

Photopolymerization Kinetics and Thermal Properties of Dimethacrylate Based on Bisphenol-S

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ABSTRACT: A dimethacrylate based on bisphenol-S (DBSMA) was prepared and characterized by Fourier Transform infrared spectroscopy (FTIR), Electrospray Ionisation Tandem Mass Spectrometry (ESI/MS) ^1H NMR, and ^{13}C NMR. DBSMA was investigated by a real-time infrared spectroscopy (RTIR), under different conditions such as varying photoinitiator type and concentration, with and without oxygen, mixing with different amounts of a reactive diluent [1,6-hexanediol dimethacrylate (HDDMA)]. The mechanical and thermal properties of these curing films were also investigated by dynamic mechanical analysis and thermogravimetric analysis. The results showed homopolymer of DBSMA has better thermal stability than copolymers of DBSMA/HDDMA systems. Also, the cured DBSMA polymer exhibited higher glass transition temperature (T_g) and better thermal stability compared with commercial available resin 2,2-Bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]propane (BIS-GMA) (CN151). © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

KEYWORDS: dimethacrylate; photopolymerization; kinetics; glass transition temperature; thermal stability

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INTRODUCTION

Epoxy (meth)acrylates are widely used resins in UV-curing systems, because the cured products of these resins showed good mechanical, thermal, and electrical properties along with rapid curing speed and low cost. Among these (meth)acrylate based on bisphenol-A (BPA) epoxy currently play a leading role and are widely used as polymeric materials, such as composite material, coatings, and adhesives.^{1–4} However, they are inefficient to satisfy the required in the field of advanced materials, which require high thermal resistance.

As we known, the different chemical structures of various epoxy (meth)acrylates lead to differences in properties, such as final double bond conversion, glass transition temperature (T_g), modulus, thermal stability.^{5–7} The properties and performance can be improved by the modification of epoxy (meth)acrylate backbone, that is, either during synthesis using agents of different structures or changing UV-curing conditions.

The thermal stability of polymer is an important performance. A large number of resins have been shown to have an increase in char formation and increase in thermal stability as the amount of crosslinking,⁸ on which the final double bond conversion has a great impact. Some special chemical structures and groups, such as the aromatic ring, silicon, sulfone group, have consider-

able influence on the thermal properties. The main decomposition of the epoxy novolac acrylates/methacrylates containing a large number of aromatic rings took place in the temperature 340°C,⁹ Siloxane-incorporated epoxy copolymers were proved to have improvements in thermal stability, owing to the introduce of silicon compounds in the curable films. They could be promising because of their outstanding thermal stability.¹⁰

Bisphenol-S (BPS) epoxy resin (DGEBS) represents an important group of epoxies. BPS has an electron-withdrawing group, $-\text{SO}_2-$,^{11,12} which affects the epoxide group more than the $-\text{C}(\text{CH}_3)_2-$ group of the BPA. The sulfone group $-\text{SO}_2-$ can provide thermal stability, chemical resistance, flexibility,¹³ and intermolecular specific interactions to materials.¹⁴ In comparison with the common epoxy based on BPA, DGEBS has more prominent advantage of resistance to deformation by heat, thermal stability, chemical resistance, and mechanical properties. The acrylated diglycidyl ether of BPS was prepared, and the curing reaction of the acrylated epoxy resin with dicumyl peroxide was investigated by Differential Scanning Calorimetry (DSC).¹⁵ If epoxy methacrylate based on DGEBS is used in UV-curing system, it is also expected to optimize properties, for example, thermal stability and mechanical properties.

In this work, methacrylate resin based on epoxy BPS was synthesized by reacting BPS with epichlorohydrin then with

methacrylic acid (MA). Studies focused on the cured properties of DBSMA with different weight fractions of 1,6-hexanediol dimethacrylate (HDDMA). The photopolymerization kinetics was monitored by real-time infrared spectroscopy (RTIR) under different UV-curing conditions. The mechanical and thermal properties of DBSMA were studied by dynamic mechanical analysis (DMA) and thermogravimetric analysis (TGA).

