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Synthesis and Investigation of Thermal and Dynamic Mechanical Properties of Urethane-Containing Epoxy Resins

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Abstract: This study aims to improve the thermal and mechanical properties of epoxy-based materials. For this purpose, the structure of epoxy resins was changed by chemical modification and epoxy resin containing urethane was synthesized. The synthesized resin was blended with commercial epoxy resin at the ratios of 25%, 50%, 75% by weight and hardened by curing. The thermal and mechanical properties of urethane-containing epoxy materials prepared in different proportions were compared with those produced from commercial epoxy resin. The structural characterization of the prepared materials was investigated by Fourier Transform Infrared Spectroscopy (FTIR) analysis, their thermal behavior was investigated by Thermogravimetric Analysis (TGA), and their mechanical properties were investigated by Dynamic Mechanical Analysis (DMA). TGA and DMA analyses of the materials showed that the presence of urethane in the structure of epoxy resins significantly changed the mechanical and thermal properties. It was observed that the storage and loss modulus values of urethane-containing resins increased approximately 2.5 times compared to commercial epoxy resins, and a decrease of approximately 10% in thermal degradation temperatures was observed.

Keywords: Epoxy, Urethane, FTIR, TGA, DMA.

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1. INTRODUCTION

Epoxy resins, an essential and widely used class of thermosetting polymers, are used in various fields such as electrical and structural applications, surface protective coating applications because they have desirable properties such as chemical resistance, mechanical and thermal properties, low shrinkage after curing, and insulation properties (DAĞ, 2023; Paul Swaraj, 1995). They contain one or more 1,2 oxirane groups in their structure and can be converted into thermoset form or three-dimensional network structure (Ellis, 1993; Stefani et al., 2001). Although the commercial production of epoxy resins began in the late 1930s, and there are different types of epoxy resins today, these materials still suffer from some disadvantages, such as considerable internal stress, brittleness, flammability, low degradability, little impact resistance, and high moisture absorption (Akovali, 2001; Ellis, 1993). In order to improve the toughness of epoxy resins, several techniques are employed, i.e., improvement of the flexibility of the cured epoxy resins (Jin et al., 2015; Jin & Park, 2008; May, 2018).

The importance of using epoxy resins in various fields is due to the easy conversion of this resin to high molecular weight materials by curing reactions. The prepolymers containing two epoxide end groups are cured with suitable curing agents to give cross-linked networks (Kricheldorf, Hans R.; Nuyken, Oskar; Swift, 2004). Increasing the toughness of epoxy resins by improving their flexibility may be done by two general procedures: I) Introduction of a toughening agent into the epoxy network or II) chemical modification of the epoxy structure. In the case of using toughening agents, liquid elastomers (Kunz & Beaumont, 1981; Levita et al., 1985), thermoplastics (Huang et al., 1997; Yang & Gu, 2009), interpenetrating polymer networks (Anand Prabu & Alagar, 2004; K. H. Hsieh et al., 2001; Prabu & Alagar, 2004), rigid phase (T. H. Hsieh et al., 2010), and inorganic particles, which may cause deterioration of the final properties, are commonly used (Demčenko et al., 2014; Jiang et al., 2012; Jin & Park, 2008). Thus, a chemical modification of the epoxy can improve the overall physical properties. Besides several chemical modifications of epoxy prepolymers with epoxy-terminated liquid polybutadiene (Barcia et al., 2003), polytriazolesulfone (Lee et al., 2019), carboxyl-terminated butadiene acrylonitrile copolymer and hydroxyl-terminated polybutadiene (Ramos et al., 2005), incorporation of a urethane group into the main chain, e.g. urethane coupling containing modified propylene carbonate (Wazarkar et al., 2016), isocyanate-capped polydimethylsiloxane (HTPDMS) prepolymer (Rath et al., 2009), polymethylene polyphenylene isocyanate (Chen et al., 2018), blocked isocyanate prepolymers (Kirillov, 2014) hyperbranched polyester-toluene diisocyanate (Dhevi et al., 2014) is easy and popular route to achieve enhanced physical properties (i.e. high flexibility, toughness).

In this study, a urethane-containing epoxy resin was successfully synthesized and blended with a commercial epoxy resin at 25, 50, and 75% ratios by weight and cured. The chemical, mechanical, and thermal properties of urethane-containing epoxy materials were investigated by Fourier Transform Infrared Spectroscopy (FTIR), dynamic mechanical analysis (DMA), thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC) analyses.

2. EXPERIMENTAL SECTION

2.1. Materials

Bisphenol A (BPA), toluene 2,4 diisocyanate (TDI), epichlorohydrin (ECH), Triethylenetetra amine (TETA), and diazobicyclooctane (DABCO) were purchased from Sigma Aldrich Sigma Aldrich Co., (USA). Commercial Epoxy Resin (CER) (epoxy equivalent weight 185–190 g/mol) was supplied from Dow Chemical Company (USA). The rest of the materials were of analytical grade and purchased from Merck, Germany.

