

RESEARCH ARTICLE

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Enhanced thermo-mechanical properties of carbon fiber reinforced thermoresistant polymer, a blend of di-functional epoxy bisphenol A and a tri-functional epoxy Tactix 742

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Abstract

Fiber reinforced polymer composite structures are particularly vulnerable to high temperatures as their strength drastically reduces under elevated temperature conditions. Therefore, it is important to improve the thermal stability of such composite structures to expand their potential range of applications. In this study, the effect of mixing two epoxy monomers, DGEBA and Tactix 742, which are di- and tri-functional, respectively, on the thermal stability of their composites is investigated. The thermo-mechanical analysis of these composites revealed that the glass transition temperature rose from 123 to 229°C when Tactix 742 is increased from 25% to 75%. The thermal resistance property is attributed to a three-dimensional crosslink network which is initiated by tri-functional Tactix 742. However, the higher percentage of Tactix led to a reduction in strength, possibly due to decreased crystallinity and formed amorphous phase material. Subsequently, a tensile strength model was employed to assess the performance at elevated temperatures. It was shown that a matrix with a mix of di- and tri-functional monomers is highly promising for manufacturing thermoresistant polymeric composites with robust mechanical properties, thereby expanding their potential applications across various engineering fields such as civil and construction industries.

Highlights

- A thermoresistant matrix by blending di- and tri-functional epoxy monomers.
- Improved thermo-mechanical properties and increased glass transition temperature of fiber reinforced composites.
- Mechanical performance of carbon fiber reinforced composites at elevated temperatures.

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- Validated a model to estimate the tensile strengths of the composites at elevated temperatures.

KEYWORDS

blending, epoxy resins, glass transition temperature, high temperature applications

1 | INTRODUCTION

In recent years, fiber reinforced polymer (FRP) composites have been used successfully in civil engineering and various construction applications due to their various advantages which include their light weight, high stiffness-to-weight ratio, and increased durability.^{1,2} However, the main concern about civil infrastructure's acceptance of FRP composites is their unpredictable structural performance at elevated temperatures. At such temperatures, FRP composites undergo significant changes since the epoxy resin which binds the fibers softens.

When FRP composites are exposed to a high temperature environment, the mechanical properties decrease in three steps. First, there is a loss in the resin matrix's capacity to transfer shear stress between reinforced fibers when close to the glass transition temperature (T_g). When the temperature is further increased to the resin decomposition temperature (T_d), the resin matrix gradually decomposes and toxic smoke is released, resulting in the second significant decrease in mechanical properties. When the temperature continues to increase, the resin matrix begins to burn, a process which releases more heat, resulting in the third significant decrease. Therefore, composites are prone to dramatically lose their stiffness and strength when being subjected to an increased temperature.³

However, thermostable matrices have the ability to maintain their elastic and fracture properties up to temperatures close to their glass transition temperature. The T_g of a polymer is dependent on the crosslink density. This density is reduced with increasing temperature. As a result, the first thermal oxidative degradation at high temperatures can be studied by measuring T_g .⁴ Therefore, the T_g of the matrix is one of the most important parameters which indicate the material's service condition.⁵ Consequently, it is a key factor determining the end applications of epoxy and its composites.

Typically, the first mechanical degradation of fiber reinforced composites occurs when exposed to temperatures greater than 120°C.^{6,7} For example, the mechanical properties of vehicle panels made of fiber reinforced composites near the engine hood can reduce drastically as their temperature can reach 130°C because of engine temperature and weather conditions.⁸ Moreover, aeronautical components like nacelles, fan cases, or engine

compressors might be exposed to temperatures between 150 and 300°C while bearing a mechanical load⁹ so their mechanical properties might be lost when typical epoxy-based composites are used.

As a result, ceramic matrix composites have been used for applications which operate in greater than 200°C environments for more than three decades. However, the use of these ceramic composites is limited to high end applications because of their high cost and special processing requirements.¹⁰ Furthermore, since most ceramic matrices require curing temperatures as high as 1000°C, commonly used high strength fibers such as carbon fibers cannot be used with ceramic matrices. Silicon carbide (Nicalon) or high temperature resistant fibers must be used as the reinforcement, which is expensive, up to \$66,000 per kilogram.¹⁰ Thus, the development of a suitable matrix is an excellent opportunity to produce a low cost, high temperature resistant composite.

Diglycidyl ether of bisphenol A (DGEBA) is one of the widely used epoxy monomers worldwide for the fiber reinforced composites.^{11–14} In recent years, there has been intensive study on the blending of different epoxies with DGEBA to improve overall performance.^{15–18} Blending is practical as it permits the preparation of a wide range of epoxy polymer varieties to arrive at the best balance and combination of properties that may not be achievable with each component alone. Multifunctional epoxies have high T_g compared to di-functional epoxies due to their high crosslinking densities.¹⁹ Therefore, the blend of di-functional epoxy with tri-functional epoxies leads to the resulting polymer blend with improved T_g compared with the corresponding di-functional epoxy.²⁰

Tactix 742 resin is a tris(4-hydroxyl phenyl) methane-based tri-functional epoxy with high T_g up to 250°C²¹ and an excellent matrix material for both commercial and military applications. Thus, this research investigates the effect of blending di-functional bisphenol A epoxy (Araldite GY 191) and tri-functional Tactix 742 epoxy monomers on thermal stability and mechanical properties of its fiber reinforced composite. Araldite GY 191 has comparatively low mechanical properties as quoted by supplier (tensile strength: 120 MPa and compression strength: 50 MPa).²² Despite its relatively modest mechanical properties, Araldite GY 191 was chosen for this study because of its multifunctional characteristics in the development of shape memory

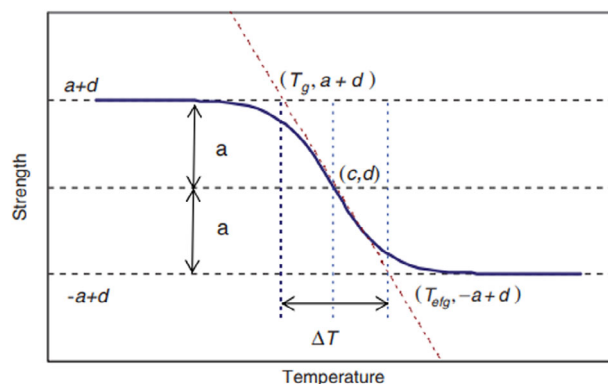
polymer composites,²³ structural supercapacitor,²⁴ and so on which will facilitate for future smart material developments.

