Effect of Reactive and Non-Reactive Diluent on Mechanical Properties of Epoxy Resin

Reaktif ve Reaktif Olmayan Seyrelticisi İlavesinin Epoksinin Mekanik Özelliklerine Etkisi

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ABSTRACT

Epoxy resins, which are frequently preferred among thermoset plastics in the industry, are materials with high viscosity, and good mechanical performance. The biggest disadvantage of epoxy resins is that they encounter difficulties in application alone due to their high viscosity. For this reason, various diluents are added to reduce their viscosity and applications are conducted. In this study, the mechanical properties of the epoxy resin, which is used in many fields, were observed with the addition of certain sizes of reactive and non-reactive diluents. Diglycidyl ether was used as the reactive diluents, and benzyl alcohol was used as the non-reactive diluent. In addition, ethylene diamine and diethylene triamine were used as curing agents. It was tried to determine the optimum modification of the hardened epoxy resin by measuring the mechanical strengths in the electromechanical test device. The results indicated the optimum composition that does not dramatically affect the mechanical performance of the epoxy resin is the composition containing 7.5% DE and 10% EDA, its tensile strength measured as 53.27 N/mm².

Keywords - Epoxy Resin, Viscosity, Mechanical Properties, Reactive and Non-Reactive Diluent

ÖZ

Endüstride termoset plastikler arasında sıkılık tercih edilen epoksili reçineler yüksek viskoziteye sahip, mekanik performansları oldukça iyi olan malzemelerdendir. Epoksili reçinelerinin en büyük dezavantajı, yüksek viskoziteleri sebebiyle tek başlarına uygulamada zorluklar ile karşılaşılmasınıdır. Bu nedenle, viskozitelerini düşürmek amacıyla çeşitli diluentler eklenerek uygulamaları yapılmaktadır. Bu çalışma kapsamında birçoğlu alanda kullanılan epoksili reçinelerin, belirli ölçüde reaktif ve reaktif olmayan seyreltiği ilavesiyle mekanik özelliğindeki değişimler gözlandı. Çalışmada reaktif diluent olarak diglisidil eter, non-reaktif diluent olarak ise benzil alkol kullanıldı. Kürleme ajanı olarak ise etilen diamin ve diyetilen triamin kullanıldı. Sertleştirilmiş epoksili reçininin elektromekanik test cihazında mekanik davranışları ölçülecek optimum modifikasyonu belirlenmeye çalışıldı. Elde edilen sonuçlar, epoksili reçinelerin mekanik performansını önemli ölçüde etkilemektedir. optimum bileşimin %7.5 DE ve %10 EDA içeren bileşiminin 53.27 N/mm² olarak ölçüldüğü gösterildi.

Anahtar Kelimeler - Epoksi Reçinesi, Viskozite, Mekanik Özellik, Reaktif ve Reaktif Olmayan Seyrelticili
I. INTRODUCTION

Diglycidyl ether of bisphenol (DGEBA) A is the most used epoxy and its viscosity varies depending on the number of repeating units (n) and molecular weight (Figure 1).

![Molecular structure of epoxy resin (DGEBA)](image)

Figure 1. Molecular structure of epoxy resin (DGEBA)

While the use of epoxy resins as binders for many materials is the biggest advantage, their high viscosity is their biggest disadvantage. As such, it represents a promising field of material research. It is known that the addition of reactive or non-reactive diluents in certain proportions by weight reduces the strength of binders relatively, and thus, a certain reduction in the strength of the cured product is observed by further increasing the diluent content [1-5]. Great emphasis is placed on the use of reactive diluents and modifiers that cause effective changes in the polymerization step as well as reduce viscosity effectively [1, 6]. Study of these diluents is necessary because of both practical problems and very limited knowledge of the effect of diluent addition on the chemical and physical properties of epoxy polymers [7]. On the other hand, these diluents cost much more than non-reactive diluents. Therefore, it is useful to perform an optimum study in which reactive and non-reactive diluents coexist.

In this study, the epoxy resin synthesized in the laboratory (E\_w = 195 g/mol), glycidyl ether as the reactive diluent, benzyl alcohol as the non-reactive diluent and ethylene diamine and diethylene triamine as curing agents were used (Figure 2).

