Cure Characterization in Bis(maleimide)/Diallylbisphenol A Resin by Fluorescence, FT-IR, and UV-Reflection Spectroscopy

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ABSTRACT: Curing bis(maleimide)/diallylbisphenol A (BMI/DABPA) results in the formation of a high-temperature thermoset resin. FT-IR, fluorescence, and UV-reflectance spectroscopy were used to investigate the cure behavior of this material under three different cure schedules. Fluorescence signals were quenched before curing due to the BMI component but increased and eventually leveled off as cure time increased. The largest fluorescence intensity increases occurred after 80% of the phenylmaleimide units were converted to phenylsuccinimide. Fluorescence signals were observed in both short-wavelength and long-wavelength regions. Model compound studies indicated that the phenolic portion of the BMI/DABPA resin has a higher quantum yield for fluorescence at a shorter wavelength than phenylsuccinimide derivatives. Therefore, fluorescence emission observed near 356 nm during curing is attributed to phenolic structures. FT-IR was used to quantify the extent of succinimide formation and to identify cross-linking processes which occurred during high temperature curing (250−260 °C). High-temperature curing processes were also identified by UV-reflection spectroscopy. Various reaction pathways are discussed in terms of their consistency with the spectroscopic data.

Introduction

The toughness of bis(maleimide) resins can be improved by copolymerizing these materials with allyl-substituted aromatic compounds. Several years ago, a high-performance thermosetting resin, which is produced by curing BMI (**1**) with diallylbisphenol A (DABPA *2*), was introduced.[1] Properties such as T_g, modulus, and toughness often depend on the extent of cure. Therefore, the optimization of polymer properties and the production of materials with consistent properties calls for convenient and reliable methods for determining the extent of cure. Many high-performance materials are composites which consist of polymeric matrices with a glass or carbon fiber as a reinforcing agent. The presence of such reinforcing agents precludes the use of UV and FT-IR transmission methods for cure monitoring. Research in this laboratory has been focused on the development of novel fluorescence and UV-reflection methods which can be used for cure monitoring of neat and filled resins.[2−6] Extents of cure for unfilled resins can be determined by FT-IR and then correlated with fluorescence or UV-reflection signals from filled or unfilled materials. In addition to being able to obtain extents of cure in filled resins, fluorescence and UV-reflectance methods can be used for on-line cure monitoring with the aide of fiber optic accessories.[3]

Although several reports on BMI/DABPA resin have appeared,[1, 7−14] a clear picture of the chemical transformations which occur during curing has not yet been reached. The following reaction types have been proposed to be involved in the curing process: Ene, Diels−Alder, homopolymerization, rearomatization, and alternating copolymerization.[1,7−14] These processes are outlined in Figures 1 and 2. In all of these transformations, maleimide moieties are converted to succinimide groups. In the preceding paper, we recently reported results on model compounds which were investigated to help assess the likelihood of proposed reaction paths

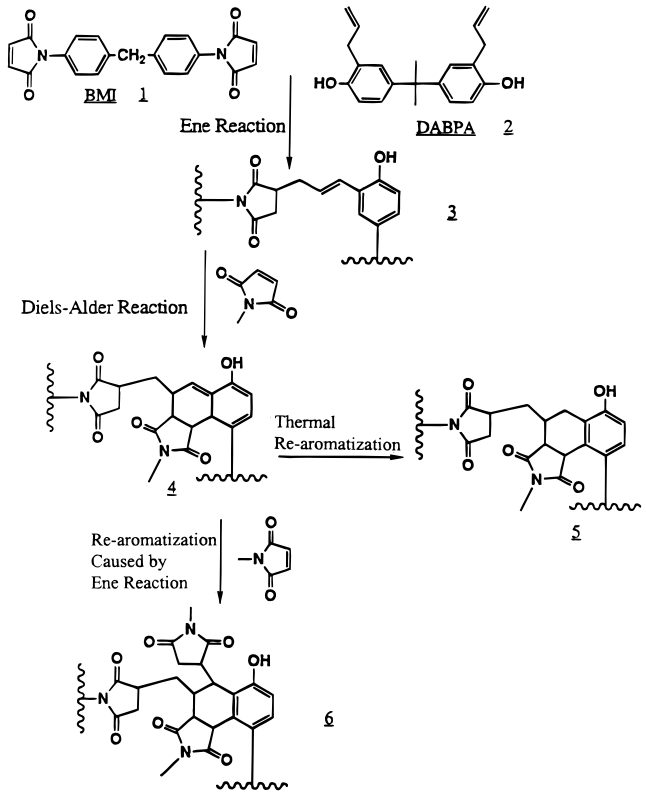

Figure 1. Proposed curing mechanisms for BMI/DABPA resin.[1,7−14]

and to make spectral assignments associated with phenylsuccinimide groups.[15] Our results indicated that styrenelike intermediates such as **3** and **7** are capable of undergoing Diels−Alder−Ene and/or alternating copolymerization reaction sequences. Therefore, processes such as those outlined in both Figures 1 and 2 are possible. In the case of the charge transfer alternating copolymerization, we found that the replacement of a phenyl substituent on the donor vinyl group by an alkyl group results in a significant reduction in molecular weight.[15] This result suggests that 1-propenylphenol cure species such as **2** can undergo charge transfer

