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# Preparation and Properties of New UV-curable Naphthyl Epoxy Acrylates

Hongbo Liu[1*], Feng Lin[1], Mingcai Chen[2], and Kai Xu[2]

(1) School of Applied Chemistry and Biological Technology, Shenzhen Polytechnic,
Shenzhen 518055, PR China
(2) Guangzhou Institute of Chemistry, Chinese Academy of Sciences,
Guangzhou 510650, PR China

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## ABSTRACT

New UV-curable di-functional naphthyl epoxy acrylates (DNEA) and multi-functional naphthyl epoxy acrylates (MNEA) were synthesized from naphthyl epoxy, acrylic acid, and hydroquinone as an inhibitor, and *N,N´*-dimethylbenzyl-amine (DMBA) as a catalyst. Different UV-curable liquid compositions were developed with naphthyl epoxy acrylates, different reactive diluents, active amine monomer and 1-hydroxycyclohexylphenyl ketone (Irgacure 184) as a photoinitiator. Thin films were prepared by curing these composite solutions using a high pressure mercury lamp. The change of double bond absorption band was identified by Fourier transform infrared spectra during the UV-curing process. Gel content, water absorption, the pendulum hardness, mechanical properties (static and dynamic) and thermal characterizations of the UV-cured films were investigated. The characteristic C=C absorption of the unsaturated acrylic groups at 1635 cm$^{-1}$ gradually disappeared with UV-cured time and was no longer detectable after irradiation for 10 s. The gel content and the pendulum hardness increased with increasing irradiation time, and these gradually turned to constant values with increasing the irradiation time as the degree of polymerization reached its saturation. With the increase of mono-functional reactive diluents butyl acrylate in the compositions, the gel content, the pendulum hardness and mechanical properties of UV-cured films decreased, except for the water absorption. It was found that the glass transition temperature and the storage modulus of MNEA compositions were higher than those of DNEA compositions by the dynamic mechanical measurements, and the glass transition temperature of MNEA compositions was 142°C. The thermogravimetric analysis of UV-cured films revealed that films prepared from naphthyl epoxy acrylates exhibited excellent thermal stability.

**Key Words:**

composites;
curing of polymers;
films;
photopolymerization;
thermogravimetric analysis.

## INTRODUCTION

Since its commercial introduction over 30 years ago, UV-curing has enjoyed steady growth in demand and usage and has become one of the fastest growing coating technology in the world [1,2]. UV-curing is known to be one of the most efficient methods for producing quasi-instant and highly cross-linked polymer materials. Due to its unique advantages of fast reaction rate, solvent-free formulation, and excellent physical properties, this technology has found a large number of industrial applications, such as fast drying varnishes and ink, optical fibres, electronic components and printing plates, and restoration materials [3-5].

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(*) To whom correspondence to be addressed.
E-mail: hbliu@mail.gic.ac.cn

In general, UV-curable systems are known to consist of reactive polymerizable oligomers, reactive diluents and photoinitiators [3,6]. Oligomers, providing the final cured films with the ultimate performance properties, commonly used for coatings, include epoxy acrylates [7,8], urethane acrylates [9-13], polyether or polyester acrylates [14], acrylic acrylates and unsaturated polyesters [15,16]. Reactive diluents are used to reduce the viscosity of the compositions to a workable consistency and also participate in the film formation by copolymerizing with the oligomers, when suitably initiated [17]. Photoinitiators for UV systems transform the energy from light into free radicals initiating the polymerization process. Additionally, non-reactive additives, such as pigments, adhesion promoters, wetting agents and so forth, are also introduced into compositions to meet specific application requirements. The compositions of the UV-cured formulations influence both the curing rate and the properties of the created polymer [18].

The performance properties of any UV coating are determined principally by the oligomers used in the formulation. The epoxy acrylates as oligomers are used extensively as protective coatings on wood, metal and plastics, because of their fast cure speed, high hardness, scratch and abrasion resistance, higher gloss, high solvent and chemical resistance and especially providing lower cost to a formulation.