EXPERIMENTAL

Materials

4,4'-Sulfonyldiphenol (BPS, $T_m = 240^\circ\text{C}$, Jiangsu, China). Epichlorohydrin Analytical Reagent (AR), NaOH (AR), tetramethyl ammonium bromide (TMAB; AR), and MA (AR) were purchased from Beijing Chemical Plant (Beijing, China). Benzyl trimethyl ammonium chloride (BTAC) was obtained from Tianjin Jinke Fine Chemical Institute (Tianjin, China), HDDMA (SR239) and Bis-GMA (CN151) were donated by Sartomer (Warrington, PA, USA). The photoinitiator, 2-hydroxyl-2-methyl-1-phenylpropane-1-one (Darocur1173), benzophenone (BP), and UVI-6976 (triaryl-sulfonium salt, 45 wt % solution in propylene carbonate), was donated by Runtech Chemical (P.R. China), ethyl-4-*N,N*-dimethylaminobenzoate (EDMAB) were obtained from Aldrich (Milwaukee, WI). All the materials in this study were used as received.

Instrumentation

NMR. The ^1H NMR and ^{13}C NMR spectra were recorded on a BrukerAV600 unity spectrometer operated at 600 MHz, with CDCl_3 as solvent and tertamethylsilane as the internal standard.

FTIR. RTIR was obtained on a Nicolet 5700 instrument (Nicolet Instrument, Thermo Company). Series RTIR was used to determine the conversions of double bond with the resolution of 4 cm^{-1} . Mixtures of the monomers with the initiator were prepared, and relatively thin films (The casting knife applied leads to a film thickness of $25\ \mu\text{m}$.) were laminated between two KBr crystals. The blend was by UV spot light source (EFOX Lite, 50-W miniature ac lamp, with 5-mm crystal optical fiber, Canada) at room temperature. The light intensity on the surface of samples was detected by UV Radiometer (Beijing Normal University, China). For each sample, the series RTIR runs were repeated three times, and the error on the reported double bond conversion as a function of polymerization time was less than 2%. And, in most case, it was less than 1%. Conversion data were obtained by monitoring the decay of the acrylate double bond $\text{C}=\text{C}$ peak at $\sim 1630\text{ cm}^{-1}$. On irradiation, the decrease of the $\text{C}=\text{C}$ absorption peak area from 1618 to 1650 cm^{-1} accurately reflects the extent of the polymerization. The double bond conversion (DC) can be expressed as follows:

$$\text{DC} (\%) = [1 - (A_t/A_0)] \times 100\% \quad (1)$$

where A_0 is the initial absorbance before irradiation and A_t is the absorbance value at irradiation time t . The polymerization rate (R_p) was calculated from the slope of the initial linear portion of the conversion-time curves as follows:

$$\frac{R_p}{[M]_0} = \frac{d(\text{DC})}{dt} \quad (2)$$

Table I. Samples and Their Corresponding Weight Fractions

Codes	A	B	C	D	E	F
<i>Percent weight fraction</i>						
DBSMA	100	90	75	50	25	0
HDDMA	0	10	25	50	75	100

where $[M]_0$ is the initial concentration of $\text{C}=\text{C}$ double bonds (mol^{-1})

DMA. Samples (the details are given in Table I) for DMA were irradiated by the same UV light source for 15 min to ensure their completely curing. Mechanical properties were measured with DMA (DMA 242C, NETZSCH, Germany). The samples were uniform size ($35 \times 5 \times 0.8\text{ mm}^3$). The temperature scan was performed over a temperature range from -80 to 200°C with a ramping rate of 4°C per min (frequency of 1 Hz) using extension mode. The loss and storage modulus, as well as the loss tangent ($\tan \delta$, ratio of loss to storage modulus), were recorded as a function of temperature. The glass transition temperature (T_g) was taken to be the maximum of the loss tangent versus temperature curve.

TGA. The decomposition temperature of UV cured films was measured by Netzsch thermal analyzer in the range between 30 and 580°C , with a heating temperature of $10^\circ\text{C}/\text{min}$ in N_2 . The sample weight was about $5 \pm 1\text{ mg}$.