![Molecular structure of glycidyl ether, benzyl alcohol, ethylene diamine and diethylene triamine](image)

Figure 2. Molecular structure of glycidyl ether, benzyl alcohol, ethylene diamine and diethylene triamine

II. EXPERIMENTAL METHOD

The epoxy resin used in this study is diglycidyl ether of bisphenol A (DGEBA) was synthesized as literature procedure [8]. The glycidyl ether, benzyl alcohol, ethylene diamine, and diethylene triamine are the most widely used diluents, and hardener were purchased from sigma and used without purification. The amounts of hardeners are determined from the stoichiometry calculations of DGEBA and amines. The amounts of hardener and diluents using in this study are listed in Table 1. All samples created by mixing at the rates listed in Table 1 were poured onto silicone molds. Samples were kept at room temperature during gelling. The samples that started to gel, the samples placed in the oven set at 70 °C were kept here for 24 hours. Mechanical properties of epoxy samples were examined by using tensile tester (Shimadzu AGS-X Series), elastic modulus and percent (%) elongation values were determined. Tensile tests 2 mm / min. It was carried out at room temperature at drawing speed. In addition, the viscosities of the resin and diluent mixtures were measured using a Brookfield viscometer.
### Table 1. The amounts of hardener and diluents for each sample

| Samples | Diglycidyl ether (DE) | Benzyl alcohol (BA) | Ethylene diamine (EDA) | Diethylene triamine (DETA) |
|---------|-----------------------|---------------------|------------------------|---------------------------|
| 1       | -                     | -                   | 10.0                   | -                         |
| 2       | 10.0                  | -                   | 10.0                   | -                         |
| 3       | -                     | 10.0                | 10.0                   | -                         |
| 4       | 10.0                  | 10.0                | 10.0                   | -                         |
| 5       | -                     | -                   | -                      | 10.0                      |
| 6       | 10.0                  | -                   | -                      | 10.0                      |
| 7       | -                     | 10.0                | -                      | 10.0                      |
| 8       | 10.0                  | 10.0                | -                      | 10.0                      |
| 9       | 5.0                   | -                   | 10.0                   | -                         |
| 10      | -                     | 5.0                 | 10.0                   | -                         |
| 11      | 5.0                   | 5.0                 | 10.0                   | -                         |
| 12      | 5.0                   | -                   | -                      | 10.0                      |
| 13      | -                     | 5.0                 | -                      | 10.0                      |
| 14      | 5.0                   | 5.0                 | -                      | 10.0                      |
| 15      | 2.5                   | -                   | 10.0                   | -                         |
| 16      | -                     | 2.5                 | 10.0                   | -                         |
| 17      | 2.5                   | 2.5                 | 10.0                   | -                         |
| 18      | 2.5                   | -                   | -                      | 10.0                      |
| 19      | -                     | 2.5                 | -                      | 10.0                      |
| 20      | 2.5                   | 2.5                 | -                      | 10.0                      |
| 21      | 7.5                   | -                   | 10.0                   | -                         |
| 22      | -                     | 7.5                 | 10.0                   | -                         |
| 23      | 7.5                   | 7.5                 | 10.0                   | -                         |
| 24      | 7.5                   | -                   | -                      | 10.0                      |
| 25      | -                     | 7.5                 | -                      | 10.0                      |
| 26      | 7.5                   | 7.5                 | -                      | 10.0                      |

### III. RESULTS AND DISCUSSION

Epoxy resin was synthesized as written in the literature [8], and characterized by FTIR and NMR measurements. The viscosity of the synthesized DGEBA resin was measured 16200 cp. In addition, the equivalent epoxide weight (EEW) value of the synthesized epoxy was calculated as 206 g / equiv from the NMR spectrum. Dilution was applied to make this resin applicable in industry. Twelve different epoxy mixtures were obtained by adding diluents in different proportions. The viscosity variations of DGEBA resin containing different amounts of the diluents, diglycidyl ether and benzyl alcohol are shown in Figure 3. With the addition of diluent, viscosities drop as expected. The highest viscosity is observed in pure epoxy resin. In addition, the viscosity value obtained by adding the same proportion of benzyl alcohol was measured lower than the addition of diglycidyl ether [9].

![Figure 3. Variation of viscosity of resin with diluents rate](https://dergipark.org.tr/tr/pub/bseufbd)
The mechanical properties of DGEBA resins (i.e. tensile strength $\sigma$, elastic module $E$ and strain at break $\varepsilon$) were determined from tensile tests. The tensile stress-strain curves of pure DGEBA resin cured with amines, EDA (1) and DETA (5) were given in Figure 4. In addition, the mechanical properties of the 26 samples obtained by adding diluents in different ratios are listed in Table 2. The tensile stress-strains of pure DGEBA, 1 and 5 were measured at 68.20 and 60.75 N/mm$^2$, respectively. In general, the addition of diluents reduced both tensile strength $\sigma$ and elastic module $E$.