In the previous work, we synthesized and studied a series of high performance naphthyl epoxy resins for the electronics industry. These were useful in circuit boards as matrix resins, because of their high glass transition temperatures [19-21]. Some researchers reported their works like preparation of bisphenol A epoxy acrylates or naphthyl epoxy, but there are few patents and papers reporting the preparation of the naphthyl epoxy acrylate used in UV-curable system. Therefore, in this work, original di-functional naphthyl epoxy acrylates and multi-functional naphthyl epoxy acrylates were synthesized by using high performance naphthyl epoxy as raw materials. The naphthyl epoxy acrylates, used as oligomers for UV-curing systems in a mixture with trimethylolpropane triacrylate (TMPTA), butyl acrylate (BA) or hexanediol diacrylate (HDDA), are discussed. The properties directly related to the polymer structure (e.g., gel content), as well as the mechanical, physical and thermal behaviours of the polymeric films obtained via the UV-curing technique were investigated.

## EXPERIMENTAL

### Materials

Di-functional naphthyl epoxy ($\overline{M}_n$ = 365) and multi-functional naphthyl epoxy ($\overline{M}_n$ = 814) were prepared from naphthol. More extensive descriptions of the synthesis and characterization were described elsewhere [19-21]. Acrylic acid, active amine monomer, hydroquinone and *N,N'*-dimethylbenzylamine (DMBA) were purchased from Guangzhou Chemical Regents Co. Hexanediol diacrylate (HDDA), butyl acrylate (BA) and trimethylolpropane triacrylate (TMPTA) were supplied by Tianjiao Paint Ltd. 1-Hydroxycyclohexylphenyl ketone (Irgacure 184, Aldrich Chemical) was used as a photoinitiator for UV-curing. All the materials in this study were used as received without further purification.

### Synthesis of Naphthyl Epoxy Acrylates

A 250-mL round-bottomed, three-necked flask equipped with a mechanical stirrer, condenser and thermometer was charged with naphthyl epoxy. While stirring, the naphthyl epoxy was heated to 100°C, followed by adding acrylic acid containing 0.05 wt% DMBA and 0.04 wt% hydroquinone based on the prepolymer. The reaction was conducted at 110°C until the acid number was less than 4 mg KOH/g. The standard alkali solution titration method was used to determine the acid content of the prepolymer. The idealized molecular structures of these are shown in Figure 1. The di-functional naphthyl epoxy acrylates and the multi-functional naphthyl epoxy acrylates are denoted as DNEA and MNEA, respectively.

### Preparation of UV-cured Film

The weight ratios of naphthyl epoxy acrylates (DNEA or MNEA), reactive diluents (HDDA, BA and TMPTA), photoinitiator (Irgacure 184) and active amine monomer (0.1 wt%) in the liquid compositions used in the reported investigations are given in

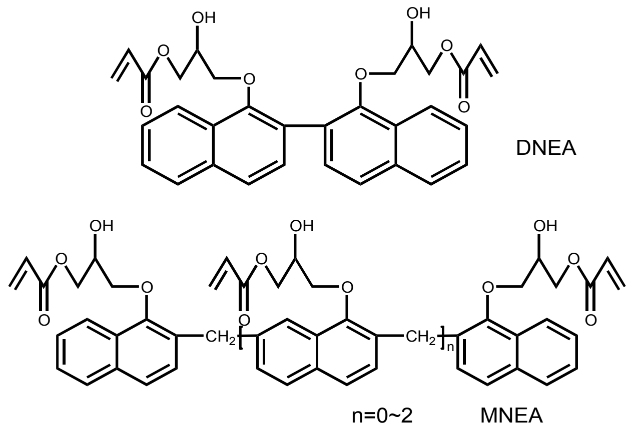

**Figure 1.** The idealized molecular structures of DNEA and MNEA.

Table 1. The different C=C densities were obtained using different percentages of diluents, so the variation regularity of the properties could be measured. These mixtures were prepared on a glass plate with a special frame as a support. The samples were UV-cured using an ultraviolet high pressure mercury lamp (1 kW, 80 W/cm, Huiyixin Electrical Light Source Technology Development Ltd., Beijing).