Synthesis

Synthesis of Epoxy Resin DGEBS. The diglycidyl ether of BPS (DGEBS) was synthesized through well-known two-step process with TMAB as catalyst (Figure 1).¹⁵ The BPS 50 g (0.2 mol), epichlorohydrin 500 g (5.4 mol), and TMAB 0.31 g (0.002 mol) were put into the four-neck flask equipped with thermometer, vacuum unit, nitrogen tank, and peristaltic pump for 3 h at 100°C under nitrogen atmosphere. Then, 8 g (0.2 mol) of sodium hydroxide aqueous solution (50 wt %) was added gradually for 3 h at 60°C . After the reaction was complete, the solution was filtered to remove sodium chloride, and the filtrate was washed by 80°C hot deionized water three times. DGEBS was obtained after epichlorohydrin was removed by rotary evaporation and dried in vacuum at 70°C for 24 h. Epoxy value of DGEBS was 0.537 mol/100 g (theoretical value of 0.552 mol/100 g). The yield was 96% was measured by the hydrochloric acid-acetone method.

The IR spectrum of DGEBS showed the characteristic peak of epoxy group at 915 cm^{-1} .

ESI/MS, m/z : calcd mass 362, $[M+H]^+ = 363.1$, $[M+NH_4]^+ = 380.2$, $[M+Na]^+ = 385.1$.

^1H NMR (CDCl_3), δ (ppm): 7.78, 7.0 (8H, Ar-H), 4.30, 3.95 (4H, $\text{O}-\text{CH}_2-\text{CH}$), 3.34 (2H, $\text{CH}-\text{CH}_2$), 2.91, 2.74 (4H, CH_2-O).

^{13}C NMR (CDCl_3), δ (ppm): 134.35 ($-\text{SO}_2-\text{C}-$), 129.57, 115.03 ($\text{CH}=\text{CH}$), 161.95 ($\text{C}-\text{O}$), 69.08 ($-\text{O}-\text{CH}_2-$), 49.78 ($-\text{CH}(\text{O})-\text{CH}_2$), 44.48 (CH_2-O).

Synthesis of Dimethacrylated Diglycidyl Ether of BPS (DBSMA). A mixture of 63.35 g (0.175 mol) of DGEBS and 1, 4-dioxane (300 mL) was placed into a 500-mL four-necked bottle equipped with stirrer, dropping funnel, thermometer, and

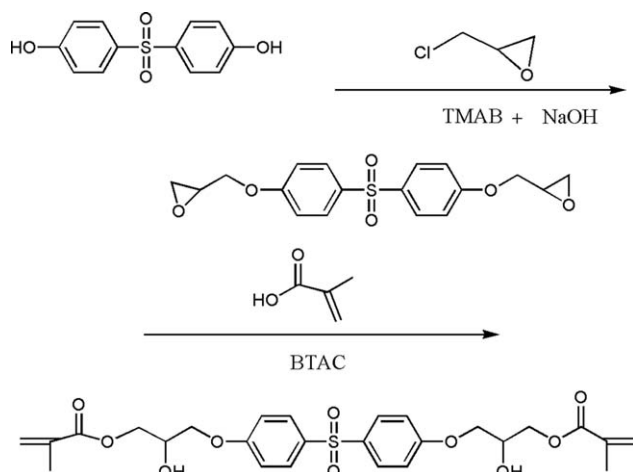


Figure 1. The synthesis process of DBSMA.

condenser, and then it was heated to 80°C. A mixture of 36.12 g (0.42 mol) MA, hydroquinone monomethyl ether (as inhibitor), and BTAC was added slowly. After dropping, the reaction was kept at 80°C for 6 h and at 90°C for about 24 h. FTIR was used to monitor the process of the reaction. The reaction was complete until the absorbance peak of epoxy group at 915 cm^{-1} disappeared. Subsequently, 1, 4-dioxane was removed by rotary evaporation. The mixture was dissolved in chloroform and washed three times with 200 mL of sodium hydroxide aqueous solution (2.5 wt %) and 200 mL deionized water to remove the MA residue and inhibitor. Then, the chloroform removed by rotary evaporation. The product was obtained, and the yield was 78%.