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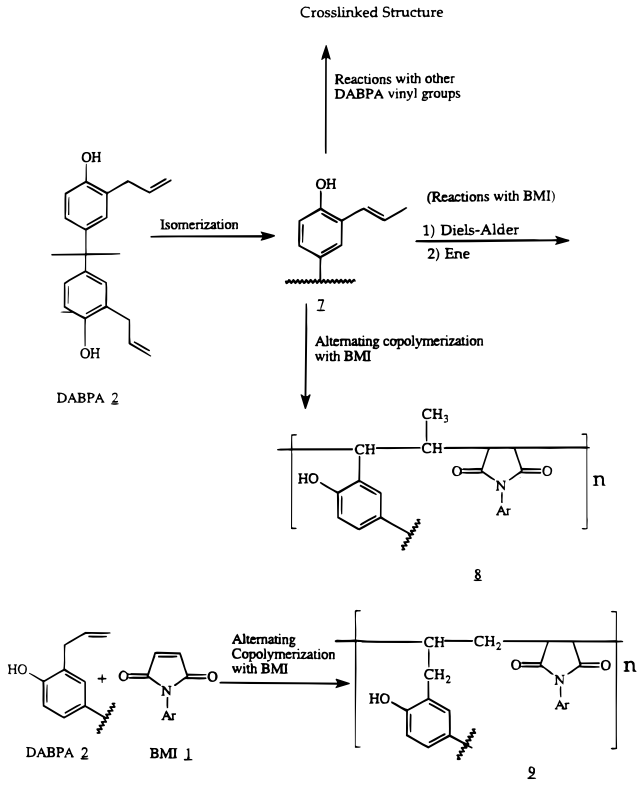

Figure 2. Additional reaction pathways during the curing of BMI/DABPA resin.[1,7−14]

polymerization, but that charge transfer polymerization is more favorable for 2-propenylphenol (styrenelike structures such as **3** and **7**) cure species. The 2-propenylphenol cure species can form by direct isomerization of an allyl group or after an allyl group has undergone the Ene reaction with a maleimide unit. In this study, we use the information obtained on the model compounds in the interpretation of the spectroscopic results obtained during cure cycles. Fluorescence, FT-IR and UV reflection spectral characteristics are used to characterize various reactions.

Experimental Section

Materials. BMI and DABPA were provided by Ciba-Geigy corporation. BMI was recrystallized from methanol/chloroform (1:1 vol/vol) before use and its purity was checked by thermal analysis. DABPA was used as received.

Preparation of Samples for Cure Studies. A 1:1 molar mixture of BMI and DABPA was typically prepared by adding 1.005 g of crushed BMI to 1.169 g of DABPA. A homogeneous mixture of these materials was then obtained by heating and stirring the mixture at 150 °C in an oil bath for about 10 min. Samples for fluorescence and UV-reflection cure monitoring were prepared by spreading molten BMI/DABPA between two quartz plates followed by clamping. Mylar strips (60−300 μm in thickness) were used as spacers. Samples for FT-IR cure monitoring were prepared by spreading molten BMI/DABPA between NaCl disks. The thicknesses of these films were such that absorbance values did not exceed 1.0. Fluorescence spectra were recorded on a Perkin−Elmer LS 50B spectrometer, UV-reflection spectra were recorded with a Perkin−Elmer Lambda 6 UV−vis spectrometer equipped with a specular reflectance accessory (Perkin−Elmer accessory No. 5500228). The incident and reflection angles are 6° in this accessory. The base lines in the UV-reflectance spectra were offset to zero.

Cure Procedures. Samples were cured in a Blue-M circulating oven at preset temperatures in air. Samples were removed from the oven at timed intervals and allowed to cool to room temperature before their spectra were recorded. Experiments were done in duplicate or triplicate. The cure schedules are as follows: (A) isothermal cure at 200 °C; (B) step cure at 180 °C (1 h), 200 °C (2 h), and 260 °C (6 h); C) step cure at 140 °C (6 h), 200 °C (4 h), and 250 °C (9 h).

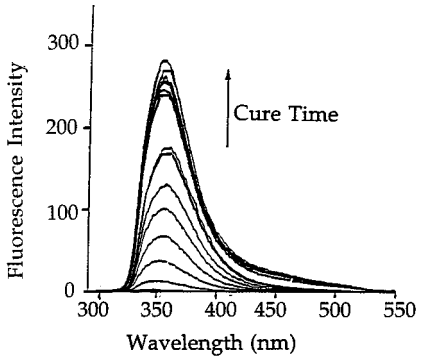

Figure 3. Fluorescence emission spectra obtained by exciting BMI/DABPA at 280 nm as a function of cure time (cure cycle C). Cure time increases from bottom to top (390, 420, 450, 480, 510, 540, 570, 600, 630, 690, 780, 900, 1020, 1140, and 1200 min).

Results and Discussion

Cure Studies. a. BMI/DABPA Curing: Fluorescence Results. On the basis of the fluorescence characteristics of DABPA, imide model compounds, and the quenching ability of BMI,[15] one might expect to observe a variety of changes in the fluorescence spectra of BMI/DABPA during curing. This was indeed the case. The fluorescence changes associated with three different cure schedules for BMI/DABPA curing are presented here.