### Fourier Transform Infrared Spectra

The conversion of acrylic groups in UV-curable liquid formulation before and after UV-irradiation were measured with a Fourier transform infrared spectrometer (Analect RFX-65A) using a KBr pellet to compare the degree of cure.

### Gel Content

A known amount of the UV-cured films was placed in a Soxhlet apparatus and extracted with acetone for 6 h. The extracted film was then dried for 12 h at 70°C. The final weight of the extracted dry film, expressed as percentage of the initial weight, was assumed as the gel content (the degree of polymerization). Three tests were taken for each sample and average values were taken.

**Table 1.** The compositions of UV-curable liquid formulations.

| Chemical (%w/w) | A1 | A2 | A3 | B1 | B2 | B3 |
|---|---|---|---|---|---|---|
| DNEA | 60 | 60 | 60 | - | - | - |
| MNEA | - | - | - | 60 | 60 | 60 |
| TMPTA | 18 | 10 | - | 18 | 10 | - |
| HDDA | 19 | 15 | 19 | 19 | 15 | 19 |
| BA | - | 12 | 18 | - | 12 | 18 |
| Irgacure 184 | 3 | 3 | 3 | 3 | 3 | 3 |

### Water Absorption

The specimens of cured polymers were immersed in deionized water at 25±0.5°C for 24 h. The water absorption capacities of the cured sample were calculated as percentage of weight differences between the dry films and those soaked in water as follows:

$$\text{Water absorption (\%)} = \frac{(\omega - \omega_0)}{\omega_0} \times 100\%$$

where $\omega_0$ and $\omega$ are sample weights before and after immersion, respectively. Three runs were made for each sample and average values were taken.

### The Pendulum Hardness

The pendulum hardness of the UV-cured film was evaluated using a QBY pendulum apparatus made by the Tianjin Instrument Company. The time was recorded when the pendular angle was changed from 6° to 3°. The pendulum hardness of a cured sample was calculated as follows:

$$\text{Pendulum hardness} = \frac{T}{T_0}$$

where T and $T_0$ are the time recorded on the UV-cured film and the standard glass (GB1730-82). The films were applied to glass plates (95 × 88 × 5 mm) by a coater with a 100 μm gap. Three tests were taken for each sample and average values were taken.

### Mechanical Properties

The measurements of mechanical properties (relative elongation and breaking strength) of the cured films were performed directly with a CMT7503, a machine for measuring tensile properties, at strain rates of 20 mm/min. The test samples of the cured film were prepared as strips of 80 × 10 × 0.45 mm. Three runs were made for each sample and average values were taken.

### Dynamic Mechanical Measurement

The dynamic mechanical properties of UV-cured films were obtained at 5 Hz using a Netzsch DMA 242 with a heating rate of 2°C/min over a temperature range of -20°C to 200°C. Rectangular test specimens (0.6 × 4 × 30 mm) were prepared. One test was taken in tensile configuration for each sample.

### Thermal Properties

The thermogravimetric analysis (TGA) of UV-cured films was obtained by a Netzsch thermal analyzer at a heating rate of 10°C/min in a test range of 20-700°C under a nitrogen purge. The sample weights were about 8±2 mg. One test was taken for each sample.