2.2. Synthesis of Urethane Containing Epoxy Resin

BPA-based epoxy resin containing urethane bonds was synthesized in two steps. In the first step, an intermediate product was obtained by the reaction of BFA and TDI. The second stage was completed with the intermediate and ECH condensation reaction.

In the first step, 0.375 moles (85.5 g) of BFA and 75 mL of acetone were placed in a three-neck reactor. The BFA was mixed in a mechanical heater stirrer at 50 °C until dissolved. After the complete dissolution of the BFA, 0.093 mol (13.49 mL) of TDI was added to the reactor in the presence of 1.01 g of catalyst (DABCO). The reaction was carried out at 80 °C under reflux. FTIR samples were taken every 15 minutes of the reaction, and the presence of isocyanate in the medium was monitored. At the end of 1 hour, the reaction was terminated, and the acetone in the reaction medium was removed under vacuum with a rotary evaporator device. The obtained product was yellow and viscous liquid.

In the second stage, the intermediate product and ECH (5 eq) were loaded into the reactor, and 40% NaOH solution was added dropwise with the help of a dropping funnel while the temperature was increasing to 100 °C. The reaction was continued for 3 hours at this temperature. Afterwards, the product was cooled to room temperature, dissolved in xylene as 1:1 (v:v) and taken into a separator funnel. After 2-3 minutes, the water phase formed in the lower part was separated and discarded. Another 50 mL portion of distilled water was added to the xylene organic phase, and phases were separated again. This process was repeated for 3-4 times. Finally, the organic solvent phase was removed with a rotary evaporator and light yellow viscous "urethane-containing epoxy resin" (UER) was obtained. The schematic representation of the whole reaction is given in Figure 1.

2.3. Sample preparation and Characterization

2.3.1. Preparation of urethane-containing epoxy-based materials

The preparation of urethane-containing epoxy-based materials was carried out in two stages. In the first stage, the synthesized UER resi*n* and CER were blended in specific proportions by weight (25%; 25UER), 50%; 50UER, and 75%; 75UER). Then, according to epoxy equivalent weight (EEW), which was determined using the dioxane-hydrochloric acid method described elsewhere (Basnet et al., 2015), they were mixed with stoichiometric amounts of curing agent (TETA). In the second stage, the mixtures were poured into rectangular Teflon moulds of 10×10×0.5 mm and allowed to be cured at room temper*a*ture for 24 hours. Cured materials were post-cured to increase the cross-link density and complete the curing reactions. Post curing process was carried out by keeping the materials in an oven at 150 °C for 3 hours. In order to make a comparative interpretation, samples containing pure UER and CER were prepared by the same procedure. Their sample codes were given as UER and CER, respectively. In addition, “-RC” and “-C” abbreviations were added to the end of the room temperature cured and post-cured sample codes, respectively. Sample codes, their UER, CER contents, EEW values, and amounts of TETA added for curing are given in Table 1.

