Research Article

Dynamic Cure Kinetics and Physical-Mechanical Properties of PEG/Nanosilica/Epoxy Composites

Haleh Nowruz Varzeghani, Iraj Amiri Amraei, and Seyed Rasoul Mousavi

Department of Material Science and Manufacturing Technology, Malek Ashtar University of Technology, Tehran 158751774, Iran

Correspondence should be addressed to Iraj Amiri Amraei; iraj_amiri@yahoo.com

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This study investigated the effect of polyethylene glycol (PEG) and nanosilica (NS) on the physical-mechanical properties and cure kinetics of diglycidyl ether of bisphenol-A-based epoxy (DGEBA-based EP) resin. For this purpose, tensile and viscometry tests, dynamic mechanical thermal analysis (DMTA), and differential scanning calorimetry (DSC) were carried out under dynamic conditions. The results showed that adding NS and PEG enhances the maximum cure temperature as well as the heat of cure reaction ($\Delta H$) in EP-NS, while it decreases in EP-PEG and EP-PEG-NS. The cure kinetic parameters of EP-PEG-NS were calculated by Kissinger, Ozawa, and KSA methods and compared with each other. The $E_a$ calculated from the Kissinger method (96.82 kJ/mol) was found to be lower than that of the Ozawa method (98.69 kJ/mol). Also, according to the KAS method, the apparent $E_a$ was approximately constant within the 10-90% conversion range. Tensile strength and modulus increased by adding NS, while tensile strength diminished slightly by adding PEG to EP-NS. The glass transition temperature ($T_g$) was calculated using DMTA which was increased and decreased by the addition of NS and PEG, respectively. The results of the viscometry test showed that the viscosity increased with the presence of both PEG and NS and it prevented the deposition of solid particles.

1. Introduction

Because of their excellent thermal and mechanical properties, epoxy (EP) resins are widely used in structural adhesives [1], insulating materials [2], resin matrices for high performance composites [3], protective coatings [4], electronic and electronic components [5, 6], etc. These wide applications are due to creep resistance [7], excellent adhesion to many substrates [8], high temperature performance [9], high stiffness [10], and high mechanical and electrical properties [11]. However, almost all EPs are brittle [12–14]. Many tougheners such as carboxyl-terminated butadiene-acrylonitrile (CTBN) [15, 16], core-shell rubber (CSR) particles [17, 18], thermoplastic or thermoset polymers [19, 20], and organic-inorganic particles [21, 22] have been incorporated to increase the toughness of EP resins. These tougheners decrease modulus, tensile strength, and glass transition temperature ($T_g$); so in order to enhance the mechanical and thermal properties, the toughened EPs are reinforced by nanosilica (NS) particles. Mousavi and Amraei [23] used EP-CSR-NS composites and found that NS partially offsets the reduction in properties caused by CSR.

The properties of cured EPs depend on curing conditions such as time and temperature. Differential scanning calorimetry (DSC) has been used to investigate the kinetics of cure process under dynamic [24, 25] and isothermal [26, 27] modes. Many kinetic models have been used to study kinetic parameters, such as Kissinger [28, 29], Ozawa [30, 31], Kissinger-Akahira-Sunose (KSA) [32, 33], Flynn-Wall-Ozawa (FWO) [28, 34], Kamal [35, 36], autocatalytic [37, 38], and Borchardt Daniels [39, 40] methods.

Many researchers [41] have observed that the main problem with thermoplastics tougheners is compatibility with the EP resin. Because of aromatic rings in DGEBA EP resin, it has high mechanical properties after curing and it is miscible with polyethylene glycol (PEG) [42, 43]. Thus, this grade of EP resin was blended with PEG and NS in this study. Further, the physical-mechanical, viscometry, and thermal properties
were investigated. Finally, the cure kinetics of EP-PEG-NS composites were studied using Kissinger, Ozawa, and KSA methods.

2. Materials and Methods

2.1. Materials. DGEBA EP resin (Epon 828) was supplied from Shell with an epoxide equivalent weight of 185-192 g/eq. The DICY as a curing agent and Diuron as an accelerator were obtained from Sigma-Aldrich. PEG used was supplied by Merck with an average molecular weight of 4000 g/mol. NS (Aerosil R972) was prepared from Evonik. All of the materials were used as received. The chemical structures of the materials used in this study are shown in Figure 1. The chemical and physical properties also are reported in Table 1.

