PREPARATION AND CHARACTERIZATION OF EPOXY RESIN WITH LOW AMOUNT OF POLYETHERIMIDE

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ABSTRACT

Diglycidyl ether of bisphenol A (DGEBA) was blended by adding polyetherimide (PEI) as thermoplastic for the mechanical properties and thermal stability with different contents of PEI. The mechanical behaviors were conducted using a universal test machine (UTM) and investigated with the thermogravimetric analysis (TGA) for the thermal stability. The mechanical properties were determined by measuring flexural strength and fracture toughness of the prepared blends. The values were enhanced as PEI was put into the epoxy resin up to 2 wt.%. In addition, the TGA results indicated that the thermal stability of the prepared blends was enhanced upon the increasing PEI. The values were displayed as the integral procedural decomposition temperature (IPDT) and the activation energy (E_t). The prepared blends containing PEI were improved than neat DGEBA in the IPDT and the E_t. Therefore, PEI-modified epoxy resin was increased by extending PEI content for thermal stability and improved up to 2 wt.% PEI for flexibility and toughness in this work.

1 INTRODUCTION

Epoxy resin is extensively used as matrices of thermosetting polymers in various applications such as advanced composites, adhesive, coating, electrical materials, and aerospace due to good chemical resistance, low shrinkage, excellent mechanical properties, and high thermal stability. Accordingly, epoxy resin was good for high performance materials. However, epoxy resin was limited for the engineering applications because they have poor crack resistance and inherent brittleness as the high cross-linking density for curing [1, 2]. To overcome these problems, many additives were used for several years and they enhanced the fracture toughness and strength in the modified epoxy resin. In addition, several studies have demonstrated that polymeric thermoplastics such as polyetherimide (PEI), polyethersulfone (PES), polycarbonate (PC), and polyetheretherketone (PEEK) were blended to epoxy resin for high performance and to improve compatibility with polymer matrices [3, 4]. Among them, PEI as modifier of epoxy resin was known for engineering thermoplastic with a wide range of processing than other polyetherimides that were difficult to handle the temperature and viscosity. Furthermore, PEI was used to enhance the interfacial adhesion between two phases by forming the strong network for increasing mechanical properties and thermal stability of the final blends without significantly decreasing the modulus or glass transition temperature (T_g) and resistance to most of the solvent, radiation, etc [5, 6]. In this study, PEI was added into epoxy resin to confirm the effect of mechanical properties and thermal stability. Furthermore, we changed it with low contents of PEI and investigated properties of the prepared blends compared to neat epoxy.

2 EXPERIMENTAL METHOD

2.1 Preparation of DGEBA/PEI blends

The diglycidyl ether of bisphenol A (DGEBA) and PEI were prepared at a weight ratio of 100:0 to 96:4. PEI was dissolved in methylene chloride by stirring at room temperature for 4 h. DGEBA was mixed with the PEI at 100 ºC for 12 h and the mixture was placed into a vacuum oven to evaporate the solvent at for 12 h. After that, the mixture was blended by stirring at 60 ºC slowly in order to remove bubbles and the solvent, thoroughly. 4, 4'-diaminodiphenylmethane as the curing agent was added into the mixture and then the final mixture was injected into a mold. The obtained blends were conducted in convection oven for curing at 110 ºC for 1 h, 140 ºC for 2 h, and 170 ºC for 1h.

2.2 Characterization

The flexural tests were performed according to ASTM D790 under a three-point bending test on a universal test machine (UTM). In addition, the critical stress intensity factor (K_{IC}) and the critical strain energy release rate (G_{IC}) of the prepared samples were conducted on the UTM according to the ASTM E399 including single edge notch (SEN) and the cross-head speed was 10 mm min^{-1}. The fracture surfaces were observed using a scanning electron microscope (SEM) to investigate the morphology of the blends after performing the fracture toughness tests. These were conducted for mechanical behaviors. The thermal stability of the prepared blends was analysed using a thermogravimetric analyser (TGA). The analysis was performed by increasing temperature from 50 ºC to 800 ºC at a heating rates of 10 ºC min^{-1} under a nitrogen condition.