## RESULTS AND DISCUSSION

The curing of the unsaturated acrylic liquid formulation was analyzed by FTIR. The characteristic C=C absorption of the unsaturated acrylic groups at 1635 cm$^{-1}$ was used to determine the extent of photopolymerization in this study. The acrylic resins were rapidly cross-linked by a free radical polymerization mechanism in the presence of an effective photoinitiator. Figure 2 shows a typical IR spectrum of the unsaturated acrylic liquid formulation before and after UV-irradiation for 2 s to 10 s. The unsaturated acrylic liquid formulation had a C=C absorption band at 1635 cm$^{-1}$ before UV-irradiation, but the band gradually disappeared with time and it was no longer detectable after irradiation after 10 s. The results in Figure 2 indicated that UV-curing was essentially completed after 10 s, and the UV-curable systems of naphthyl epoxy acrylates showed the same fast cure speed with that of bisphenol A epoxy acrylates. The oxygen inhibition of UV-curing process was resolved by addition of active amine monomer using high pressure mercury lamp.

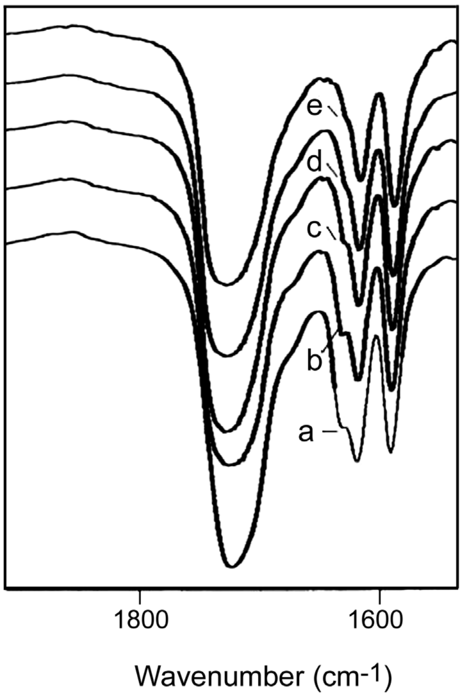

**Figure 2.** FTIR spectra of unsaturated acrylic liquid formulation before (a) and after UV-curing (b) 2 s, (c) 5 s, (d) 8 s, and (e) 10 s.

The extent of photopolymerization of the unsaturated acrylic liquid formulation, after exposure to UV, was also measured by Soxhlet extraction in terms of the gel content. Figure 3 shows the change in gel content as a function of irradiation time for the DNEA oligomers consisting of different reactive diluents. It was found that there were dramatic increases in gel content, up to 80.8% within 2 s for A1. Thereafter, the gel content of UV-cured films slowly increased by increasing the irradiation time to 10 s, and there were very small gains in gel contents with further irradiation time. The gel content decreased with increasing BA content in liquid

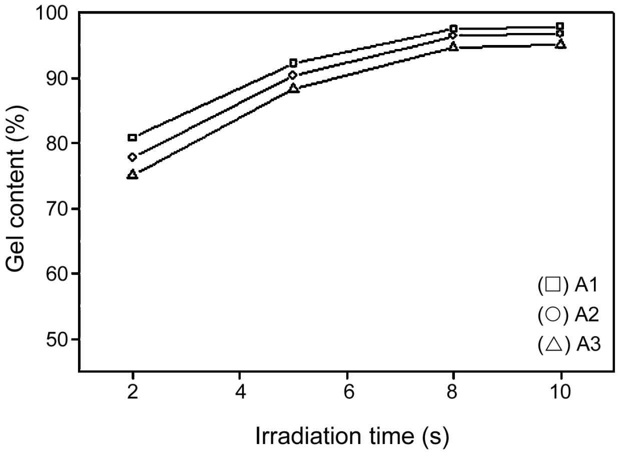

**Figure 3.** The gel contents of UV-cured films with different irradiation times (the relative average deviation ≤ 0.3%).