Although DABPA displays strong fluorescence in both dilute solution and a neat state, fluorescence is essentially quenched in mixtures prepared by melting 1:1 molar amounts of BMI and DABPA together. As previously noted,[15] BMI is responsible for quenching DABPA fluorescence and, therefore, little or no fluorescence is observed near the beginning of BMI/DABPA curing. After sufficient curing, enough BMI is converted to succinimide and one can observe fluorescence signals associated with phenolic-like structure as well as phenylsuccinimide units. Figure 3 shows fluorescence emission spectra, when excited at 280 nm, for BMI/DABPA curing. The increases in intensity which occur as a function of cure time reflect reduced quenching of DABPA fluorescence as maleimide is converted to succinimide. These spectra are believed to correspond to a phenolic-like structure because they are very similar to the fluorescence emission spectrum obtained by exciting a neat sample of DABPA at 280 nm. Figure 4 shows the effect of different cure schedules on phenolic emission intensity observed at 356 nm during BMI/DABPA curing. As can be seen in Figure 4, fluorescence is essentially quenched at the start of curing. Intensity then increases as cure proceeds and finally appears to level off. As one might expect, cure schedules A and B (Figure 4) give similar intensity−time profiles. Curve C corresponds to a lower initial cure temperature. Therefore, fluorescence intensity begins to increase at correspondingly longer cure times relative to cure schedules A and B.

According to the model compound studies, fluorescence signals associated with phenylsuccinimide moieties should occur at longer wavelengths than phenolic signals.[15] Fluorescence excitation spectra for BMI/DABPA when scanned at 456 nm as a function of cure time are shown in Figure 5. As expected, these signals

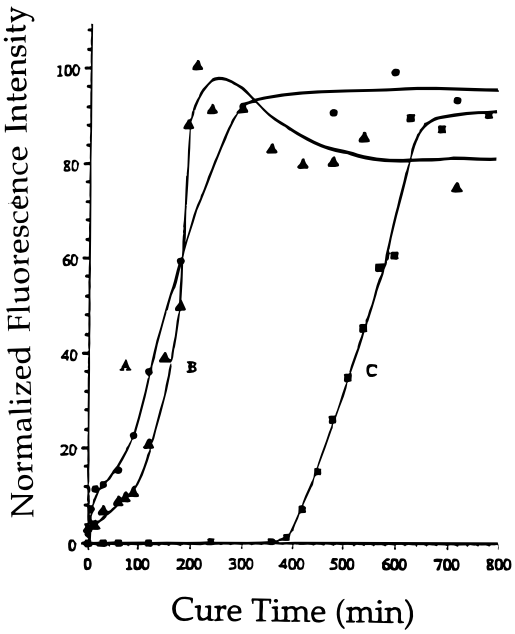

Figure 4. Effect of different cure schedules on fluorescence emission intensity at 356 nm for BMI/DABPA curing, with excitation at 280 nm

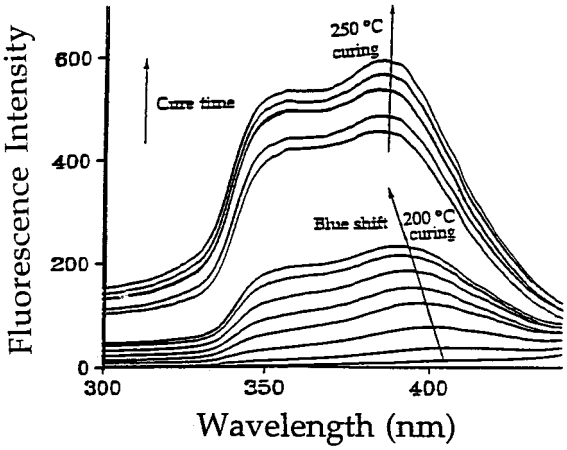

Figure 5. Fluorescence excitation spectra as a function of cure time for BMI/DABPA (cure schedule C), with emission wavelength at 456 nm. Cure time increases from bottom to top (390, 420, 450, 480, 510, 540, 570, 600, 630, 690, 780, 900, 1140, and 1200 min).

occur at longer wavelengths than the phenolic excitation signals. The phenolic species show excitation peaks near 280 nm while the phenylsuccinimide species show excitation peaks near 350−400 nm. As can be seen in Figure 5, the excitation peak intensities increase with increasing cure time. In addition, the phenylsuccinimide peak near 405 nm is shifted to a shorter wavelength during 200 °C curing and the first 30 min of 250 °C curing (cure schedule C). A total blue shift of about 23 nm occurs during curing. The blue shifting might be due to decreases in polarity which accompany curing.[2] According to model compound studies, the excitation spectra shown in Figure 5 are due to the formation of phenylsuccinimide units. A likely reason for the long wavelength fluorescence associated with the phenylsuccinimide groups is charge transfer interactions.[15,16] Figure 6 shows the effect of different cure schedules on fluorescence excitation peak intensity at 385 nm. The variation of the fluorescence excitation peak intensities over time are very similar to the 356 nm emission peak intensity−time profiles corresponding to the reduced