The improvement in thermal stability, understanding the crosslinking mechanisms, comprehensive mechanical property analysis at elevated temperatures is the significance of this study. Although increasing the percentage of tri-functional epoxy enhances thermal stability by forming a three-dimensional crosslink network, it reduces the strength and stiffness due to decreased crystallinity and formed amorphous structure. Therefore, it is essential to carefully balance these factors and optimize the material properties to meet the requirements of the intended application.

Recently, few researchers have studied Tactix-modified epoxy mixtures as matrices for carbon fiber composites for high temperature applications. For instance, Foti et al.⁹ studied the effect of high temperature fatigue on cross-ply carbon fiber composite in three different environment conditions (air, 2 bar N₂, 2 bar O₂) and evaluated how an oxidizing environment promotes the fatigue degradation of the composite. In their study, a thermally stable matrix was synthesized by mixing HTS40 epoxy with Tactix 742. According to the dynamic mechanical analysis (DMA), the developed matrix had T_g between 190 and 250°C. Furthermore, Gigliotti et al.²⁵ studied thermooxidative-induced chemical shrinkage in carbon fiber composites for high temperature applications. They also used an HTS/Tactix 742 carbon/epoxy composite system for their study as it had an improved T_g . However, the effect of Tactix on thermal stability and the change of chemical structure of the epoxy mixtures is still poorly explored.

Therefore, we investigated the possibility of improving the thermal behavior of epoxy system prepared with bisphenol A-based epoxy (Araldite GY 191) and tri-functional epoxy; Tactix 742. In this regard, we prepared modified epoxy systems with varying weight ratios of tri-functional Tactix (0%, 25%, and 75%) in DGEBA epoxy resin and fabricated three composites by using wet lay-up (hand lay-up) method. Hand lay-up is relatively simple, easy to realize, and easily employed to develop different shapes with suitable molds that will fulfill the specific demands of different applications. Interestingly, it is possible to achieve comparable mechanical properties using the hand lay-up method as with some advanced methods such as the prepreg method.²⁶

The mechanical properties of carbon fiber reinforced composites prepared with different composition of Tactix were assessed through tensile, compression, flexural, and impact tests. In addition, the structural and chemical identifications for the developed polymer were probed by



$$\sigma = \frac{1}{2} (\sigma_0 - \sigma_r) \tanh \left[-\frac{1}{\Delta T} \left(T - \left(T_g + \frac{\Delta T}{2} \right) \right) \right] + \frac{1}{2} (\sigma_0 + \sigma_r)$$

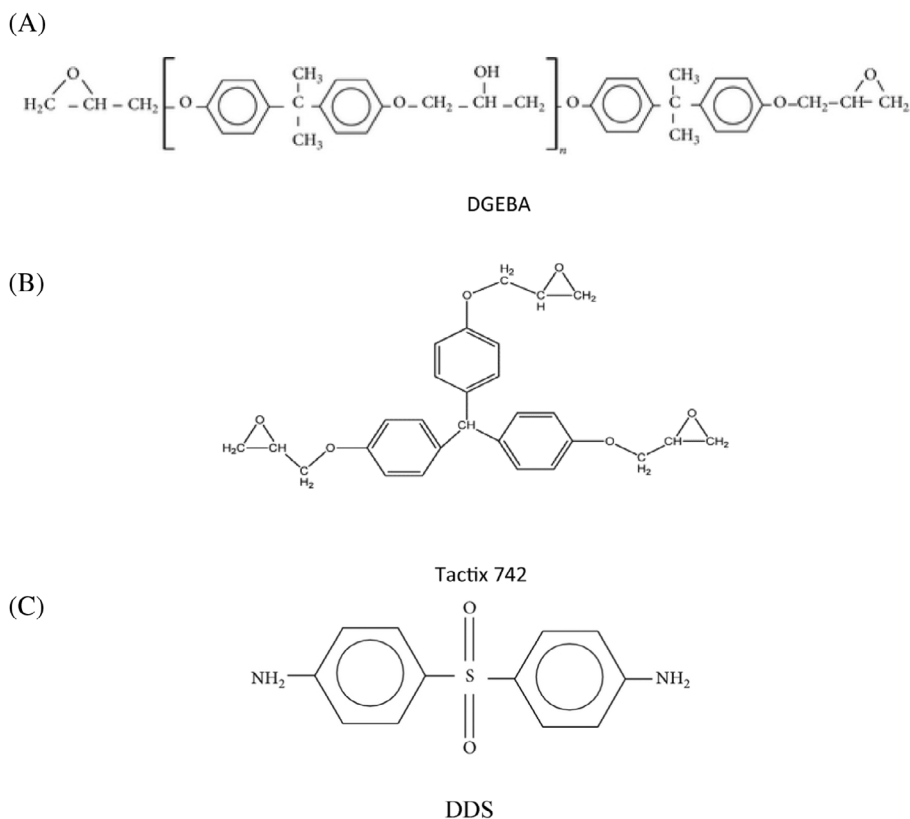
FIGURE 1 Representation of the model developed by Cao et al.²⁸

thermogravimetric analysis (TGA), Fourier transform infrared (FTIR) spectrometry and scanning electron micrographs (SEM).