2.2. Sample Preparation. Initially, EP, DICY, and NS were homogeneously mixed by a Perl Mill. Then, they were blended with Diuron and PEG using a High-Shear mixer (IKA T25). Next, the mixture of resin and other materials was degassed in a vacuum oven at 50°C for 30 min. Afterwards, the samples were gently poured into mold and cured at 120°C for 90 min. The details of experimental setup were according to Mousavi and Amraei previous work (Figure 2) [23]. Details of the composition of the formulations are listed in Table 2.

2.3. Mechanical Properties. A Santam STM-150 tensile/compression testing machine was employed to the investigate mechanical properties of dumbbell shaped specimens at a crosshead speed of 5 mm/min according to ASTM D-638 (I-type).

2.4. Dynamic Mechanical Thermal Analysis. Dynamic mechanical thermal analysis (DMTA) was conducted using a TA Instruments DMTA 2980 analyzer. Rectangular specimens with dimensions of $2 \times 3 \times 48 \text{ mm}^3$ were used.
Temperature sweeps were performed at a rate of 5°C/min and a frequency of 1 Hz, from 25 to 200°C.

2.5. Viscosity Test. Viscosity measurement was performed using a Brookfield viscometer ranging from room temperature up to 65°C according to ASTM D2196.

2.6. Kinetic Models. The curing kinetics of the samples was examined on a differential scanning calorimeter (DSC, Netzsch DSC 200 F3) under nitrogen atmosphere. Dynamic measurements were carried out from 25 to 250°C at heating rates of 2.5, 5, and 10°C/min.

In the DSC analysis, we can assume that the area under the exothermic curve is proportional to the degree of conversion (α), which is expressed as follows [44, 45]:

$$\alpha = \frac{H_t}{\Delta H}$$  \hspace{1cm} (1)

where $H_t$ is the partial heat of cure reaction at time $t$ and $\Delta H$ is the total heat of cure reaction. Curing kinetics of EP is a very complex process, and there are a variety of physico-chemical reaction, and the activation energy ($E_a$) changes at any point in time. Accordingly, in this study, the $E_a$ has been calculated by different dynamic methods. These methods are described as follows:

2.6.1. Kissinger Method.

$$\ln \left( \frac{\beta}{T_p^2} \right) = \ln \left( \frac{AR}{E_a} \right) - \frac{E_a}{RT_p}$$  \hspace{1cm} (2)

where $\beta$ represents the heating rate, $T_p$ is the absolute temperature at the peak, $A$ denotes the preexponential factor, and $R$ is the gas constant. If the curve of $\ln (\beta/T_p^2)$ versus $1/T_p$ is plotted for different heating rates and linear extrapolation is obtained, the slope of that line gives the value of $E_a$.

2.6.2. Ozawa Method.

$$\frac{d(\ln \beta)}{d(1/T_p)} = 1.052 \left( \frac{E_a}{R} \right),$$  \hspace{1cm} (3)

where all of parameters are the same as described before. If the curve of $\ln \beta$ versus $1/T_p$ is plotted for different heating rates and linear extrapolation is obtained, the slope of that line gives the quantity of $E_a$.

2.6.3. Kissinger-Akahira-Sunose (KAS) Method.

$$\ln \left( \frac{\beta}{T_a^2} \right) = C - \frac{E_a}{RT_a},$$  \hspace{1cm} (4)

where $T_a$ is the absolute temperature at a fixed $\alpha$ and $C$ is a constant. If the curve of $\ln (\beta/T_a^2)$ versus $1/T_a$ is plotted for different heating rates and linear extrapolation is obtained, the slope of that line gives the value of $E_a$.

3. Results and Discussion

3.1. Mechanical Properties. Table 3 presents the ultimate tensile strength (UTS), Young’s modulus, and strain at break. It was observed by adding NS, UTS, and Young’s modulus of the EP increased without any reduction in strain at break. It is because as the tensile force is applied to the specimens, the silica nanoparticles lead to increased deformation resistance. On the other hand, the deformation decreases in EP-PEG-NS due to the presence of flexible chains in PEG. As such, the tensile strength declines slightly with respect to EP-NS. Cured EP resin has high internal stresses due to the shrinkage resulting from the cooling from curing temperature to room temperature. These internal stresses affect the performance through producing cavities and microcracks.