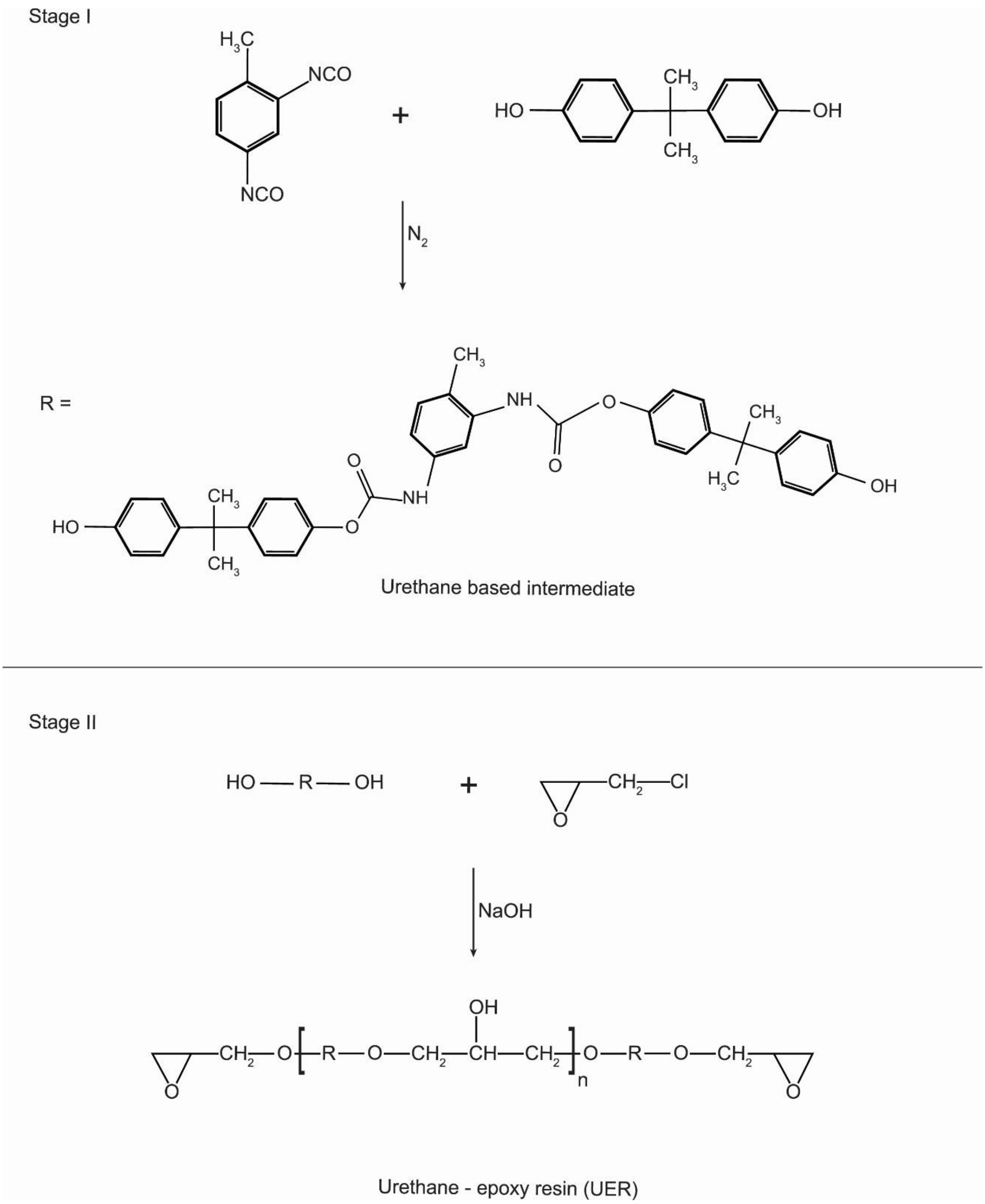

Figure 1: Schematic representation of the synthesis of UER.

Table 1: Sample contents and codes.

Resin Code	CER (g)	UER (g)	EEW	TETA(g)	Room temperature cured sample	Post-cured sample code
UER	--	3	442	0.58	UER-RC	UER-C
75UER	1.2	3.6	338	0.19	75UER-RC	75UER-C
50UER	2	2	272	0.40	50UER-RC	50UER-C
25UER	3	1	228	0.41	25UER-RC	25UER-C
CER	4	--	195	0.49	CER-RC	CER-C

2.3.2. Characterizations

The chemical structures of the synthesized products were elucidated with Agilent Technologies brand Cary 630 model Fourier Transform Infrared Spectrophotometer (FT-IR).

Thermal Gravimetric Analysis (TGA) Thermal analyses of the obtained products were carried out with Linsesis brand STA P 750 TGA model Thermal Gravimetric Analyzer. TGA studies were performed with approximately 20 mg samples under an air atmosphere by heating from room temperature to 800 °C with a heating rate of 10 °C/min. The temperatures at which the products first begin to decompose (IDT) and at which 5%, 10%, 25%, and 50% weight losses occur were determined.

Dynamic Mechanical Analysis (DMA) studies were performed with the Seiko brand Extar 6000 model instrument. For the tests, post-cured rectangular cross-section samples of 10x10x0.5 mm were used. DMA tests are performed under a nitrogen atmosphere at 1 Hz oscillation frequency, in the 20-150 °C temperature range with 3°C/min heating rate. The storage module and loss module of the prepared products were determined.

3. RESULTS AND DISCUSSION

3.1. FT-IR Analyses

The progress of the first step reaction in the synthesis of urethane-containing epoxy resins was followed by FTIR analyses. The presence of isocyanate groups was monitored by taking samples from the reaction medium every 15 minutes, and the obtained FT-IR spectra were given in Figure 2. As seen, the intensity of the characteristic peak of the isocyanate groups at 2270 cm^{-1} decreases over time. It disappears after 60 minutes, while a new peak is observed in the 1700 cm^{-1} region depending on the formation of the urethane bond (Socrates, 2001). These findings indicate that the intermediate, whose chemical structure is given in Figure 1, was successfully synthesized.