IR: 3475 cm^{-1} ($-\text{OH}$), 2958, 2856 cm^{-1} (CH), 1715 cm^{-1} ($\text{C}=\text{O}$), 1637 cm^{-1} ($\text{C}=\text{C}$).

ESI/MS, m/z : calcd mass 534, $[\text{M}+\text{H}]^+ = 535.1$, $[\text{M}+\text{Na}]^+ = 557.1$, $[\text{M}+\text{K}]^+ = 573.1$.

^1H NMR (CDCl_3), δ (ppm): 7.78, 6.93 (8H, Ar-H), 4.22, 3.98 (4H, $\text{O}-\text{CH}_2-\text{CH}$), 4.35 (2H, $-\text{CH}-\text{OH}$), 4.45, 4.20 (4H, $-\text{CH}_2-\text{OC}(=\text{O})$), 2.03 (2H, $-\text{OH}$), 1.90 (6H, $-\text{CH}_3$), 6.09, 5.57 (4H, $=\text{CH}_2$).

^{13}C NMR (CDCl_3), δ (ppm): 134.17 ($-\text{SO}_2-\text{C}-$), 129.53, 115.06 ($\text{CH}=\text{CH}$), 161.95 ($\text{C}-\text{O}$), 69.13 ($-\text{O}-\text{CH}_2-$), 68.25 ($-\text{CH}(\text{OH})-\text{CH}_2$), 65.40 (CH_2-O), 167.46 ($-\text{O}-\text{C}(=\text{O})$), 126.45, 135.71 ($\text{CH}_2=\text{CH}-$), 18.28 ($-\text{CH}_3$).

RESULTS AND DISCUSSION

Photopolymerization Kinetics

The concentration of the photoinitiator was a key factor to affect the photopolymerization kinetics. Optimum cure rate was generally obtained at certain concentration of photoinitiator, while further increases in photoinitiator did not produce corresponding increases in rate of cure. Figure 2 shows the double bond conversion (DC) and the rate of polymerization (R_p) versus the irradiation time of the neat DBSMA with different concentrations of Darocur1173 (0.5–3.0 wt %). It indicated that with increase of Darocur 1173, both the final double bond conversion (DC_f ; from 68.4 to 96.4%) and the maximum polymerization rate (R_p^{max} ; from 0.018 to 0.050 s^{-1}) increased until

reached 2.0 wt % (high concentration photoinitiator produced more free radicals to initiate polymerization when exposed to UV light), but then decreased slightly when initiator concentration was 3.0 wt %. It might attribute to excessively high concentration of photoinitiator, leading to the generation of excess free radicals, which can easily couple each other to terminate propagation reaction. In addition, when concentration of photoinitiator increased, a large number of initiator molecules on the surface layer absorbed a great amount of light energy, resulting in lower light energy transmitted to deep layer of the system.¹⁶

Types of photoinitiators affected the kinetics of photopolymerization. There were great difference among the efficiency of initiators and the final double bond conversion of the monomer. Photoinitiators for free-radical polymerization proceed by either a cleavage mechanism (Type I photoinitiator) or a hydrogen abstraction mechanism (Type II photoinitiator).¹⁷ Figure 3 shows the kinetics of DBSMA photoinitiated by Type I initiator 1173, Type II initiator BP (EDMAB as a coinitiator), and UVI-6976 (cationic initiator). The obtained results revealed that R_p^{max} (0.05 s^{-1}) and DC_f (92%) of 1173 were the highest. Because the generation of free-radical by Type I photoinitiator is generally faster than Type II photoinitiator. However, compared with 1173, UVI-6976 had much lower R_p^{max} (0.013 s^{-1}) and DC_f (63.3%).

Oxygen inhibition could have a great influence on the radical polymerization process. To overcome this shortcoming, several strategies have been used, such as higher initiator concentration and light intensity, thicker film, and inert atmosphere.