1.1 | Modeling tensile strength at elevated temperatures

The performance of FRP composites may be significantly affected by temperature variations in service. Therefore, it is necessary to thoroughly investigate the temperature-dependent performance of FRP composites prior to incorporating them into critical engineering applications. Currently, there is extensive research on the high temperature performance of composites, although less on the analytical methods to estimate temperature dependent performance. Developing an empirical method to evaluate the temperature dependent properties for a specific application saves both time and effort in conducting experiments. In this regard, some researchers have investigated the effect of temperature on the tensile strength of composites and developed a correlation between tensile strength and the storage modulus measured by DMA.^{27,28} For example, Cao et al.²⁸ developed a model to estimate tensile strength at elevated temperatures based on storage modulus behavior. As shown in the model (Figure 1), tensile strengths at elevated temperatures depend on the initial/room temperature tensile strength (σ_0), the residual strength of the reinforced composites after the glass transition region (σ_r) and the glass transition temperature (T_g). Therefore, once the tensile strength is measured at a reference temperature (e.g., room temperature), a simple relationship can be used to estimate the tensile strength at higher temperatures, using the temperature dependence of the storage modulus.

FIGURE 2 Chemical structure of (A) DGEBA resin, (B) Tactix 742, (C) DDS.



2 | MATERIALS AND METHODS

2.1 | Materials

Diglycidylether of bisphenol A (DGEBA) (Araldite GY 191) as the di-functional epoxy resin (Figure 2A), Tactix 742 as the tri-functional epoxy monomer (Figure 2B) and 4-Aminophenyl sulfone (DDS) (Figure 2C) hardener were used to synthesize the epoxy blend. All chemicals were obtained from Huntsman Ltd., USA. Carbon woven fabrics (0/90) 200 gsm as the reinforcement were obtained from ATL Composite Australia. All chemicals and materials were used as received.

2.2 | Sample preparation

Prior to the preparation of the epoxy mixtures, the tri-functional epoxy, Tactix 742 was heated to 60°C for 1 h to attain a workable viscosity. Then, blends were formulated with DGEBA:Tactix weight ratios of 100:0, 75:25, and 25:75. The resin mixtures were thoroughly stirred for 30 min until they were homogenous. These modified resins were mixed with the curing agent DDS, continuously stirring for another 15 min. The temperature was kept at 60°C for proper mixing. Then three composite panels containing six layers of carbon fiber fabrics were



FIGURE 3 Experimental setup for tensile test at high temperatures.

fabricated using the three different epoxy mixtures by hand lay-up. These layups were initially cured at 177°C for 3 h and then 250°C for 2 h using a convection oven. The samples of 0/100, 25/75, and 75/25 (Tactix/DGEBA)