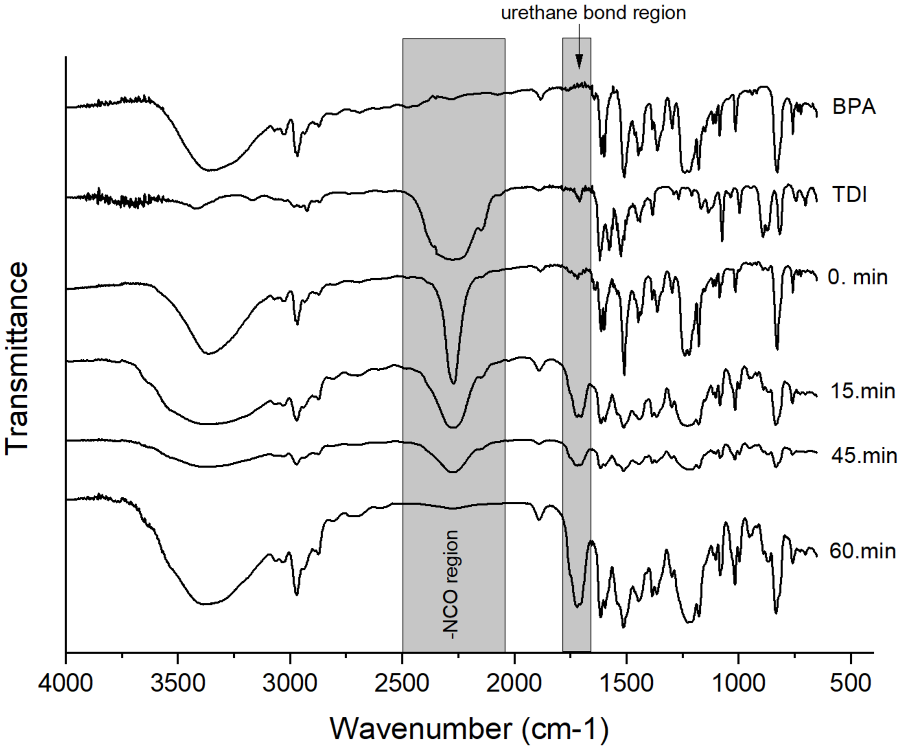

Figure 2: The variation of the isocyanate IR peak during the reaction and the FT-IR spectrum of the intermediate.

FTIR spectra of CER and synthesized UER are given in Figure 3. The peak at 912 cm^{-1} confirms the presence of the epoxide functionality in both resins. In addition, the peak at 1700 cm^{-1} region shows the urethane bond functionality in UER. Furthermore, in the spectrum of UER, the intensity of the widespread peak due to -OH bonds in the 3300 cm^{-1} region also increases. This interesting finding indicates that the molecular weight increases during the synthesis of urethane-epoxy resin. As a matter of fact, when the EEW values given in Table 1 are examined, it is seen that the EEW value of UER is 442, which indicates that the molecular weight of the resin is higher than that of CER. As the molecular weight of epoxy-based resins increases, the number of -OH groups in the structure increases due to the repeating unit. Thus, it is an expected result that the intensity of the -OH characteristic band increases in the FTIR spectrum. These findings indicate that the UER, whose chemical structure is given in Fig. 1, was successfully synthesized.

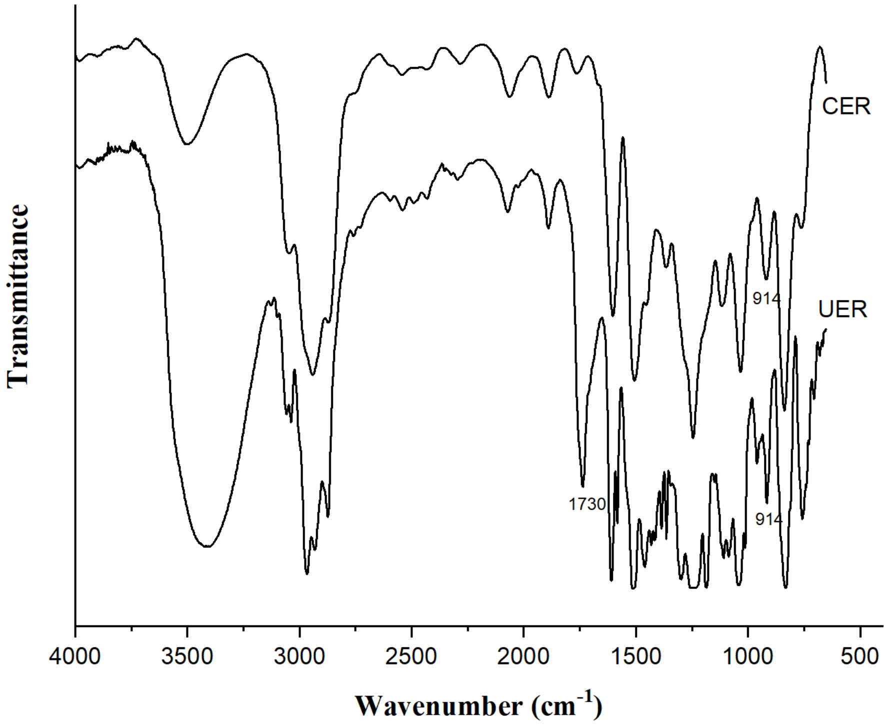

Figure 3: The FT-IR spectrum of commercial epoxy resin (CER) and urethane-epoxy resin (UER).