3 RESULTS AND DISCUSSION

3.1 Mechanical properties

Figure 1 exhibits the flexural strength (σ_f) and elastic modulus (E_b) of the specimens and the values were calculated using the following equation.

$$\sigma_f = \frac{3PL}{2bd^2} \tag{1}$$

$$E_b = \frac{L^3}{4bd^3}\frac{\Delta P}{\Delta m} \tag{2}$$

where P is the applied load, L is the span length, b is the width of specimen, d is the thickness of the specimen, ΔP is the change in force in the linear portion of the load-deflection curve, and Δm is the change in deflection corresponding to ΔP.

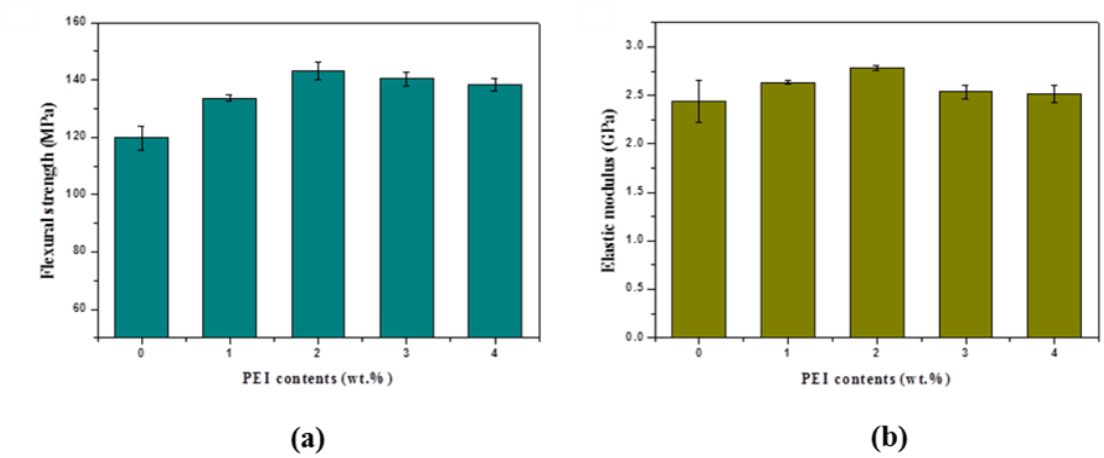

Figure 1: (a) The flexural strength (b) elastic modulus of the prepared specimens.

The values of flexural strength and elastic modulus of the prepared specimens were observed from 119 to 143 MPa and from 2.43 to 2.78 GPa, respectively. In addition, the maximum value was presented to 2 wt.% of the PEI content into the epoxy resin with improvements of 120.19 % in flexural strength and 114.40 % in elastic modulus than neat DGEBA.

Figure 2 shows the fracture toughness of the prepared blends by using K_{IC} and G_{IC} and the values were calculated using the following equation.

$$K_{IC} = \frac{P \cdot L}{bd^{3/2}} Y \tag{3}$$

$$Y = \frac{3a/d^{1/2}[1.99 - (a/d)(1 - a/d)(2.15 - 3.93a/d + (2.7a^2)/b^2)]}{2(1 + 2a/d)(1 - a/d)^{3/2}} \tag{4}$$

$$G_{IC} = \frac{(1 - v^2) \cdot K_{IC}^2}{E} \tag{5}$$

where P is the critical load for crack propagation, L is the length of the span (mm), Y is the geometrical factor, b is the specimen width (mm), d is the specimen thickness (mm), a is the pre-crack length (mm), v is the Poisson's ratio taken to be 0.3 and E is the tensile modulus obtained from fracture testing.