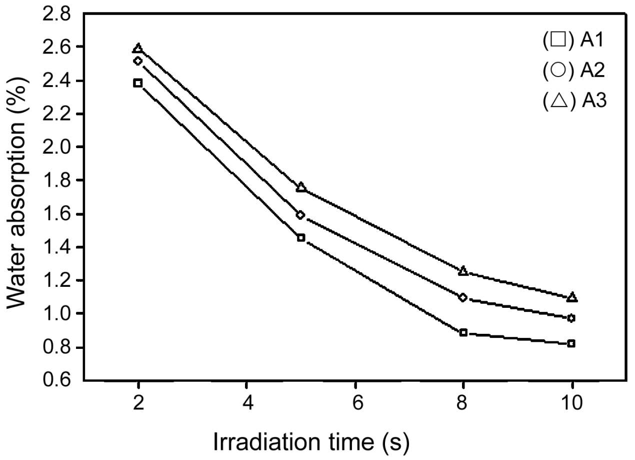

**Figure 4.** The water absorptions of UV-cured films with different irradiation times (the relative average deviation ≤ 0.6%).

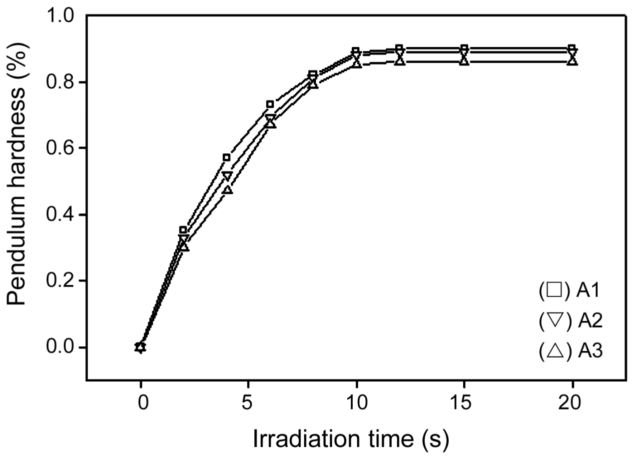

**Figure 5.** The pendulum hardness of the DNEA films with different reactive diluents (the relative average deviation ≤ 0.5%).

formulation. When the irradiation time was 10 s for A1, A2 and A3 liquid formulations, the gel contents were 97.8%, 96.7% and 95.1%, respectively. These results indicated very good cross-linking with various acrylic monomers present in these different compositions.

In contrast, with prolonged irradiation time, the water absorptions of UV-cured films decreased as shown in Figure 4. The water absorption varied between 2.3% and 2.6% for DNEA compositions when cured 2 s. The water absorption decreased to about 0.9% when irradiation time was prolonged to 10 s. The lowest water absorption value was obtained with formulation A1 which was followed by A2. The highest water absorption value of A3 was only 1.13%. When gel content reached at its highest level, namely, the structure of the UV-cured films was densest (the most compact): the films resisted most penetration of water molecules into films.

The pendulum hardness is an important test for UV-cured films, and it is related to the cross-linking network density and structure, and the conversion of acrylic groups in the molecular chain. Results of the pendulum hardness of the UV-cured films, prepared with different compositions versus irradiation time, are graphically shown in Figures 5 and 6. It can be seen that prolonging the irradiation time led to an increase of the pendulum hardness because more light energy causes higher conversion of acrylic groups. For instance, the pendulum hardness of UV-cured A1 film was 0.73, 0.82 and 0.90, when the irradiation time was 6 s, 8 s and 10 s, respectively. The highest pendulum hardness of all compositions was obtained for those cured 10 s, that is, the gel content of UV-cured films had reached its highest level. However, with the increase in mono-functional reactive diluents (BA) in DNEA compositions, the highest pendulum hardness of UV-cured film