3.2. Thermal Analysis

In order to examine the thermal-oxidative degradation properties of the room-temperature cured and post-cured urethane-epoxy materials, TGA studies were performed, and the results were illustrated in Figure 4 and 5. In addition, the temperatures at which the products begin to decompose (initial decomposition temperature; IDT) and the temperatures at which 5%, 10%, 25%, and 50% weight losses occur were listed in Table 2 and 3.

Table 2: Thermal-oxidative degradation behavior of room temperature cured compositions.

Sample	IDT (°C)	$T_{(5)}$ (°C)	$T_{(10)}$ (°C)	$T_{(25)}$ (°C)	$T_{(50)}$ (°C)
UER	165	211	252	286	326
75UER	163	215	255	286	323
50UER	184	245	276	304	334
25UER	126	205	261	307	332
CER	196	289	319	339	366

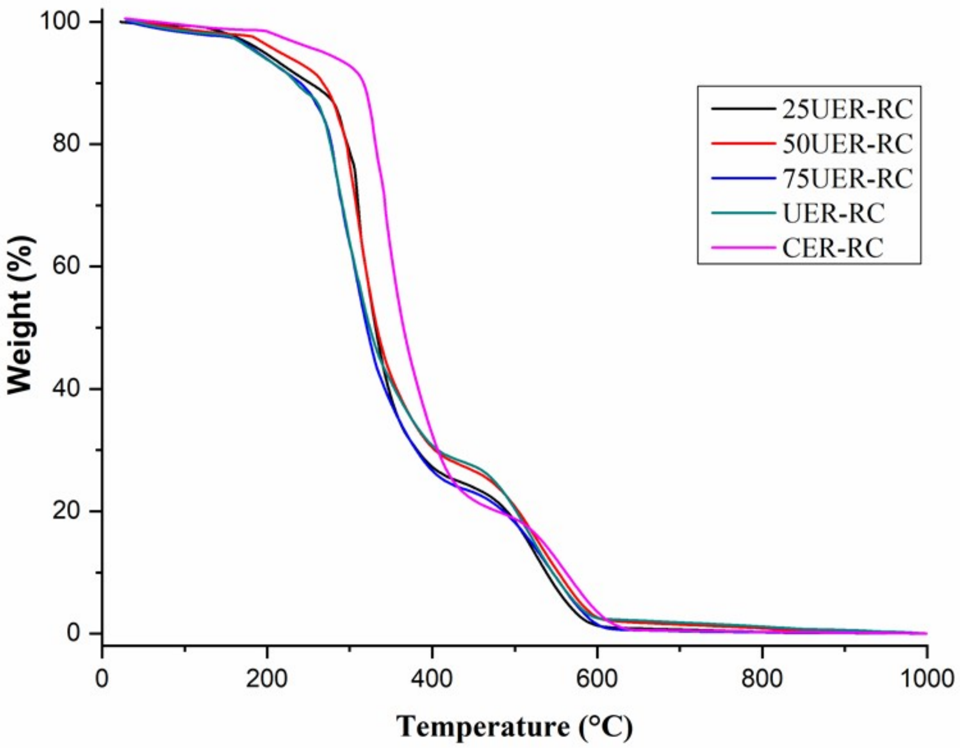

Figure 4: TGA curves of room temperature cured materials with different compositions.

According to the TGA results of the room temperature cured materials prepared in different compositions given in Figure 3 and Table 2, it is seen that the first weight losses of room temperature cured urethane-containing epoxy materials (UER-RC group) started at approximately 250 °C. The thermal behavior of CER-RC differs from other UER-RC groups. For example, while the first degradation temperature of CER-RC occurs at 196 °C, this value is determined as 126 °C in 25UER-RC. In addition, while 5% weight loss of CER-RC occurs at 289 °C, approximately 25% weight loss is observed in the UER-RC group at this temperature.

Table 3: Thermal-oxidative degradation behavior of post-cured compositions.