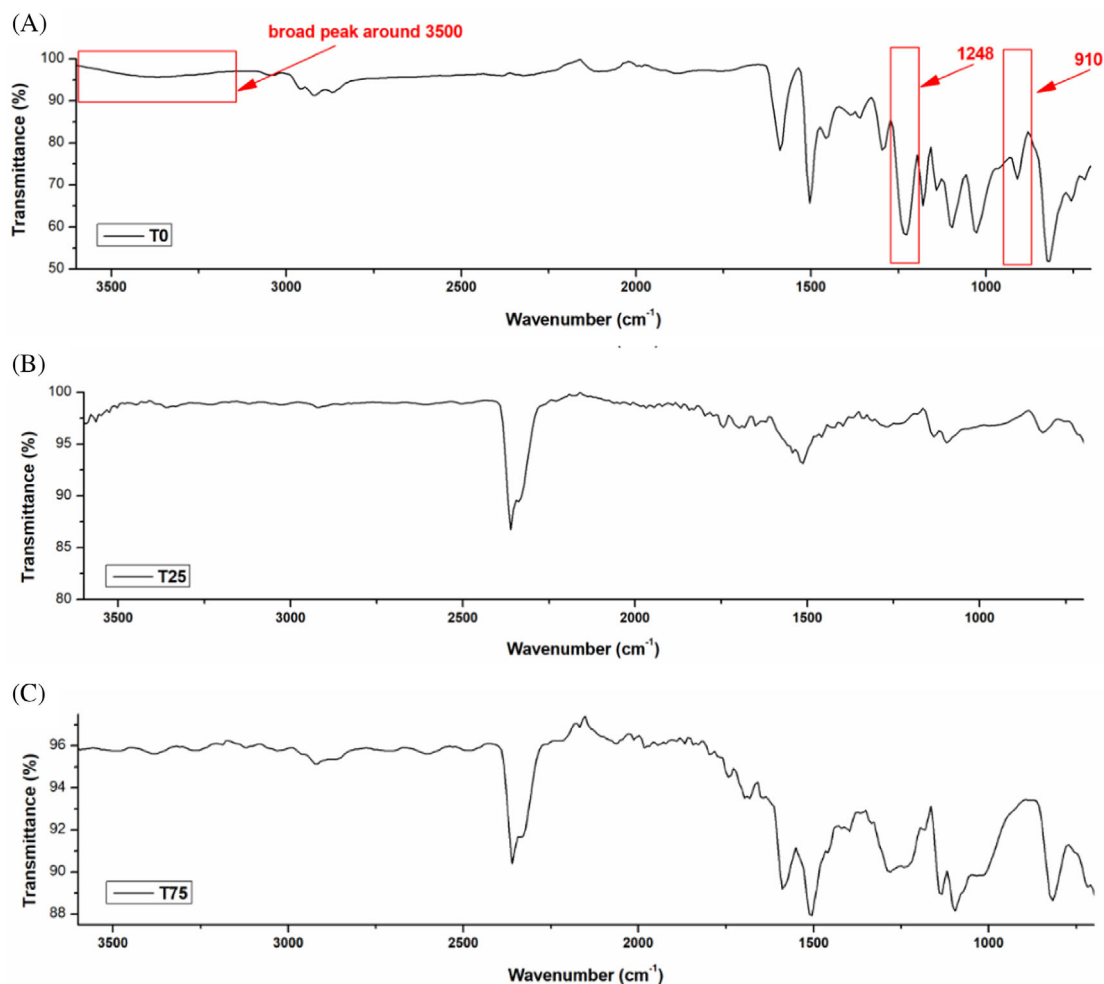


FIGURE 4 Fourier transform infrared spectra (A) T0, (B) T25, (C) T75.

blends were designated as T0, T25, and T75, respectively. The dimensions of the final panels were 300 mm \times 300 mm \times 3 mm, which allowed for cutting standard coupons for thermo-mechanical testing.

2.3 | Experimental method

2.3.1 | Fourier transform infrared characterization

An FTIR analysis was performed to study the chemical structure of the modified epoxy (T0, T25, and T75) at room temperature. The samples were scanned from 4000 to 400 cm^{-1} at a resolution of 4 cm^{-1} .

2.3.2 | Dynamic mechanical analysis

DMA was performed to determine the T_g of the synthesized composite using a TA instrument hybrid rheometer

(Discovery HR-2). Three rectangular samples with dimensions of 45 mm \times 8 mm \times 3 mm were subjected to temperature sweeps from 25 to 300°C at a heating rate of 5°C/min in a dual-cantilever mode using a frequency of 1 Hz and amplitude of 25 μm . The T_g ($\tan \delta$) was estimated from the maximum value of the $\tan \delta$ plot from an average of three tested specimens.

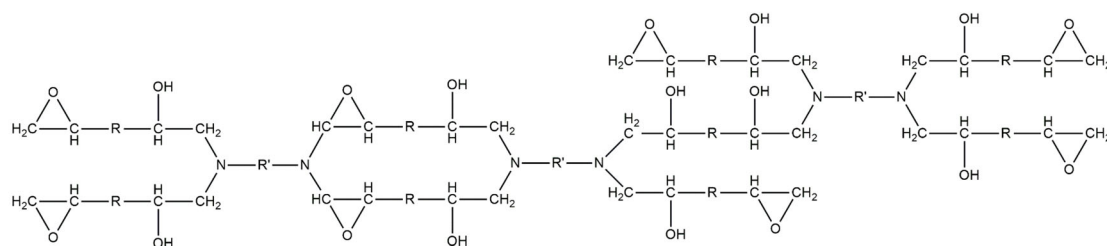
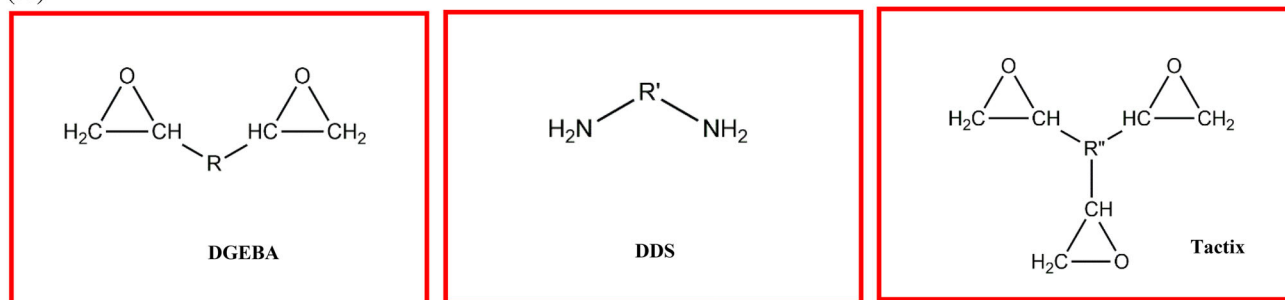
2.3.3 | Thermogravimetric analysis

Thermogravimetric analysis was performed to evaluate thermal stability using a Universal V4.5A TA instrument. Three samples (T0, T25, and T75 in mgs) were heated at a rate of 10°C/min from 30 to 600°C.

2.3.4 | Mechanical testing

Mechanical tests for determining tensile, compression, flexural and impact strengths were conducted according to

(A)



(B)

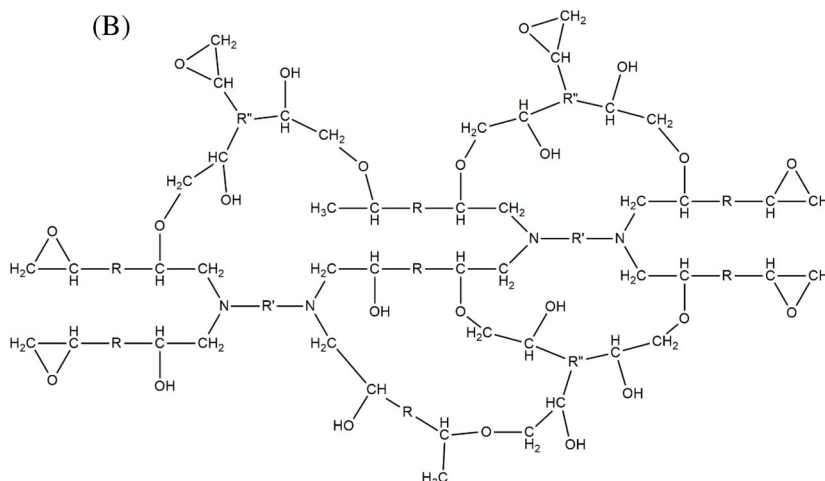


FIGURE 5 Chemical reactions (A) two-dimensional linear polymer formation from DGEBA with DDS, (B) three-dimensional polymer formation due to crosslinking in the presence of Tactix with DDS.