### Table 2: Details of composition of the formulations.

| Sample         | DICY (phr) | Diuron (phr) | PEG (phr) | NS (phr) |
|----------------|------------|--------------|-----------|----------|
| EP             | 7          | 3            | 0         | 0        |
| EP-PEG         | 7          | 3            | 0         | 10       |
| EP-NS          | 7          | 3            | 10        | 0        |
| EP-PEG-NS      | 7          | 3            | 10        | 3        |

phr: parts per hundred parts of resin by weight.

### Table 3: Mechanical properties of different samples.

| Sample        | UTS (MPa) | Young’s modulus (GPa) | Strain at break (%) |
|---------------|-----------|-----------------------|---------------------|
| EP            | 73 ± 3    | 2.9 ± 0.05            | 3.1 ± 0.02          |
| EP-MEG        | 69 ± 1    | 2.96 ± 0.1            | 3.24 ± 0.05         |
| EP-NS         | 83 ± 2    | 3.17 ± 0.2            | 4.1 ± 0.3           |
| EP-PEG-NS     | 75 ± 2    | 3.22 ± 0.05           | 3.52 ± 0.2          |

2.6.4. Kissinger-Akahira-Sunose (KAS) Method.
Induction of PEG soft segments to EP dramatically reduces these internal stresses.

3.2. Dynamic Mechanical Thermal Analysis. Figure 3(a) indicates storage modulus ($E'$) versus temperature. A temperature at which the maximum value of tan $\delta$ occurs is the glass transition temperature, $T_g$. All the samples are shown in Table 4. Adding PEG reduces the modulus and $T_g$. One of the reasons for the fall in $T_g$ is the low $T_g$ of PEG (~23°C). The reduction in $T_g$ is also attributed to a decrease in $T_g$.

**Table 4:** Glass transition temperature for all samples.

| Sample       | $T_g$ (°C) |
|--------------|------------|
| EP           | 129.5      |
| EP-PEG       | 103.2      |
| EP-NS        | 124.7      |
| EP-PEG-NS    | 105.2      |

**Table 5:** The tan $\delta$ curve peak of the samples.

| Sample         | Temperature at peak (°C) | tan $\delta$ at peak |
|----------------|--------------------------|----------------------|
| EP             | 142.7                    | 0.7467               |
| EP-PEG         | 128.5                    | 0.4621               |
| EP-NS          | 143.9                    | 0.8721               |
| EP-PEG-NS      | 128.8                    | 0.5004               |

**Table 6:** The effect of temperature elevation on the viscosity of samples.

| Sample         | 25°C   | 50°C   | 60°C   | 65°C   |
|----------------|--------|--------|--------|--------|
| EP             | 26800  | 1940   | 574    | 450    |
| EP-PEG         | 36522  | 1696   | 615    | 530    |
| EP-NS          | 73400  | 1280   | 530    | 370    |
| EP-PEG-NS      | 76350  | 7680   | 3600   | 2915   |
in the rigidity of the polymer chains and a drop in the density of crosslinks due to the presence of flexible PEG chains. The highest $E'$ belonged to EP-NS, EP-PEG-NS, and EP-PEG, respectively. As can be seen, the presence of flexible chains in PEG has strongly affected the modulus of samples and the mobility of chains.

Figure 3(b) shows variations of $\tan \delta$ versus temperature. The $\tan \delta$ indicates which sample is better at absorbing energy. The highest $\tan \delta$ curve peak was observed in EP-NS. This suggests that this sample has the maximum extent

| Sample  | $T_{\text{peak}}$ (°C) | $\Delta H$ (J/g) |
|---------|------------------------|-----------------|
| EP      | 151.3                  | 382.1           |
| EP-PEG  | 152                    | 341             |
| EP-NS   | 153.5                  | 414.7           |
| EP-PEG-NS | 162              | 366.3           |
of energy loss. Table 5 reports the tan δ curve peak of the samples. Adding NS transfers tan δ curve peak to higher temperatures while adding PEG transfers tan δ curve peak to lower temperatures. The wider the area under the tan δ curve, the greater toughness is. The EP-PEG has a wider area under curve compared EP-NS. This indicates that EP-PEG has greater fracture energy than EP-NS.

3.3. Viscometry Test. Table 6 shows the effect of temperature rise on the viscosity of samples. Viscosity-temperature diagrams are also displayed in Figure 4. EP-PEG-NS had a higher viscosity over the entire range of 25 to 65 °C compared to the other samples. This indicates that the viscosity increased with the presence of both PEG and NS and it prevented the deposition of solid particles. This is attributed to the melting temperature of PEG (50-58 °C). At this temperature, PEG has a viscosity of 70-80 cp. After the melting of PEG, OH groups of the PEG react with O groups of the NS (Figure 5), and by forming active groups on the surface of NS, it results in the formation of agglomerations thereby enhancing viscosity.