Sample	IDT (°C)	$T_{(5)}$ (°C)	$T_{(10)}$ (°C)	$T_{(25)}$ (°C)	$T_{(50)}$ (°C)
UER-RC	267	275	281	300	342
75UER-RC	235	258	274	293	332
50UER-RC	254	274	286	308	341
25UER-RC	257	281	291	312	337
CER-RC	310	323	330	347	381

TGA results of post-cured materials with different compositions are given in Figure 5 and Table 3. As it is seen, after the post-curing process, the IDT for CER-C is determined as 310 °C and a significant increase in thermal oxidative resistance is observed for all materials. In the case of post-cured urethane-containing epoxy materials (UER-C group), the IDT values increase and are around 250 °C for the whole UER-C group. These IDT values are 70-130 °C higher than their room temperature-cured counterparts. In conclusion, due to the increased amount of cross-linking by the post-curing process, significant increases are observed in the thermal stability of all materials. The temperature values at which 10% weight loss occurs, an essential value for commercial applications, are determined as 330 °C for CER-C and between 280-290 °C for the UER-C group.

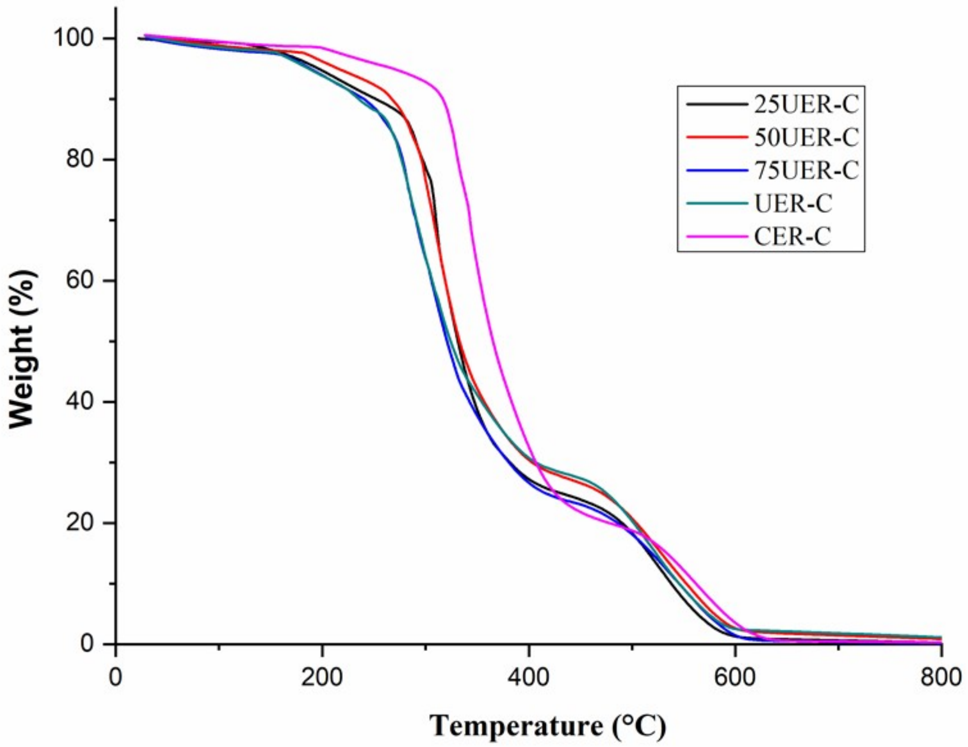

Figure 5: TGA curves of post-cured materials with different compositions.

3.3. Dynamic Mechanic Analysis

The dynamic mechanical analysis (DMA) test results of the epoxy-based materials, elastic modulus – temperature, storage modulus – temperature, and tan δ- temperature curves are shown in Figures 6-8, and the obtained data are listed in Table 4. In DMA curves, the response of a viscoelastic material to an applied force, energy, is divided into two terms: elastic response and viscose (dissipation) response. While the pure elastic response is considered a reversible deformation, the viscose is considered energy dissipation due to movement (segmental in solid-state), friction between molecules and the heat generated by such mechanisms. The calculated tan δ value is a very useful indicator for a material's glass transition temperature (T_g). In addition, its height indicates the material is viscose (large tan δ) or elastic (small tan δ) under the given condition.

As it is seen from Figures 6-8 and Table 4, the storage modulus value of CER-C is low, and a significant increase is observed when it is blended with urethane-containing epoxy resin. In the case of the UER-C group, it is determined that the storage modulus value of UER-C is approximately 2.5 times that of CER-C, and there is a significant increase in the values of other resins containing urethane in different ratios, 25, 50 and 75 UER-C.

When the lost modulus values are compared, a significant increase is observed in the modulus values of the UER-C group compared to the CER-C. This case is probably observed due to the presence of urethane in the structure of epoxy resins, increasing the viscoelastic properties of these materials.

When the tan δ temperature values of the prepared materials are compared, as the amount of resin containing urethane increases, it is seen that the tan δ temperature values shift to the higher region compared to the CER-C. This situation is interpreted as an increase in the α transition temperatures of the prepared materials, in other words, T_g, depending on the increase in the cross-link densities of the materials with the post-curing process. This change, which occurs due to the restriction of molecular mobility due to the increase in cross-link density, is an expected situation, and the observed temperature values are also consistent with previous similar studies (Garcia et al., 2007).