ASTM D3039, ASTM D6641, ASTM D790, and ASTM D256 standards, respectively. An MTS 100 kN (Insight Electromechanical) uni-axial testing machine was used to evaluate the tensile strength at room temperature, while an MTS 10 kN testing machine was used to evaluate the tensile strengths at two elevated temperatures (70 and 130°C). The compression and flexural strengths were determined using the MTS 10 kN machine. An Instron Dynatup 8200 drop weight impactor was used to conduct the impact test. The fiber weigh fraction was evaluated for three composite samples (T0, T25, and T75) according to ASTM D3171.

2.3.5 | Residual strength testing at 250°C

The residual tensile strengths were determined by conducting a tensile test at a temperature beyond the glass

transition region. This was carried out at 250°C using the apparatus shown in Figure 3, since this temperature is beyond the glass transition region of each sample.

3 | RESULTS AND DISCUSSION

3.1 | Formation of a three-dimensional crosslink network

The FTIR spectra for the DGEBA/DDS and DGEBA/Tactix/DDS epoxy systems are presented in Figure 4A–C, respectively. In the presence of DDS, the DGEBA starts to cure and form a long-chained polymer. The hydroxyl groups and residual epoxy rings are still available in the polymer structure, and this is shown in the FTIR spectra for T0 (Figure 4A). The broad peak at around 3500 cm⁻¹

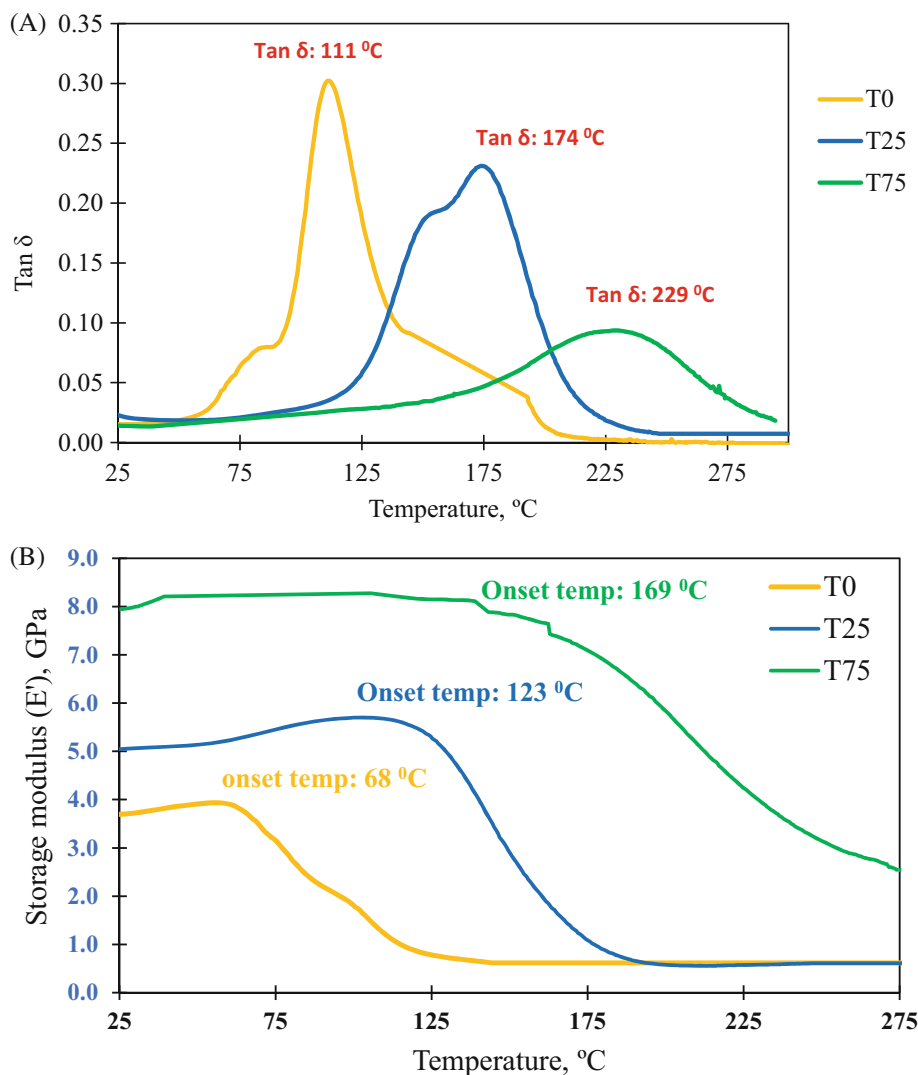


FIGURE 6 Dynamic mechanical analysis results (A) tan δ versus temperature, (B) onset temperature.

corresponds to the hydroxyl groups in branched polymer in T0 and the sharp peaks at 1248 and 910 cm^{-1} correspond to epoxide groups of T0. With the addition of Tactix, these hydroxyl and epoxide groups react with the epoxide groups of the Tactix and form three dimensional networks. Due to these crosslink formations, the concentration of hydroxyl and epoxide groups in the polymer reduced. This is clearly illustrated by the disappearance of the corresponding peaks in the T25 and T75 (Figure 4B,C). Figure 5 illustrates the chemical structure of T0, T25, and T75 and three-dimensional crosslink network which occurs due to Tactix.