Figure 4 shows the result of photopolymerization of DBSMA with and without oxygen. In laminate condition, double-bond conversion reached 70% in less than 150 s, and its DC_f could be 84.56%. However, in air, the polymerization rate slowed down a little, and DC_f was 81.52%. So, oxygen inhibition could have no significant impact on the radical polymerization process of DBSMA. A few reasons could be used to explain it. The DBSMA has high viscosity that could decrease the oxygen diffusion, and the build-up of the network could also slow down the

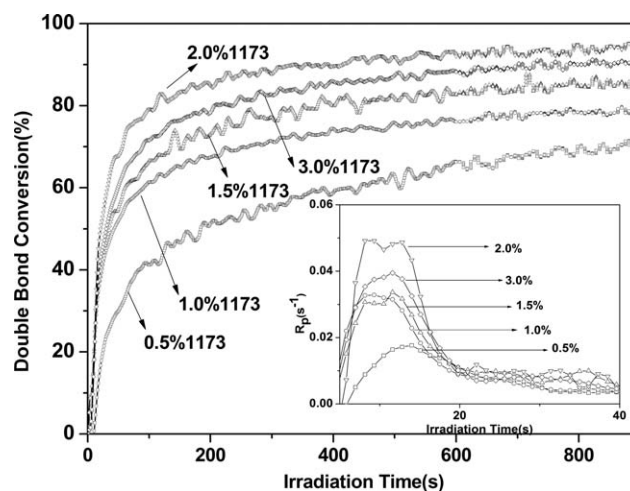


Figure 2. Effect of 1173 concentration on photopolymerization of DBSMA. Inset: polymerization rate plots ($I = 30 \text{ mW}/\text{cm}^2$).

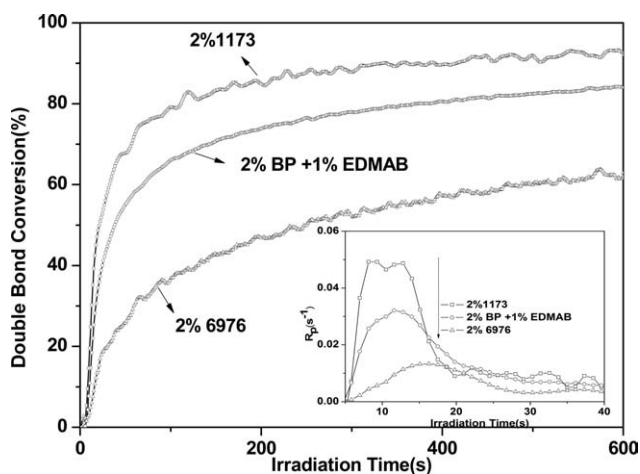


Figure 3. Effect of photoinitiator type on the photopolymerization of DBSMA. Inset: polymerization rate plots ($I = 30 \text{ mW/cm}^2$).

diffusion of O_2 into the sample. Moreover, $-\text{SO}_2-$ could be associated with hydroxyl, and thus, O_2 could be difficult to diffuse in sample. The low sensitivity of methacrylates to oxygen inhibition could also be an important factor.¹⁸

To meet the requirement of practical applications such as workable viscosity, excellent physical, and mechanical properties, the reactive diluents are preferred, which could provide systems with appropriate reactivity, viscosity and coatability before cure, desired cross-link density, chemical resistance, and so forth.

Different weight ratios of HDDMA (Figure 5) were used as reactive diluents for DBSMA. The obtained results showed that different weight ratios of HDDMA developed different DC_f . For the neat DBSMA and HDDMA, their DC_f were 78.4% and 66.5%, respectively (see Table III). The monomer composition of DBSMA/HDDMA = 75/25 wt % (molar ratio 5 : 4) had the highest R_p^{max} , and 50/50 wt % (molar ratio 1 : 2.5) obtained the highest DC_f . The maximum reactivity in resins occurred in the vicinity of the equimolar, and the highest final conversion happened at molar ratio 1 : 2.5 as had been reported for the