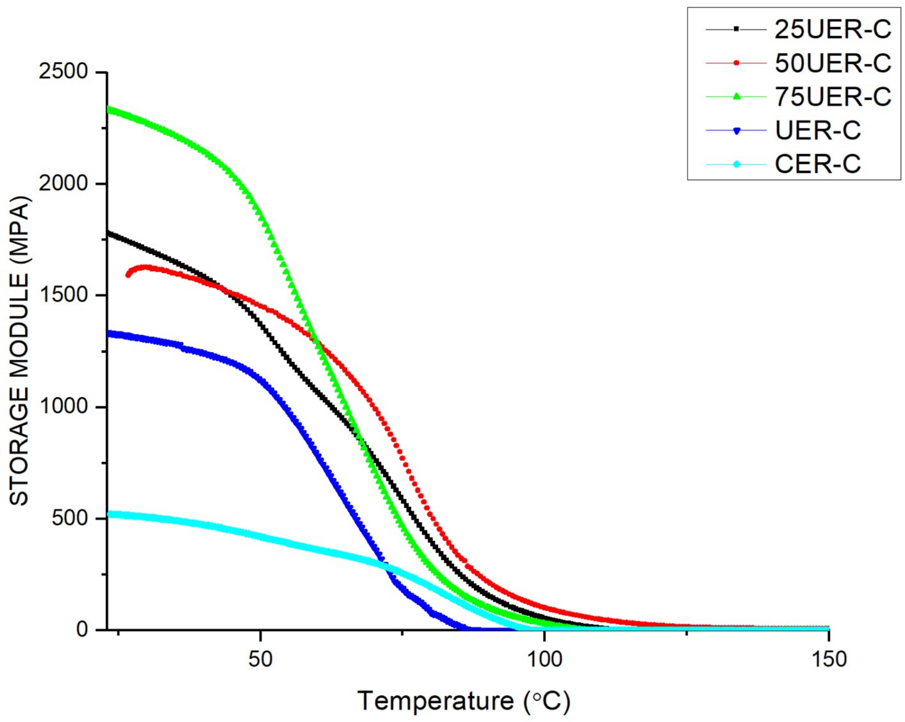

Figure 6: Store modulus-temperature curves of the post-cured materials with different compositions.

Table 4: Storage modulus, loss modulus, and tan δ values of post-cured materials.

Sample Code	**Storage (MPa)**	**modulus Loss modulus (MPa)**	**Tan δ temp.**
(°C)			
UER-C	1329	1412	90.5
75UER-C	2337	2188	110
50UER-C	1652	1483	113
25UER-C	1810	1474	117
CER-C	520	398	99

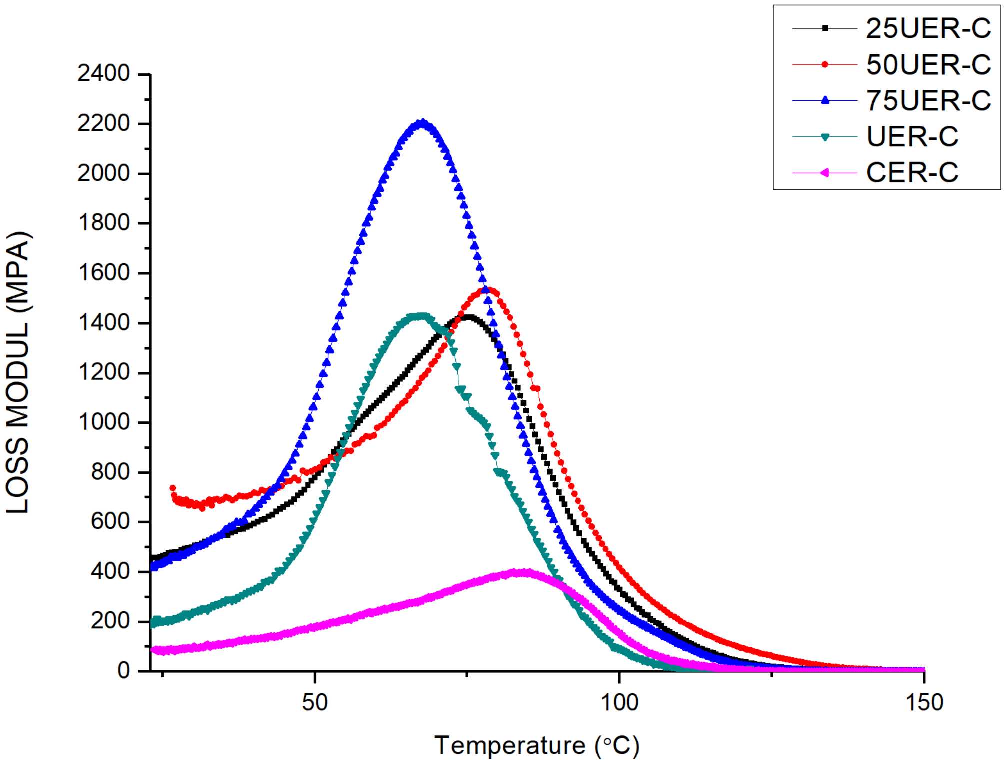

Figure 7: Loss modulus-temperature curves of the post-cured materials with different compositions.