**Table 2.** Properties of UV-cured films with different liquid compositions (the relative average deviation ≤ 0.6%).

| Formulation | Gel content (%) | Water absorption (%) | Pendulum hardness (%) | Young's modulus (%) | Breaking strength (%) | Relative elongation (%) |
|---|---|---|---|---|---|---|
| A1 | 97.8 | 0.82 | 0.90 | 918 | 34.3 | 3.8 |
| A2 | 96.7 | 0.97 | 0.89 | 848 | 32.2 | 4.5 |
| A3 | 95.1 | 1.09 | 0.86 | 792 | 30.0 | 5.2 |
| B1 | 98.3 | 0.79 | 0.94 | 957 | 35.9 | 3.5 |
| B2 | 97.2 | 0.91 | 0.91 | 910 | 33.7 | 4.3 |
| B3 | 96.1 | 0.99 | 0.88 | 841 | 31.5 | 5.0 |

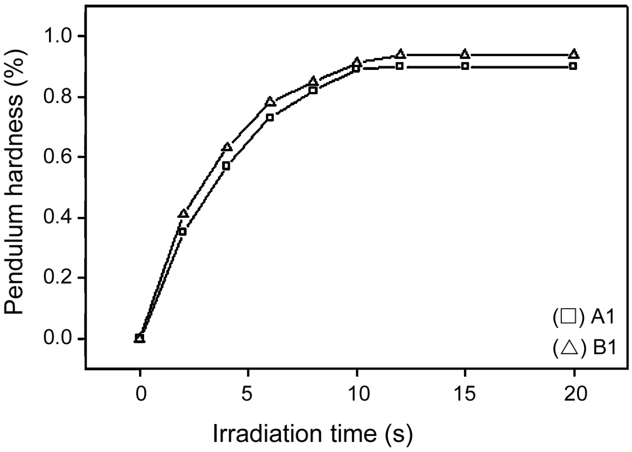

**Figure 6.** The pendulum hardness of the DNEA and MNEA films with the same reactive diluents (the relative average deviation ≤ 0.5%).

decreased as well (Figure 5). The highest pendulum hardness of UV-cured A1 film was 0.9, and that of A3 film was 0.85. With the same reactive diluents, the highest pendulum hardness of UV-cured film using DNEA as oligomer was lower than that of UV-cured film using MNEA as oligomer (Figure 6).

In these series of experiments, the results of investigations of the UV-cured films are presented in Table 2. The mechanical properties of UV-cured films were dependent on the different reactive diluents concentration in the same manner. The values of pendulum hardness, Young's modulus and breaking strength diminished with increasing mono-functional reactive diluent (BA) concentration, namely, with the decrease in the degree of polymerization. However, the relative elongation-at-break increased.

Compared with the properties of the UV-curable bisphenol A epoxy acrylates systems, the results of naphthyl epoxy acrylate systems indicated that the physical and mechanical properties of the DNEA systems had similar values to those of bisphenol A epoxy acrylates systems, but the properties of the MNEA systems were superior to the bisphenol A epoxy acrylate systems.

The dynamic mechanical properties of the UV-cured films for samples A2 and B2 are shown as a function of temperature in Figures 7 and 8. The peak position of tan δ, related to glass transition temperature ($T_g$), had the order B2>A2. As it is shown in Figure 7, the $T_g$ values of samples A2 and B2, estimated from the maximum peak of tan δ, were 131°C and 142°C, respectively. The E' value of sample B2 was also higher than that of sample A2. The breaking strength (Table 2), also showed the same trend as the $T_g$ behaviour. The higher mechanical strengths of samples prepared from MNEA may be related to their high $T_g$. In other measurements, the $T_g$ values of the bisphenol A acrylates systems only changed from 70°C to 100°C with different diluents. Due to the higher $T_g$, these naphthyl epoxy acrylates could be used as oligomers for UV-cured coatings for some special electronic products.