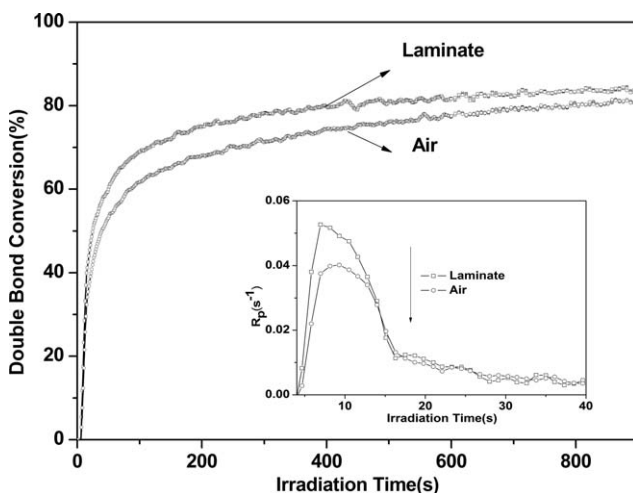


Figure 4. Effect of oxygen on the photopolymerization of DBSMA. Inset: polymerization rate plots (1173 = 1%, $I = 30 \text{ mW/cm}^2$).

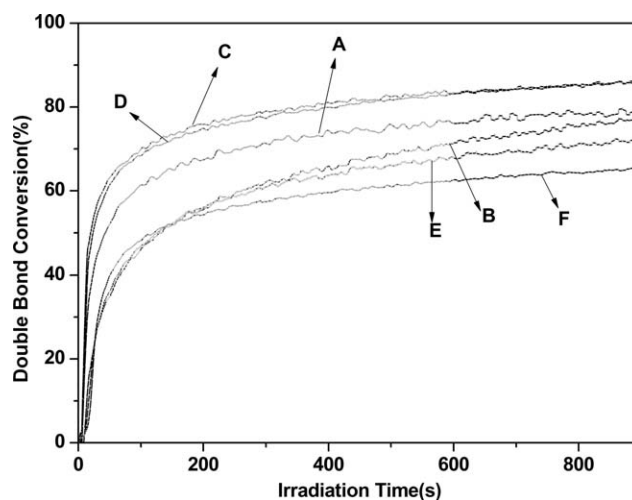


Figure 5. Effect of monomer composition of DBSMA/HDDMA (Table I). Inset: polymerization rate plots (1173 = 1%, $I = 30 \text{ mW/cm}^2$).

acrylate analog of Bis-GMA and for Bis-GMA/TEGDMA: Triethylene Glycol Dimethacrylate mixtures.^{5,19–21} This could be expected that a type of intermolecular interactions between HDDMA and DBSMA molecules ($\text{C}=\text{O}$ and OH groups). The carbonyl oxygen of HDDMA undergoes H bonding with the hydroxyl group of DBSMA, and the formation of the hydrogen bond shifts some of the electron density of the carbonyl oxygen to the hydrogen bond. The partial positive charge at the carbonyl carbon will be enhanced and resulting in a stronger electron deficiency at the double bond to adjust for this loss in electron density. So, a high electron deficiency of a double bond led to an increase in radical addition rate,²² and the effects were maximized at equimolar. Increasing the amounts of reactive diluent (more than 50%) caused the decrease of the rate of polymerization and the increase of the DC_f . This type of behavior for the 75 wt % HDDMA system suggested that the reaction was no longer diffusion controlled, because the HDDMA had given the monomer an even greater amount of mobility.

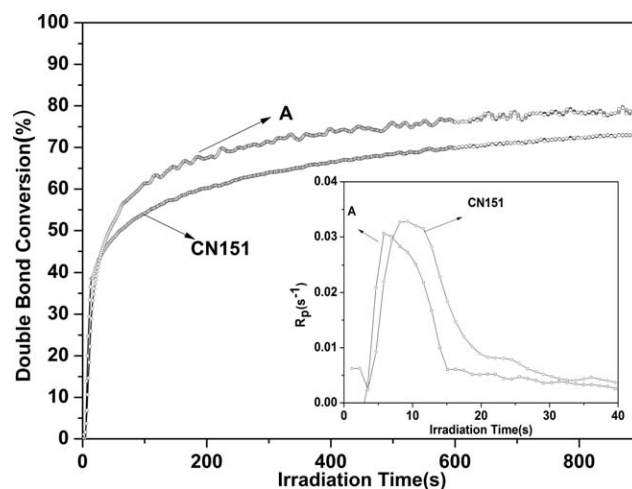


Figure 6. Conversion and rate versus time curves neat DBSMA (A) and CN151. Inset: polymerization rate plots ($I = 30 \text{ mW/cm}^2$).

