparing acrylic PSAs by stepwise (primary and secondary) UV/UV curing. In doing so, we synthesized a range of PSAs by slow radical polymerization and studied the effect of primary curing conditions on physical properties, revealing that adhesion properties and modulus deteriorated when an overly low primary curing energy (2.5 J) was used, which was ascribed to the fact that no marked molecular weight increase could be achieved under these conditions. In addition, maximal hardness was at a primary curing energy of 4 J. Thus, the physical properties of acrylic PSAs could be controlled by adjustment of the primary curing energy. In addition, we confirmed that the occurrence of marginal portion of the pre-polymer was not a non-reaction monomer through the gel-fraction. However, this criterion might depend on the identities of the pre-polymer and cross-linking agent change, which will be addressed in our further work.

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