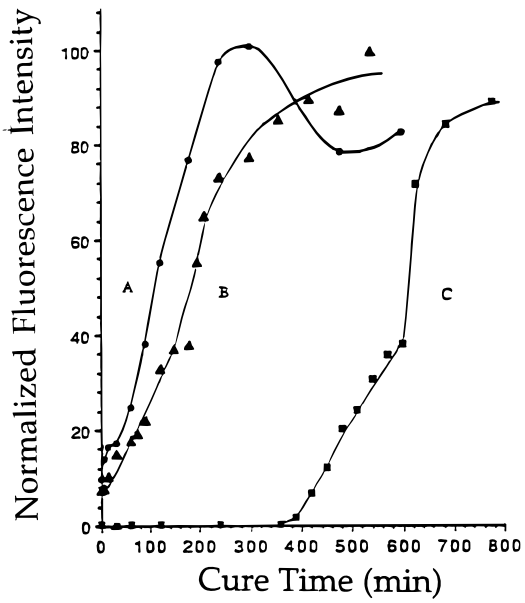

Figure 6. Effect of different cure schedules on fluorescence excitation peak intensity at 385 nm for BMI/DABPA curing, with emission wavelength at 456 nm.

quenching of DABPA (Figure 4). The emission intensity from a phenolic-like structure should depend upon how much phenylmaleimide has been converted to phenylsuccinimide, because phenylmaleimide units quench fluorescence from phenolic-like structures. The phenylsuccinimide fluorescence is also quenched until most of the maleimide units have been consumed.

Excitation of BMI/DABPA near 380 nm produced emission peak near 440 nm from the phenylsuccinimide portion of the resin corresponding to a red shift of 84 nm relative to the emissions from the phenolic species. The intensity of this peak increases as a function of cure time. Similar trends were observed, when comparing emission intensity at 440 nm with the corresponding excitation spectra. Although cure schedules A and B are similar for the first three hours of curing, cure schedule B involves curing at 260 °C in its final step while schedule A calls for continued curing at 200 °C. The fluorescence intensity−time profile for schedule A appears to exhibit a maximum while schedules B and C increase initially and then level off. It is obvious from plots of T_g versus cure time at different cure temperatures that BMI/DABPA cured at 250 °C or higher is more highly cross-linked than resin which is not cured at this temperature.[1] Therefore, the latter stages of BMI/DABPA curing appear to involve cross-linking processes which have little impact on fluorescence. It is likely that the cross-linking processes involve reactions of vinyl groups in DABPA residues. According to the mechanism in Figure 1, one allylphenol unit can consume 2−3 units of phenylmaleimide. However, no allyl groups are present at the end of curing according to FT-IR results. These groups are believed to be involved in cross-linking reactions during the latter stages of cure. Support for such a scenario is discussed in the FT-IR cure results below.

b. BMI/DABPA Curing: FT-IR Results. FT-IR was employed to determine the extents of various reactions involved in BMI/DABPA curing. The previously described cure schedules A, B, and C were also used here. Examination of Figures 1 and 2 reveals that phenylmaleimide units are converted to phenylsuccinimide during Ene, Diels−Alder, and copolymerization

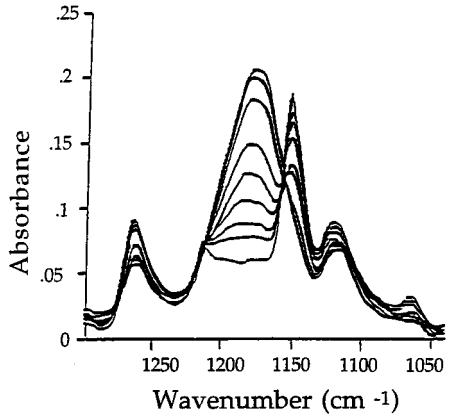

Figure 7. FT-IR spectral changes associated with the conversion of maleimide moieties (1149 cm^{-1}) to succinimide moieties (1180 cm^{-1}) for BMI/DABPA curing, with data from cure cycle C. Cure times were 0, 30, 60, 120, 240, 360, 390, 420, and 600 min.

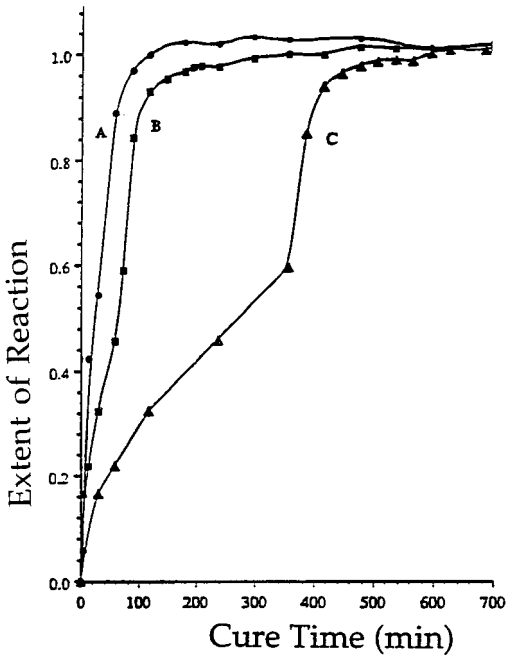

Figure 8. Effect of different cure schedules on extent of succinimide formation as determined by FT-IR spectroscopy.