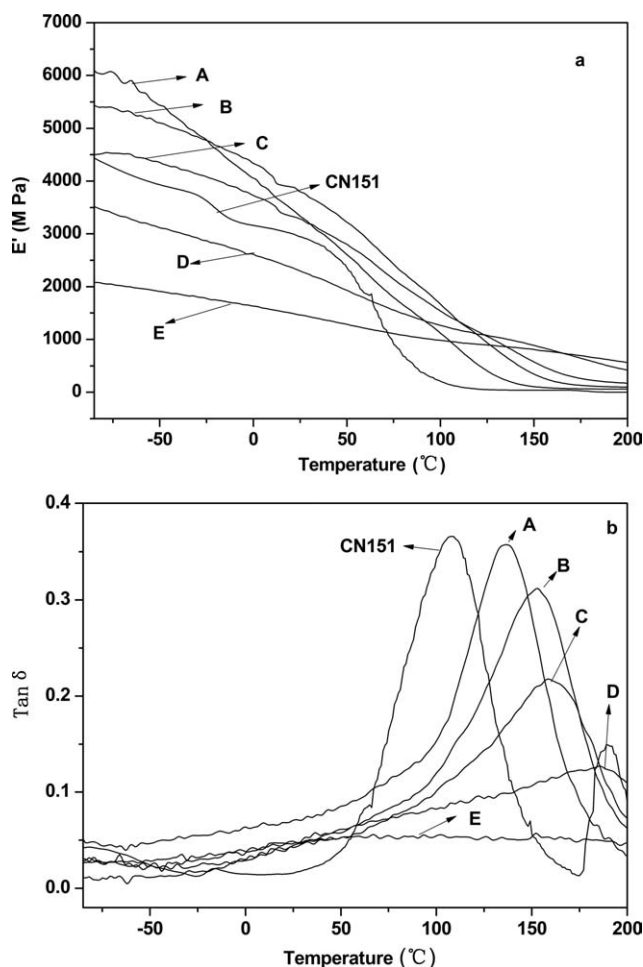


Figure 7. Storage modulus E' (a) and the loss factor $\tan \delta$ (b) for different cured samples.

The reactivity of the UV-curable resin depends on the functionality and chemical structure of the (meth)acrylated oligomer used to cure, as well as on its viscosity, which affects the propagation and termination rate constants.²³ Figure 6 shows conversion and rate versus time curves for methacrylates exposed to UV radiation in the presence of 1% 1173. DC_f values of A and CN151 were 78.4% and 73.6%, respectively, whereas R_p^{\max} values of that were 0.0306 S^{-1} and 0.0328 S^{-1} . This result could be attributed to the fact that different chemical structure and viscosity of methacrylates.

Mechanical Property of DBSMA

The DMA results for different samples are shown in Figure 7 and Table II. Figure 7(a) shows the modulus decreased in glassy regions and increased in rubbery regions as the amount of

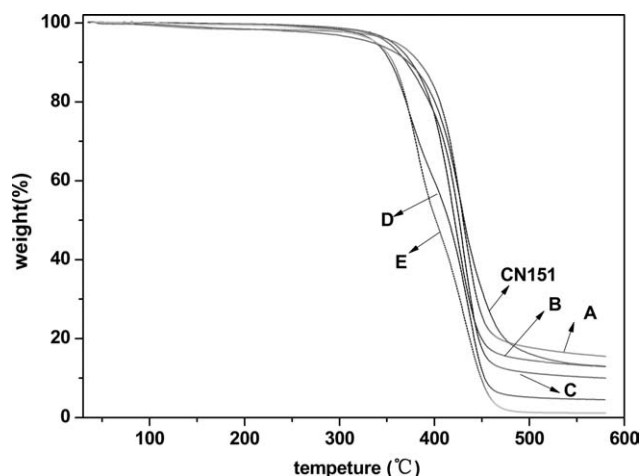


Figure 8. TGA measurements of the different cured samples.