Another interesting result determined in DMA studies is the intensity of the tan δ curve: All UER-C groups except for 25UER-C show a more intense peak than the CER-C without following any sequence. Since the peak intensity in the tan δ curves is a measure of the total amount of energy that a material can absorb, the large area (and/or the intense peak) under the tan δ curve indicates a significant degree of molecular mobility that translates to better-damping properties (Zhou et al., 2018). That means that the material can better absorb and dissipate energy. For this reason, it is concluded that the UER-C group have better damping properties than that of CER-C.

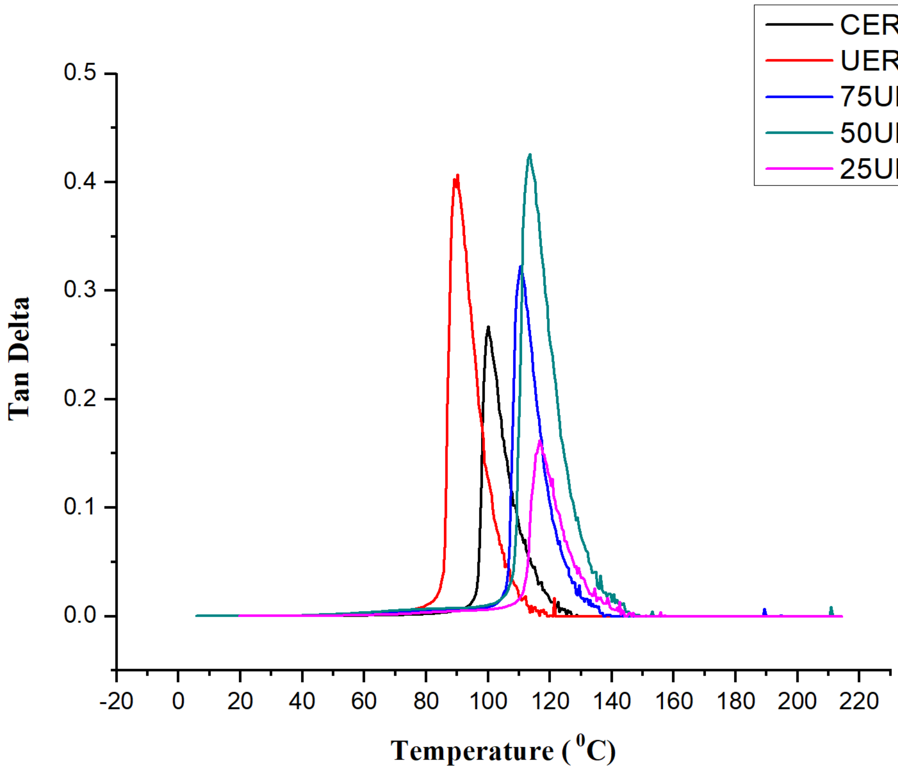

Figure 8: Tan delta-temperature curves of the post-cured materials with different compositions.

4. CONCLUSION

Modified epoxy resins with reduced brittleness and high thermal stability benefit many commercial applications today. According to the results of TGA studies, the modified epoxy resins obtained with the modifications carried out in this study exhibited higher thermal stability than the modified commercial resins. In addition, the DMA analysis determined that the storage modulus of UER-C was approximately 2.5 times that of CER-C, and similarly, 25, 50 and 75 UER-C have significantly higher storage modulus than CER-C. The same applies to lost module properties. In the case of tan δ data, it is determined that all UER-C compositions have significantly high tan δ values, which indicate that these materials can better absorb and dissipate energy, so the UER-C group have better-damping properties than that of CER-C. However, in the study, it was determined that the curing process performed with TETA at room temperature was insufficient; thus, a post-curing process procedure was optimized with DSC analyses (results not given) at 150 °C for two hours.

In conclusion, it has been determined that significant improvements can be achieved, especially in terms of damping properties, of the products obtained by incorporating urethane bonds into the epoxy main structure. In future studies, helpful studies can be carried out to produce products with improved damping properties, an essential area for materials science, by working on the ideal curing process of these compositions and similar ones.

5. CONFLICT OF INTEREST

The authors declare no conflict of interest.

6. ACKNOWLEDGMENTS

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