reactions. Therefore, changes in the C−N−C bands correspond to a composite measurement of Ene, Diels−Alder, and copolymerization reactions. Several distinct changes occur in the FT-IR spectra of BMI/DABPA resin during curing. An absorbance band associated with C−N−C maleimide[18] (centered at 1149 cm^{-1}) decreases while a new absorbance band at 1180 cm^{-1} corresponding to C−N−C succinimide[18] increases as shown in Figure 7. The disappearance of a C−H maleimide band (3101 cm^{-1}) is also readily detected by FT-IR. In addition, the consumption of DABPA allyl groups can be measured from a peak at 914 cm^{-1}. The extent of reaction based on the formation of succinimide for various cure cycles is shown in Figure 8. The extent of succinimide formation was calculated using eq 1.

$$\text{extent of succinimide formation} = \left(\frac{(A_{1180}/A_{1710})_t - (A_{1180}/A_{1710})_0}{(A_{1180}/A_{1710})_\infty - (A_{1180}/A_{1710})_0}\right) \quad (1)$$

The absorbance due to the imide carbonyl at 1710 cm^{-1}

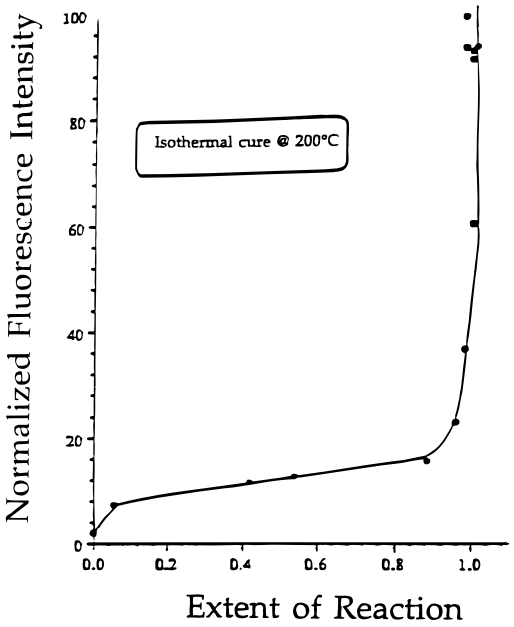

Figure 9. Correlation of fluorescence emission intensity (356 nm) with extent of succinimide formation (determined by FT-IR) for BMI/DABPA curing. Fluorescence excitation wavelength was 280 nm.

(A_{1710}) was used as an internal reference band. The term $(A_{1180}/A_{1710})_t$ is the absorbance at 1180 cm^{-1} at cure time t corrected for thickness while the term $(A_{1180}/A_{1710})_0$ is the absorbance at 1180 cm^{-1} at cure time zero, corrected for thickness. The term $(A_{1180}/A_{1710})_\infty$ corresponds to the infinite time value for the 1180 cm^{-1} absorbance after internal reference correction. Comparison of conversion curves in Figure 8 with fluorescence intensity cure time profiles (Figure 4 or 6) indicates that the largest increases in fluorescence intensity occur after approximately 70−80% of the maleimide units have been converted to succinimide. The conversion of maleimide to succinimide appears to be nearly complete after BMI/DABPA has been subjected to about 200 min of curing under the conditions of cure cycles A or B. The succinimide formation is nearly complete after 500 mins for cure cycle C. Figure 9 shows a plot of fluorescence intensity versus extent of succinimide formation determined by FT-IR. It is clear from this plot that fluorescence intensity changes are most sensitive to the latter stages of maleimide to succinimide conversion. Fluorescence intensity apparently stays low until a high conversion of maleimide to succinimide is achieved. Once most of the maleimide is consumed, fluorescence quenching is less severe. Figure 10a shows the percentage of C−H maleimide for BMI/DABPA curing as a function of cure time for cure schedule C. The loss of C−H maleimide is nearly complete after 500 min of curing. As previously noted, the formation of succinimide is also nearly complete after 500 min of curing under conditions of cure cycle C. From this observation, it is clear that the loss of C−H maleimide parallels the formation of succinimide. Figure 10b shows a plot of percentage of =C−H allyl from DABPA as a function of cure time for cure cycle C. The loss of allyl groups is nearly complete after 500 min of curing. This result can not be explained alone by the mechanism in Figure 1. This mechanism calls for the consumption of one allylphenol unit for every two or three phenylmaleimide units. Therefore, in a 1:1 molar formulation of BMI/DABPA, one would expect one-half to two-thirds of the allyl groups to remain after