![Stress-strain curves of the pure epoxy resin with curing (a) EDA (b) DETA](image)

**Figure 4.** Stress-strain curves of the pure epoxy resin with curing (a) EDA (b) DETA

| Samples | Tensile strength, $\sigma$ (N/mm$^2$) | Strain at break, $\varepsilon$ (%) | Elastic module, $E$ (N/mm$^2$) |
|---------|-----------------------------------|-----------------------------------|---------------------------------|
| 1       | 68.20                             | 5.68                              | 1974.14                         |
| 2       | 52.71                             | 4.57                              | 1912.27                         |
| 3       | 47.97                             | 5.75                              | 1743.89                         |
| 4       | 14.46                             | 20.28                             | 880.68                          |
| 5       | 60.75                             | 4.34                              | 2046.97                         |
| 6       | 46.47                             | 5.42                              | 1617.36                         |
| 7       | 37.87                             | 5.93                              | 1781.31                         |
| 8       | 25.46                             | 11.79                             | 1080.16                         |
| 9       | 53.27                             | 7.19                              | 1787.13                         |
| 10      | 49.22                             | 5.72                              | 1916.44                         |
| 11      | 41.48                             | 4.67                              | 1850.53                         |
| 12      | 51.29                             | 3.52                              | 1937.26                         |
| 13      | 50.26                             | 4.12                              | 2088.49                         |
| 14      | 49.56                             | 5.50                              | 1787.62                         |
| 15      | 60.67                             | 5.35                              | 1852.99                         |
| 16      | 48.59                             | 4.42                              | 1910.55                         |
| 17      | 50.83                             | 4.26                              | 1946.28                         |
| 18      | 59.31                             | 6.13                              | 1846.11                         |
| 19      | 48.45                             | 4.37                              | 1817.69                         |
| 20      | 55.30                             | 6.46                              | 1889.37                         |
| 21      | 53.36                             | 4.44                              | 1938.07                         |
| 22      | 38.25                             | 4.42                              | 1817.02                         |
| 23      | 20.74                             | 7.64                              | 1348.69                         |
| 24      | 48.00                             | 3.91                              | 1818.94                         |
| 25      | 26.72                             | 2.01                              | 1764.20                         |
| 26      | 32.26                             | 5.09                              | 1583.01                         |
The tensile stresses of different samples are compared in Figure 5 (a) and (b). Figure 5 (a) shows the variation of tensile stress with the addition of 10% DE, BA, and both. With the addition of 10% DE and BA, 52.71 and 47.97 N / mm² were measured, respectively. It was observed that the addition of BA decreased the stress more than the addition of DE. With the addition of both diluents the value dropped to 14.46 N / mm². When Figure 5 (b) is examined, it is seen that the tensile stress of the samples with more diluent added by mass decreases. When the graph is examined, the highest tensile stress was observed with 60.67 N / mm² in the sample 5. The difference between samples 1 and 5 is due to the curing agent. The samples 15, 9, 21 and 2 are samples diluted with 2.5, 5, 7.5 and 10% DE, respectively, and the tensile stresses of these samples were measured as 60.67, 53.27, 53.36 and 52.71 N / mm², respectively. According to these results, with the use of both reactive and non-reactive diluents, the tensile stress decreases with increasing the number of diluents [10].

![Figure 5](image_url)

**Figure 5.** Stress-strain of (a) sample 1, 2, 3 and 4, (b) sample 2, 6, 9, 12, 15, 18, 21 and 24

**IV. CONCLUSIONS**

Epoxy resins are in the family of high viscosity thermoset plastics. Various diluents are added to reduce their viscosity in industry, as their application alone creates difficulties due to their high viscosity. In this study, by adding reactive and non-reactive diluents to epoxy resin, conditions providing optimum mechanical performance that would not critically affect the strength performance were determined. For this purpose, experiments were carried out in different proportions by mass based on the amount of epoxy resin using DE as a reagent and BA as a non-reactive. Considering the test results obtained as a result of the studies under the name of modification of epoxy resin, it was seen that using EDA as a hardener increased the mechanical performance. The
use of BA as a diluent also reduces the mechanical performance compared to the use of DE. Since there are epoxy groups in DE, the amount of hardener added is added according to the amount of epoxy resin and DE in the mixture. Therefore, the number of epoxy groups in the mixture increases, and the amine groups in the hardener are cross-linked with more epoxy groups. This affects the mechanical performance positively. The results show that the optimum composition that does not dramatically affect the mechanical performance of the epoxy resin is the composition containing 7.5% DE and 10% EDA. In some applications in the industry, there is a need for thermoset plastics that provide satisfactory results in terms of both flexible and mechanical performance. According to the mechanical test results, using DE and BA together reduces the mechanical performance of the epoxy sample but increases the elongation performance. When the trials where both diluents were used together are compared, it is seen that the addition of 10% DGE and 10% BA increases the elongation performance critically.

When this mixture is cured with EDA, the elongation performance increases, but its mechanical performance drops critically. When the same mixture is cured with DETA, optimum mechanical performance and elongation performance is achieved. The composition containing 10% DE, 10% BA and 10% DETA meets the desired flexibility and mechanical performance when high flexibility performance and moderate mechanical performance are required from epoxy resin.

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