HDDMA increased. The modulus was affected by two factors: bulky rigid BPS content and cross-linking density.^{24,25} The glass transition temperature T_g was indicated by the peak of $\tan \delta$ [see Figure 7(b)]. Similar to above, T_g was also controlled by two factors: BPS content and cross-linking density. Incorporation of the bulky rigid BPS structure increased rigidity and acted as a motion barrier and resulted in a higher T_g . An increase of the cross-linking density could reduce the chain mobility and, therefore, increased the T_g . However, more DGEBS in the network will result in relatively lower cross-linking density, because HDDMA has lower molecular weight than DGEBS. As a compromise, comonomer containing 50% HDDMA (D) displayed the highest T_g . The cured DGEBS homopolymer showed a relatively higher glass transition temperature and storage modulus than that of CN151. This could be that the double bonds of DGEBS have higher reactivity due to the sulfone linkage in DGEBS than that of isopropyl in CN151, which resulted in higher cross-linking density.¹⁵

The Thermal Stability

TGA is the most favored technique for rapid evaluation in the thermal stability and degradation behaviors of various polymers.^{26–28} Figure 8 and Table III show the TGA thermograms of the cured polymers in N_2 . Figure 8 indicates that $T_{d,5\%}$, $T_{d,10\%}$, and char yield at 600°C of polymers decreased with increasing comonomer HDDMA. $T_{d,5\%}$ indicates the apparent thermal stability of the cured polymers. It could be due to the rigid BPS structure content of copolymer decreasing. From the maxima of weight loss rate, it could be inferred that two general degradation mechanisms were taking place, which could be related to the prepolymer composition. The cured DGEBS

Table II. Some Properties of Samples

	A	B	C	D	E	F	CN151
<i>Samples codes</i>							
Final DC (%)	78.4	76.4	84.5	86.4	70.8	66.4	73.6
Viscosity (Pa·s)	59.2	23.1	5.0	0.88	0.028	0.008	41.5
T_g ($^\circ\text{C}$)	137.1	153.1	158.9	186.1	-	-	104.1

Table III. TGA of Cured Samples

Samples code	$T_{d,5\%}^a$ (°C)	$T_{d,10\%}^b$ (°C)	T_{max}^c (°C)	Char _{600°C} (%)
A	359.1	384.3	429.2	15.49
B	355.6	375.9	419.9	12.96
C	350.7	370.3	430.2	10.04
D	338.1	353.5	374.2/436.5	4.52
E	339.5	356.3	379.6/430.7	1.14
CN151	325.7	363.5	413.0	12.88

^aTemperature of 5% weight loss, ^bTemperature of 10% weight loss, ^cTemperatures of maximum weight loss rate.

homopolymer degraded in one single step with the maximum weight loss rate around 430°C. As more than 50% HDDMA was added, a new maximum of weight loss rate appeared around 380°C, which became the main degradation step as the HDDMA content further increased. As explained earlier, the introduction of HDDMA as comonomer decreased the thermal stability, and a second degradation step appeared at a lower temperature. So, low viscosity comonomer was gained at the cost of sacrificing thermal stability of polymer. The homopolymer of DGEBS showed much higher $T_{d,5\%}$, $T_{d,10\%}$, T_{max} , and Char_{600°C} than that of CN151. This result could attribute to the fact that the $-\text{SO}_2-$ bond was more stable than the $-\text{C}(\text{CH}_3)_2-$ bond.

CONCLUSIONS

A dimethacrylate DBSMA based on BPS was successfully synthesized. Results from photopolymerization kinetics showed oxygen inhibition had no significant impact on the radical polymerization process of DBSMA. Although homopolymer of DBSMA had higher viscosity and lower glass transition temperature, it had better thermal stability than copolymers of DBSMA/HDDMA systems. With the introduction of BPS in the skeleton, the resulting the cured film of DBSMA exhibited higher glass transition temperature and better thermal stability than Bis-GMA (CN151).

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