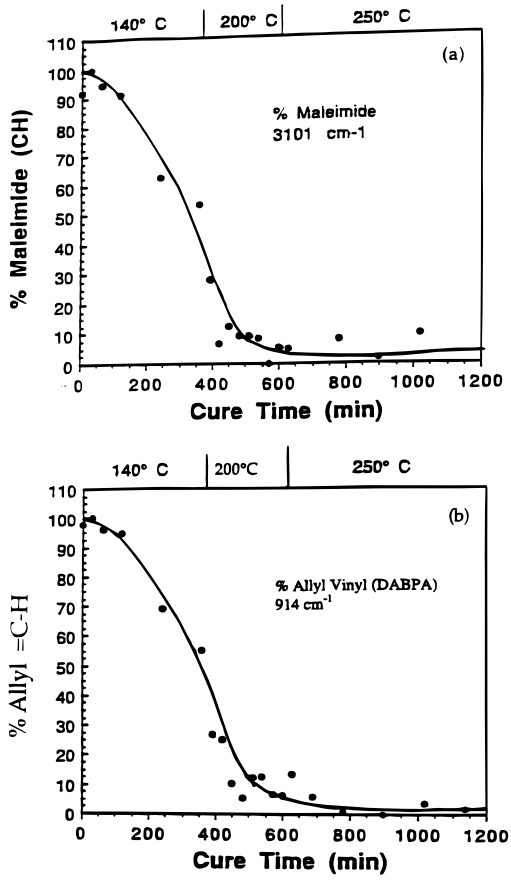

Figure 10. (a) Percent (C−H) maleimide determined by FT-IR at 3101 cm^{-1} as a function of cure time for BMI/DABPA curing for cure schedule C. (b) Percent allyl (from DABPA) determined by FT-IR at 914 cm^{-1} as a function of cure time for BMI/DABPA curing for cure schedule C.

all the maleimide is converted to succinimide. However, examination of Figure 10b clearly demonstrates that allyl groups do not remain at the end of the cure cycles within the detection limit of FT IR (~2%). One explanation for this might be that some of the allyl groups isomerize to yield styrene-like groups, **7**, as shown in Figure 2. The isomerized groups would be expected to react further during high temperature curing cycles (250 or 260 °C). Figure 11 shows that the relative changes due to 2-propenylphenol groups at 974 cm^{-1} for BMI/DABPA curing according to cure cycles A, B, and C are consistent with this scenario. In each case, there is a build up of 2-propenylphenol groups followed by a decrease. In cases involving high-temperature curing (cure cycles B and C), the cure species as indicated at 974 cm^{-1} decreases more sharply after reaching maxima than in cure cycle A. The decrease in the 974 cm^{-1} species is consistent with additional cross-linking reactions involving styrene-like groups (i.e. 2-propenylphenol groups) because this BMI/DABPA which is subjected to curing at 250−300 °C has a significantly higher T_g than BMI/DABPA which is cured at 200 °C.[1] If all reactions involved copolymerization processes such as those outlined in Figure 2, then there would not be an excess amount of unsaturated groups from DABPA. Alternating copolymerization could occur by reaction of phenylmaleimide with the allyl group of DABPA[13] or by reaction with the unsaturated groups in species such as **7**. Species such as **7** can form by thermal isomerization of DABPA[17] or by Ene reaction of maleimide units with DABPA allyl groups.[1,10,11] Alternating copolymerization would mandate resin cure to be complete

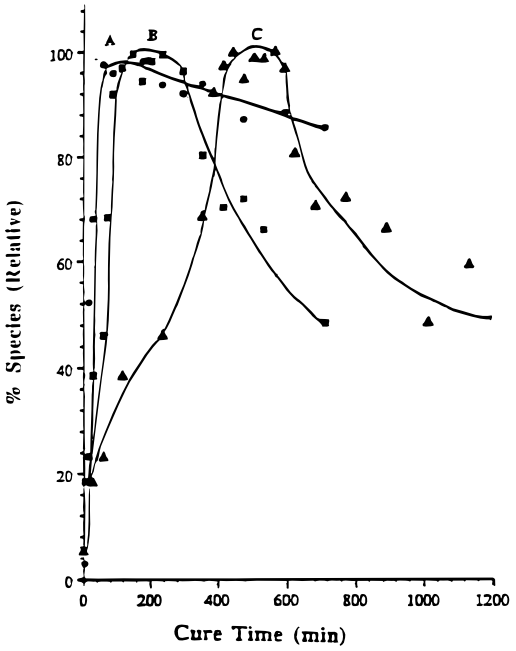

Figure 11. Effect of different cure schedules on relative changes at 974 cm^{-1} band for BMI/DABPA curing.

once all maleimide had been converted to succinimide. Our results clearly demonstrate that BMI/DABPA resin continues to cure even after the conversion of maleimide to succinimide is complete. After all maleimide and unsaturated groups are consumed, the only reactions in Figure 2 which could occur are the cross-linking reactions of the 2-propenylphenol groups. The only reaction which could occur in Figure 1 at this stage is thermal rearomatization.

c. BMI/DABPA Curing: UV-Reflection Results. As previously mentioned, fluorescence spectroscopy can be used to monitor curing in thick samples or in samples which contain fillers or reinforcing agents. However, not all resin systems show variations in their fluorescence signals throughout their entire cure cycles. Fortunately, many polymerization systems do show UV−vis absorption bands which are sensitive to changes in functionality which accompany curing. The UV-reflection changes which accompany the curing of BMI/DABPA resin are discussed below. Cure schedules A, B, and C were also followed in the UV-reflectance work. It should be noted that UV-reflection spectra typically have their peak maxima red shifted by 10−15 nm relative to UV-transmission spectra. The relationship between reflection spectral peak positions and absorption peak positions has been recently discussed by Yu and Sung.[4]