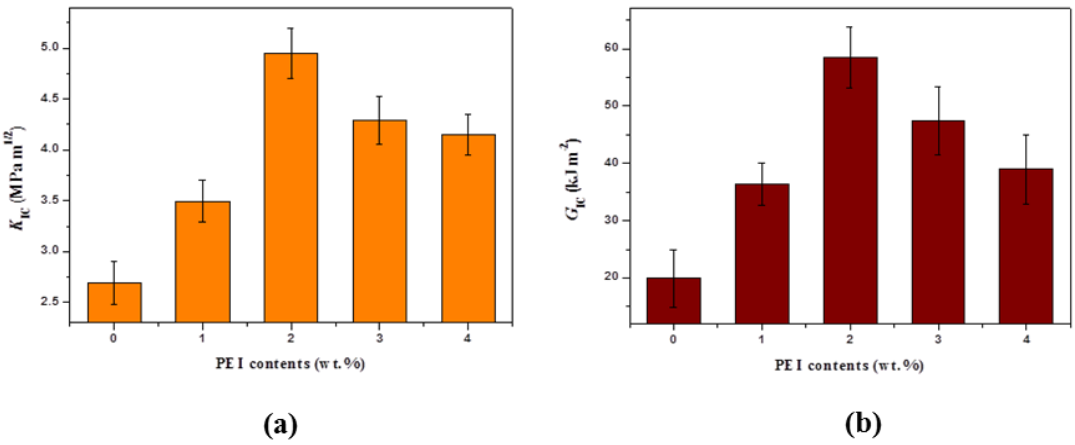

Figure 2: The fracture toughness parameter (a) K_{IC} (b) G_{IC} of the prepared specimens.

The values of K_{IC} were observed from 2.69 to 4.94 MPa $m^{1/2}$ and from 19.89 to 58.49 kJ m^2 in G_{IC}. Furthermore, the values of two parameters were with similar trends and increased by extending PEI contents than neat DGEBA up to 2 wt.% PEI. These enhancements presented 183.64 % in K_{IC} and 294.06 % in G_{IC} compared with neat DGEBA. However, it tended to decrease more than 2 wt.% PEI content because of the declined ability in the interpenetrating polymer networks (IPN) induced phase separation.

The morphologies on fractured surfaces of neat DGEBA and the blends with 2 wt.% PEI in epoxy resin after the fracture tests were shown in Figure 3. The SEM micrograph of neat DGEBA and the blends including 2 wt.% PEI was indicated with cracks in the fracture surface. The PEI-modified DGEBA resin was described with increasing cracks and rough surface than neat DGEBA. Consequently, the fracture surface of the prepared blends was observed with a lot of the crack than the

epoxy resin and the cracks split into some branches to avoid progressing through the dispersed PEI particles.

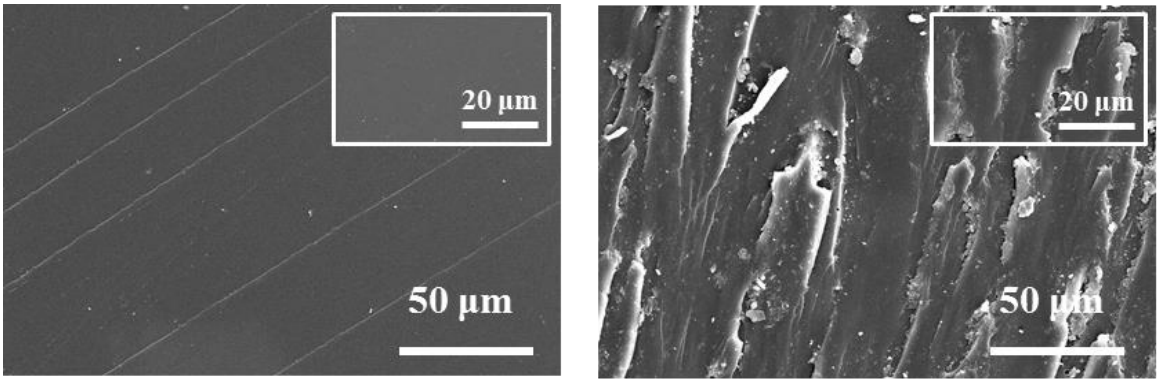

Figure 3: SEM images of fracture surfaces: (a) neat DGEBA and (b) 2 wt.% PEI blends.