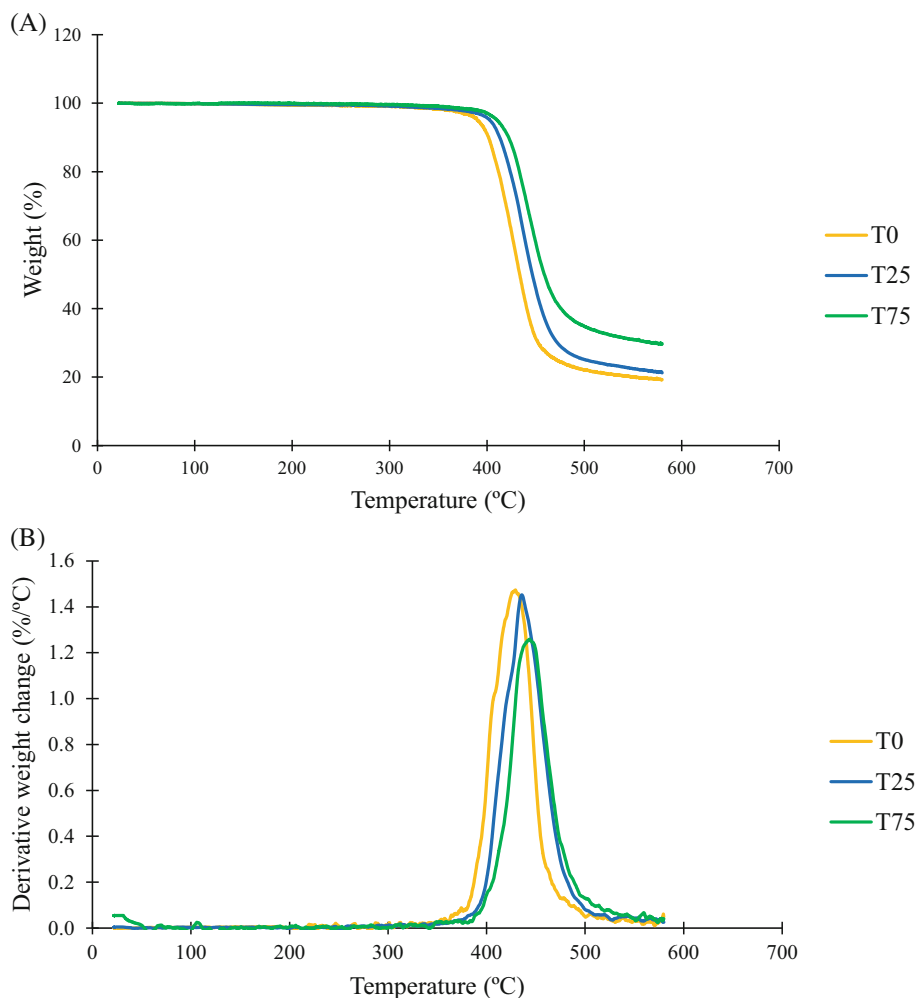
3.2 | Thermal resistance and stability

A DMA analysis for the composites prepared with the different mixtures of the di-functional and tri-functional epoxy monomers was performed. The T_g of the composites, both peak tan δ and onset temperature,

increased with increasing Tactix content, as indicated by Figure 6A,B. As explained in Section 3.1, crosslink density increases with increasing Tactix, and therefore glass transition temperature progressively increased in T0, T25, and T75, displaying T_g values of 111 , 174 , and 229°C , respectively. These T_g values are close to the aerospace-qualified epoxy matrices and therefore, these composites are suitable for a wide range of applications requiring stability at high temperatures.¹⁹

The thermal stability of modified epoxy systems is presented in Figure 7A,B as thermogravimetric (TG) and derivative weight loss versus temperature (DTG) curves. The maximum degradation temperatures were 434 , 437 , and 446°C in T0, T25, and T75, respectively. According to the results, the thermal stability of the epoxy system prepared by mixing DGEBA and Tactix is marginally higher than the DGEBA epoxy system. However, T75 has shown a minimum residual weight loss with increasing temperatures beyond the maximum degradation temperatures. The increase of thermal stability can be attributed to the

FIGURE 7 Thermogravimetric analysis (A) TG curves, (B) DTG curves.



formation of three-dimensional networks in the Tactix/DGEBA/DDS mixture.

Furthermore, T0 has shown comparable thermal stability with T25 and T75, although its T_g is lower as 111°C (tan δ). Similar results have been found in many syntheses of DGEBA based polymers.^{29,30}

3.3 | Mechanical analysis

The fiber weight fractions of three composites were calculated as 37.5%, 38.3%, 37.8% for T0, T25, and T75 composites (in average 38%), respectively. As there is no significant difference in weigh ration of fiber to resin, the variations in mechanical properties among the three composites are merely due to the differences in the composition of epoxy blends.

The mechanical properties of three composites are illustrated in Figure 8 and a summary is shown in Table 1. The highest tensile strength was recorded as 359.57 MPa for T0 composite. In this study, woven (0/90) carbon fibers were used as the reinforcement due to many advantages

compared to unidirectional fibers.³¹ Fiber orientation is one of the main factors which determine the in-plane properties and some studies have proved that tensile strength of woven CF composite can decrease by half compared to unidirectional CF composites.³² However, based on recent advancements in fiber-reinforced composites for structural applications,^{33–38} the properties of the developed composite still meet the requirements of certain applications.