Figure 12 shows UV-reflection spectra of BMI and DABPA. Although the spectral regions of these two materials overlap, some distinctions can be made. First, the DABPA shows a weak but well-defined peak near 295 nm due to fine band structure typical of phenolic groups while BMI displays weak tailing in this region. Secondly, BMI has a well-defined peak near 230 nm while DABPA displays a shoulder in this region. UV-reflection spectra of BMI/DABPA resin before and after "full cure" show an overall decrease in the percentage reflectance near 230 nm and 295 nm. An overall increase in the percentage reflectance occurs near 266 nm. Because the changes in the percentage reflectance which accompany the curing are small, UV-reflection difference spectra were generated. These spectra were

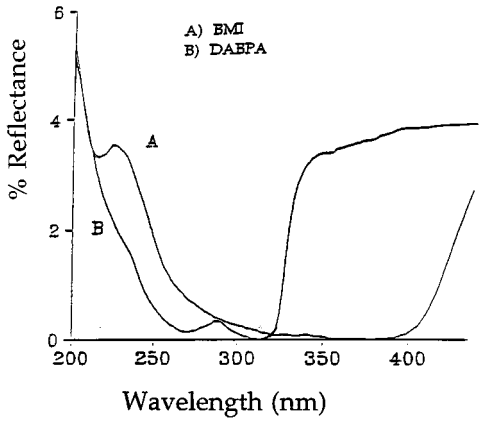

Figure 12. UV-reflection spectra of BMI and DABPA.

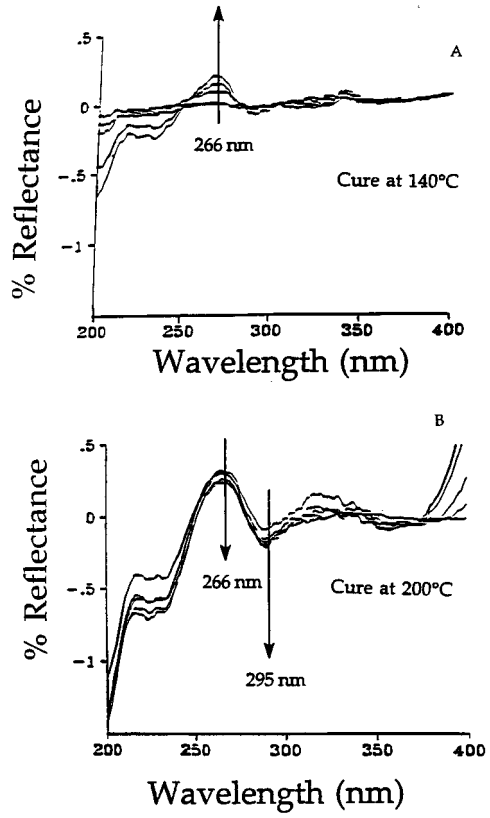

Figure 13. UV-reflection difference spectra for BMI/DABPA as a function of cure for cure schedule C: (A) cure time from bottom to top (30, 60, 120, 240 and 360 min); (B) Cure time from top to bottom (390, 420, 450, 510, 540 and 600 min).

obtained by subtracting a UV-reflection spectrum of uncured BMI/DABPA from BMI/DABPA spectra at various cure times. Some of these spectra are shown in Figure 13. Signals which appear above the zero mark correspond to processes which lead to increases in the percentage reflectance (relative to uncured BMI/DABPA) while peaks which appear below the zero mark correspond to processes which lead to decreases in the percentage reflectance. One can clearly see peaks in the difference spectra near 266, 295, and 325 nm. The changes in the percentage reflectance at 295 and 266 nm are best displayed when they are plotted against cure time. There is no clear trend associated with the 325 nm peak. The effect of different cure schedules on the changes in the percentage reflectance at 295 nm is shown in Figure 14. Comparison of these plots for cure cycles B and C reveals that the initial decreases in the percentage reflectance at 295 nm are followed by

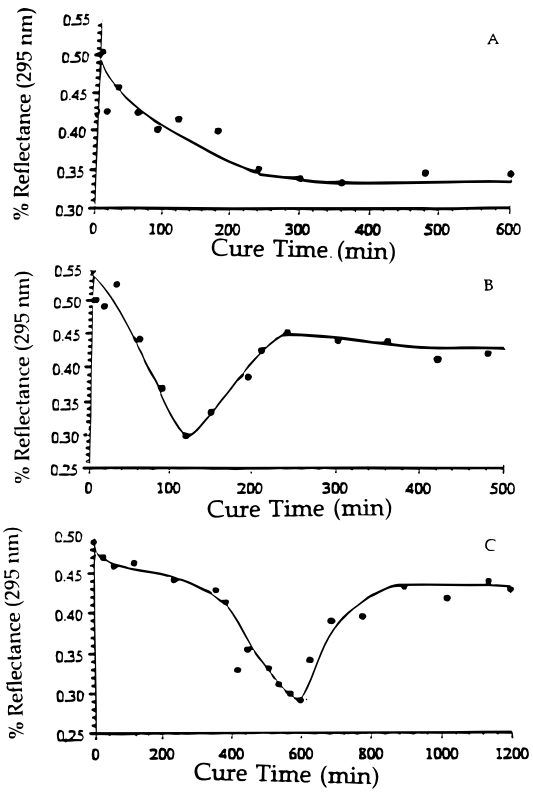

Figure 14. Effect of different cure schedules on the percent reflectance at 295 nm during BMI/DABPA curing.