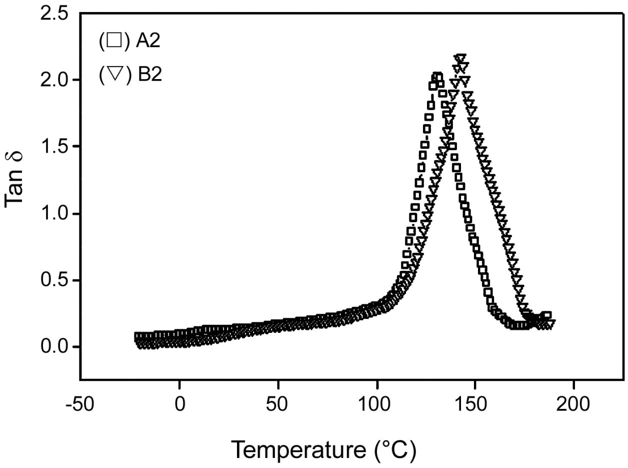

**Figure 7.** Tan δ of UV-cured films of samples A2 and B2.

It seems that the relationships described above may be interpreted as follows: cross-linking structure and content have an effect on the properties of

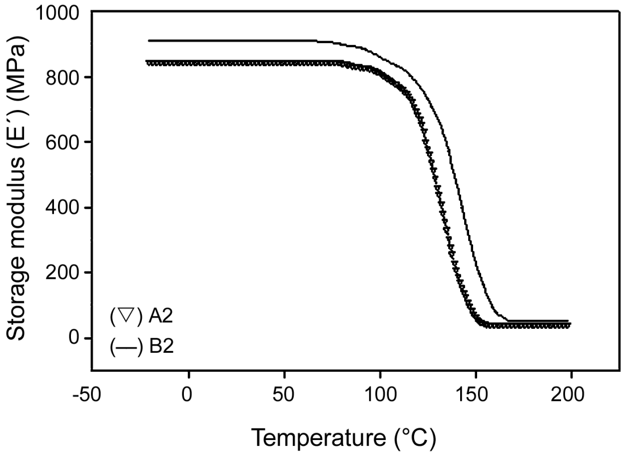

**Figure 8.** Storage modulus of UV-cured films of samples A2 and B2.

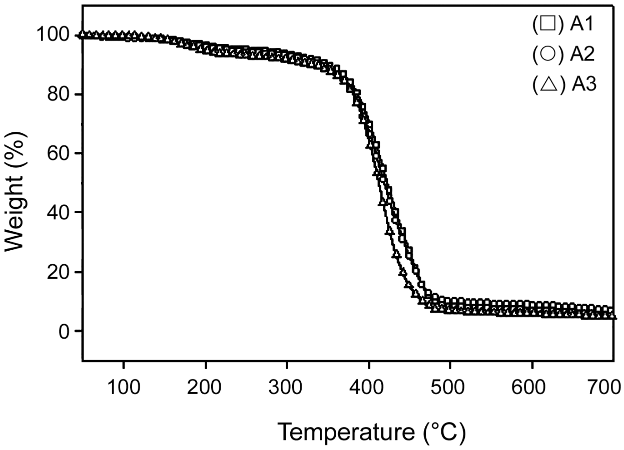

**Figure 9.** TGA of UV-cured films with DNEA as oligomers.

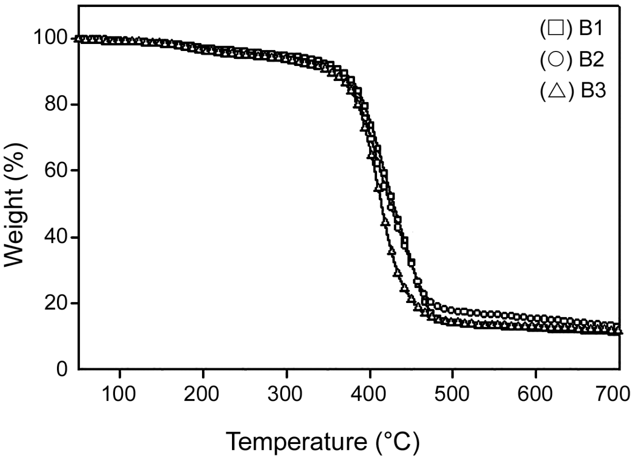

**Figure 10.** TGA of UV-cured films with MNEA as oligomers.

UV-cured polymeric films [22]. An increase in mono-functional reactive diluents (BA) content caused a decrease in the concentration of the double bonds and the cross-linking density in the UV-cured films, which led to a decrease in the gel content, the pendulum hardness and the breaking strength. Unpolymerized molecules in the network lowered the density of cross-links and acted as plasticizers, causing greater extension prior to break. Because there were 3-5 acrylic groups in the MNEA molecular structure and DNEA molecule had only 2 acrylic groups, the multi-functional MNEA formed a network structure more easily than the di-functional DNEA. Therefore, the gel content, the pendulum hardness, the glass transition temperature and the breaking strength of MNEA compositions were higher than those of DNEA compositions, with the same reactive diluents.

The thermogravimetric analyses (TGA) of UV-cured films are shown in Figures 9 and 10. The thermal decomposition temperatures of UV-cured films increased slightly with increasing degree of polymerization (gel content). All the thermal decomposition temperatures of UV-cured films were beyond 200°C when the naphthyl epoxy acrylates were mixed with various reactive diluents. The thermal decomposition temperature of sample B1 was up to 293.5°C. The results showed that the decomposition temperatures of the naphthyl epoxy acrylates systems were slightly higher than those of bisphenol A epoxy acrylates systems. In other words, the UV-cured films exhibited excellent thermal stability. The heating temperatures, to reach certain residues during the thermal decomposition process, and the final residues (heated to 700°C), are listed in Table 3.