3.4. DSC Analysis. Figure 6 depicts the heating flow rates versus temperature for all samples at heating rates of 10 °C/min. The results of the DSC test are presented in Table 7. As can be seen, adding NS and PEG raises the maximum cure temperature. The heat of cure reaction (the area under the exothermic peak, ΔH) increases in EP-NS and diminishes in EP-PEG and EP-PEG-NS (more reduction in EP-PEG).

Figure 7 displays the DSC scan of PEG. As can be seen, within the temperature range of 60 to 70 °C, it indicates a melting peak. The DSC scan of the cured EP-PEG-NS is also shown in Figure 8 which is used to check for complete curing of the sample. As it is seen, there is no melting peak suggesting the complete curing of the EP-PEG-NS. The T_g obtained from DSC is about 86.6 °C, which differs by about 18 °C from the T_g obtained from DMTA.

3.5. Investigating of Curing Kinetics of EP-MEG-NS. In order to investigate the dynamic cure kinetics of the EP-PEG-NS, first, the DSC test was performed on the uncured sample at heating rates of 2.5, 5, and 10 °C/min. Figure 9 presents the heating flow rates versus temperature and time for uncured EP-PEG-NS. As expected, as the heating rate increased, the exothermic peak shifted to higher temperatures.

The degree of conversion (α) versus temperature and cure rate (da/dt) versus the degree of conversion at heating
rates of 2.5, 5, and 10°C/min is shown in Figures 10 and 11, respectively. As can be seen in Figures 9 and 11, the curing process has occurred under two reactions: kinetic control and penetration control. First, the curing process is the kinetic control, and the graph reaches the peak at a considerable speed. The reason is that as the reaction progresses, more crosslinks are formed between the polymer chains and its movement has become more difficult. Hence, the contribution of the penetration control is more than that of the kinetic control at higher curing degrees.

The variations of $E_a$ at different $\alpha$ values were also calculated using KAS equation (equation (4)). Table 8 shows $E_a$ at
Figure 11: The cure rate versus degree of conversion for uncured EP-PEG-NS.

Table 8: \( E_a \) at different conversions of EP-PEG-NS.

| Conversion | 10%   | 20%   | 30%   | 40%   | 50%   | 60%   | 70%   | 80%   | 90%   | Average |
|------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|---------|
| \( E_a \) (kJ/mol) | 92.53 | 89.38 | 87.45 | 90.81 | 93.98 | 94.85 | 97.18 | 102.47| 98.65 | 94.14   |

Figure 12: The plots obtained using (a) Kissinger and (b) Ozawa methods.
different conversions of EP-PEG-NS. As can be seen, the apparent \( E_a \) was approximately constant within the 10-90% conversion range.

\( E_a \) of the curing reaction was calculated using Kissinger and Ozawa methods with the peak temperature determined from DSC curves. In the Kissinger method, \( E_a \) was found to be 96.82 kJ/mol. \( E_a \) calculated from the Ozawa method (98.69 kJ/mol) was found to be higher than that of the Kissinger method. Singh et al. [45] also reported similar results for EP-based composites. Figure 12 indicates the plots obtained using Kissinger and Ozawa methods.

4. Conclusions

In the present paper, physical-mechanical and cure kinetics of blends of EP/DICY with PEG and NS were studied through tensile and viscometry test, DMTA, and DSC. By adding NS and PEG, the maximum cure temperature as well as the heat of cure reaction (\( \Delta H \)) increased in EP-NS, while it diminished in EP-PEG and EP-PEG-NS. \( E_a \) calculated from the Kissinger method (96.82 kJ/mol) was found to be less than that of the Ozawa method (98.69 kJ/mol). Also, according to the KAS method, the apparent \( E_a \) was approximately constant within the 10-90% conversion range. The results of mechanical tests revealed that tensile strength and modulus increased by adding NS, while the tensile strength dropped slightly by adding PEG to EP-NS. The glass transition temperature (\( T_g \)) was calculated using DMTA; according to the results, NS and PEG led to heightened and diminished \( T_g \), respectively. Also, the viscosity increased with the presence of both PEG and NS, and it prevented the deposition of solid particles.

Data Availability

The data used to support the findings of this study are currently under embargo while the research findings are commercialized. Requests for data, 12 months after publication of this article, will be considered by the corresponding author.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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