The mechanical properties of T25 composite are significantly closer to those of the T0 composite. However, the addition of 75% of Tactix into the epoxy mixture results in a reduction of tensile and flexural strength. Compared to T25, the tensile strength of T75 was reduced by around 40%. T0 composite breaks at 3.3% strain while T75 breaks with an elongation of 2.6%, reflecting typical brittle fracture behavior, compared to T0. Moreover, T75 has shown sharp, brittle failure at multiple locations, while the other samples (T0 and T25) failed with delamination (Figure 9). This proves that 75% Tactix-modified DGEBA undergoes a lower percentage of stress before failure, which will significantly contribute to reducing the toughness of the mixed epoxy resin. This can be explained by the formation of crosslink network

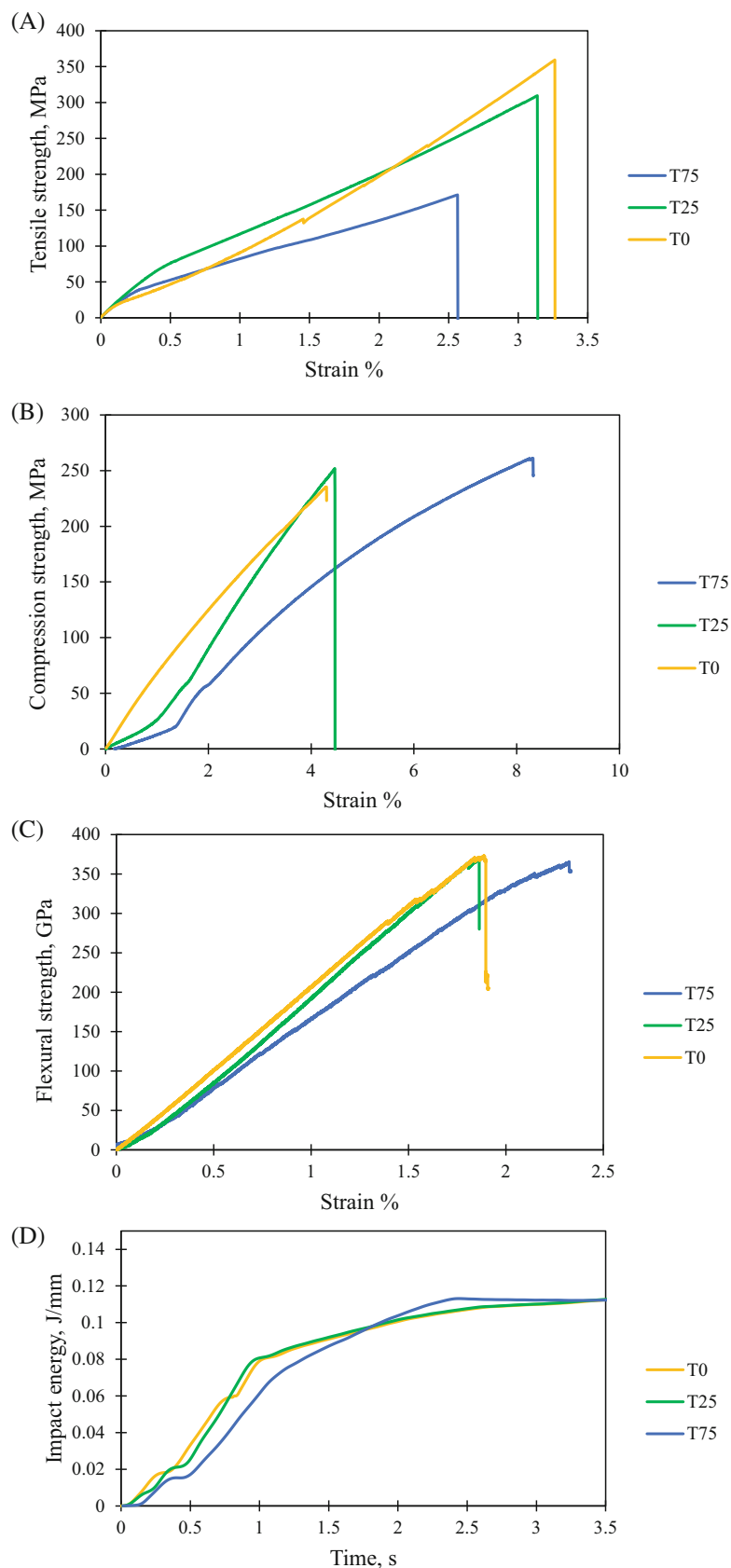


FIGURE 8 Mechanical properties (A) tensile stress versus strain %, (B) compression stress versus strain%, (C) flexural stress versus strain %, (D) impact energy, energy versus time.

when DGEBA is mixed with Tactix. Especially, the higher crosslink network in T75 hinders crystallization and generates a largely amorphous phase.³⁹ As a result, the

tensile strength is reduced. This is further confirmed by the reduction of the flexural strength values of T75 compared to T0 (Figure 8C).

TABLE 1 Mechanical properties of carbon fiber composite with modified epoxy system at room temperature (25°C).

Property	Unit	T0	T25	T75
Tensile strength	MPa	359.57	309.57	171.32
Elastic modulus	GPa	39.50	31.62	29.60
Compression strength	MPa	235.42	251.92	261.19
Compression modulus	GPa	58.11	52.93	33.70
Flexural strength	MPa	373.08	371.07	365.58
Impact load	kN	1.48	1.48	1.17
Impact energy	J/mm	0.15	0.15	0.11