**Table 3.** Thermogravimetric analysis: decomposition temperature at certain residues (%w/w).

| Sample | Residual weight percent | | | | | | Residue (%) |
|---|---|---|---|---|---|---|---|
| | 95% | 90% | 80% | 50% | 20% | 10% | |
| A1 | 241.1 | 351.5 | 386.7 | 422.9 | 459.2 | 480.1 | 5.8 |
| A2 | 223.6 | 340.7 | 381.4 | 419.2 | 458.1 | 503.5 | 6.9 |
| A3 | 202.9 | 330.4 | 381.1 | 412.4 | 441.2 | 465.6 | 4.8 |
| B1 | 293.5 | 367.8 | 393.2 | 428.3 | 466.2 | - | 11.0 |
| B2 | 250.1 | 357.6 | 387.8 | 424.3 | 474.5 | - | 12.6 |
| B3 | 245.2 | 352.1 | 384.9 | 413.2 | 453.1 | - | 11.5 |

## CONCLUSION

New kinds of UV-curable naphthyl epoxy acrylates were synthesized from di-functional naphthyl epoxy or multi-functional naphthyl epoxy, acrylic acid, and DMBA as a catalyst. UV-curable liquid compositions were prepared from those naphthyl epoxy acrylates (DNEA and MNEA), different reactive diluents and Irgacure 184. UV-cured films of those liquid compositions were obtained by using a high pressure mercury UV lamp.

FTIR spectra identified the change of C=C absorption band during the UV-curing process. The effect of different reactive diluents and different oligomers on the physical, mechanical and thermal properties of UV-cured films was investigated. The gel content and the pendulum hardness increased with increasing irradiation time, and these gradually turned to constant values with increasing the irradiation time (≥ 10 s). The water absorption decreased to its lowest value when gel content reached a maximum, and the structure of UV-cured films was at its densest. With the increase of mono-functional reactive diluent (BA) in the compositions, the physical and mechanical properties of UV-cured films decreased, except for the water absorption. Dynamic mechanical studies showed different properties for compositions using different naphthyl epoxy acrylates as oligomers. The glass transition temperature was found to decrease in the order of B2 and A2. The storage modulus was in the same order as the glass transition temperature. The results of thermogravimetric analysis (TGA) for the naphthyl epoxy acrylates cured films indicated good thermal stability with no appreciable weight loss until well above 200°C.

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