3.2 Thermal stability

Figure 4 shows the thermogram of the prepared blends for the thermal stability. The degradation curve of the blends was occurred between 360 ºC and 420 ºC and the thermal stability factors were presented by using the polymer decomposition temperature (PDT) and the integral procedural decomposition temperature (IPDT). The IPDT was proposed by Doyle correlates the volatile parts of the polymeric materials and calculated using the following equations:

$$IPDT = A^* \cdot K^* \cdot (T_f - T_i) + T_i \tag{6}$$

$$A^* = \frac{(S_1 + S_2)}{(S_1 + S_2 + S_3)} \tag{7}$$

$$K^* = \frac{(S_1 + S_2)}{S_1} \tag{8}$$

where A^* is the area ratio of the total experimental curve defined by the total TGA thermogram, K^* is the coefficient of A^*, T_i is the initial experimental temperature, T_f is the final experimental temperature. S_1, S_2, and S_3 are the areas of the three regions in which the TGA curve is divided as shown in Figure 5.

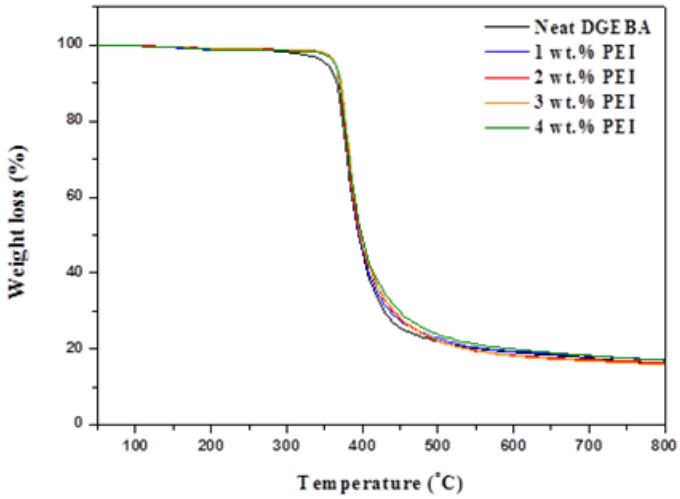

Figure 4: TGA thermograms of the prepared specimens.

The values of PDT in the prepared specimens were indicated with similar results without significant change because the low amount of PEI was added into epoxy resin. However, the values of IPDT were increased from 399.52 to 410.74 °C as increase of PEI contents.

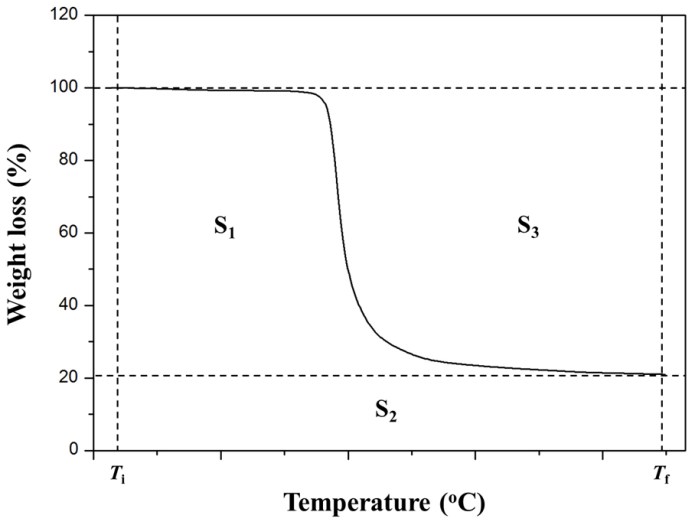

Figure 5: Schematic representation of S_1, S_2, and S_3.

9 CONCLUSIONS

In this study, we observed mechanical properties and thermal stability of neat DGEBA and PEI-modified epoxy . In addition, we discovered that a small amount of PEI as thermoplastic for reinforcement of epoxy resin was effective in epoxy system. In case of mechanical properties, the prepared blends were strengthened than neat DGEBA up to including 2 wt.% PEI content. However, the thermal stability was increased with extending PEI contents.

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