increases. The increases occur mainly during the high temperature curing steps (i.e. 250 and 260 °C curing). In the case of isothermal curing at 200 °C (cure cycle A), only decreases in percentage reflectance at 295 nm are observed, which suggests that high temperature curing is required for the recovery in the percentage reflectance at 295 nm. The cure profiles for cycles B and C (295 nm changes) are indicative of a process which involves the consumption and subsequent reformation of a species. According to the mechanism in Figure 1, this would involve destruction of DABPA aromatic rings during Diels−Alder reactions followed by a rearomatization step. It has been suggested that rearomatization can occur thermally[1,14] or by the Ene reaction.[10,11] We believe that some rearomatization occurs without the aid of phenylmaleimide because the recovery of the 295 nm UV-reflection peak occurs after succinimide conversion as determined by FT-IR is complete. We have found that FT-IR changes in C=C aromatic absorbance are not reliable for monitoring dearomatization and rearomatization processes for BMI/DABPA.[19] Figure 15 shows the effect of different cure schedules on the percentage reflectance at 266 nm during BMI/DABPA curing. Grunden et al.[20] found that styrene shows a peak in this region whose reflectance decreases as polystyrene forms. Therefore, it is likely that the 266 nm peak corresponds to a styrene-like intermediate (i.e. a 2-propenylphenol species). As can be seen in Figure 15, the percentage reflectance at 266 nm initially increases and then decreases for all three cure cycles. However, the decreases are much less pronounced for cure cycle A. The higher curing temperatures in cycles B and C are likely to be responsible for the greater decreases in the percentage reflectance at 266 nm for cycles B and C. The cure profiles for cycles B and C are characteristic of the formation and subsequent consumption of an intermediate species. A possible explanation for this behavior could be the

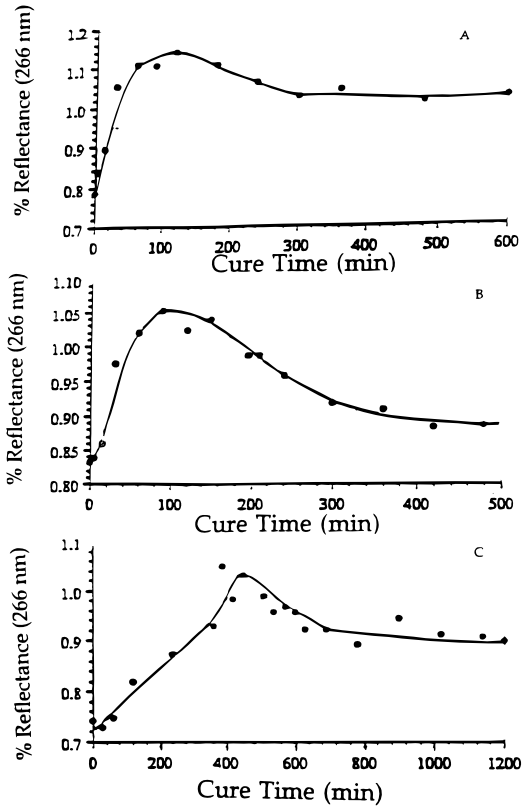

Figure 15. Effect of different cure schedules on the percent reflectance at 266 nm during BMI/DABPA curing.

conversion of allyl groups in DABPA to styrenelike groups (2-propenylphenols) through Ene and isomerization reactions. The styrene-like groups could then be consumed in high-temperature (250 and 260 °C) cross-linking reactions.

Summary

None of the spectral signals in this study were observed to vary monotonically for the entire BMI/DABPA cure schedules. However, UV-reflectance peaks and the 974 cm^{-1} FT-IR peak do appear to vary throughout the entire BMI/DABPA cure cycle. The nature of these spectral changes require data collection throughout entire cure cycles to determine when the BMI/DABPA resin is "fully cured" within the detection limits of these techniques, which are estimated to be about 95−98% of cure. Fluorescence and FT-IR can be used to monitor the conversion of maleimide to succinimide. However, fluorescence is most effective for monitoring the latter stages of maleimide to succinimide conversion. The final stages of curing appear to involve cross-linking reactions of olefin groups in the DABPA residues. FT-IR and UV-reflection spectra appear to be sensitive to these changes while fluorescence appears to be insensitive to these reactions. To "fully cure" the BMI/DABPA resin, high temperature treatments (250−260 °C) appear to be necessary. On the basis of the study of model compounds, the phenolic portion of the BMI/DABPA resin is expected to have a higher quantum yield and fluoresce in a shorter wavelength region than the phenylsuccinimide portion of the resin. Therefore, the emission near 356 nm is attributed to phenolic structure in the BMI/DABPA resin. The emission intensity at 356 nm increases and then appears to level off as cure proceeds. One might expect to observe a decrease in this intensity during a dearomatization reaction. Although there are some fluctuations in the fluorescence signals, there is no clear trend. Apparently any decrease in fluorescence intensity due to a dearomatization process is compensated for by the consumption of quenching species (maleimide units). The FT-IR, fluorescence, and UV-reflection studies reported here and the previously reported model compound studies[15] provide a clearer picture of the processes likely to be involved in the curing of BMI/DABPA resin.

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