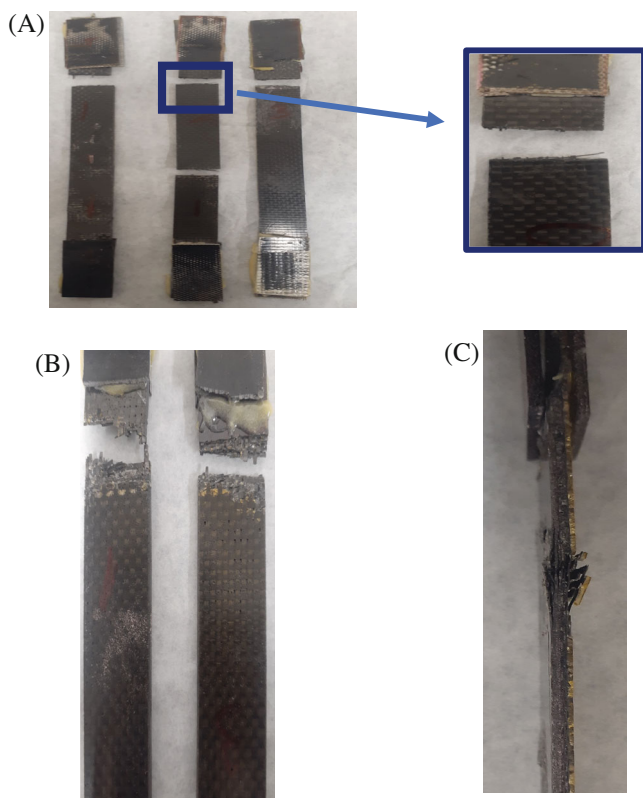


FIGURE 9 Broken samples (A) T75, (B)T25, (C) T0.

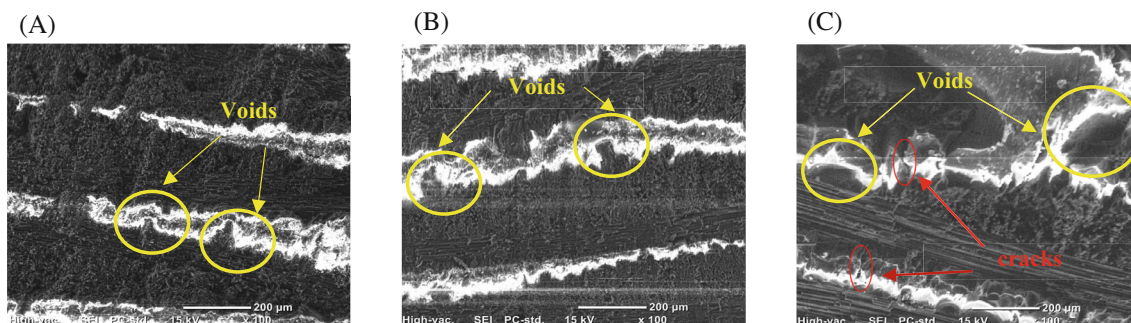


FIGURE 10 Scanning electron micrographs for (A) T0, (B) T25, (C) T75.

As shown in Figure 8B, T75 epoxy blend has the highest compression strength among the samples under investigation. The lower molecular flexibility as a result of higher crosslink density in epoxy blends rich in Tactix (T75) influences the compression. The impact strength is calculated by dividing impact energy by the thickness of the notch and this is shown in Figure 8D. The impact energies of these three types of composites, T0, T25, and T75, are almost similar and are significantly higher when compared to the fiber reinforced

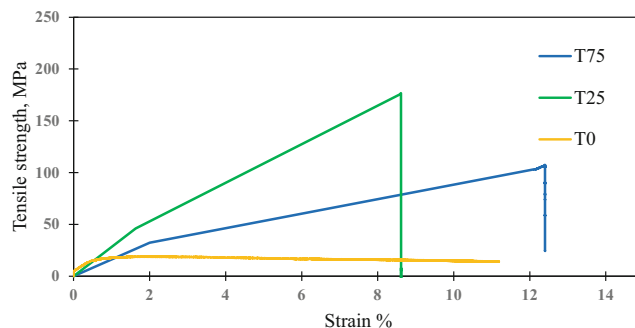


FIGURE 11 Residual strength at 250°C.

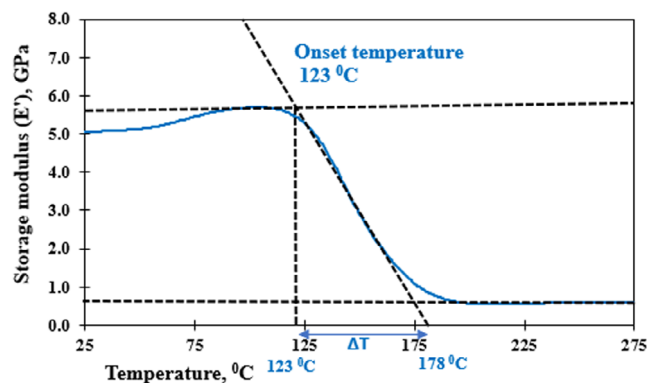


FIGURE 12 Corresponding model parameters for T25.

composites developed for modular constructions with shape memory properties.³⁰

3.4 | Morphology of the carbon fiber reinforced DGEBA/Tactix composites

Scanning electron microscopy (JEOL JCM-6000) was used to evaluate surface morphology and microstructure of three

blends. Figure 10 shows SEM characterization for the three composites (T0, T25, and T75) at 100 magnification and the mechanical properties were well explained with the details of SEM images. In Figure 10C, the SEM image shows a glassy surface with few cracks and voids on T75 samples and, the increased number of voids compared to T25 samples. This might be due to the T75 composite's brittleness.⁴⁰ However, Figure 10A,B have less cracks, voids, and ridges, confirming T75's low mechanical performance compared to T0 and T25.

TABLE 2 Experimental versus estimated tensile strengths of T25 at different temperatures.

Temperature	Experimental value	Estimated value (from model)
70°C	304 MPa	309 MPa
130°C	290 MPa	285 MPa

3.4.1 | Residual strength at 250°C

The residual tensile strengths of three types of composites, T0, T25, and T75, were measured as 18, 176, and 111 MPa respectively, as shown in Figure 11. The strain at the failure was over 12%, revealing T75's excellent thermal stability,

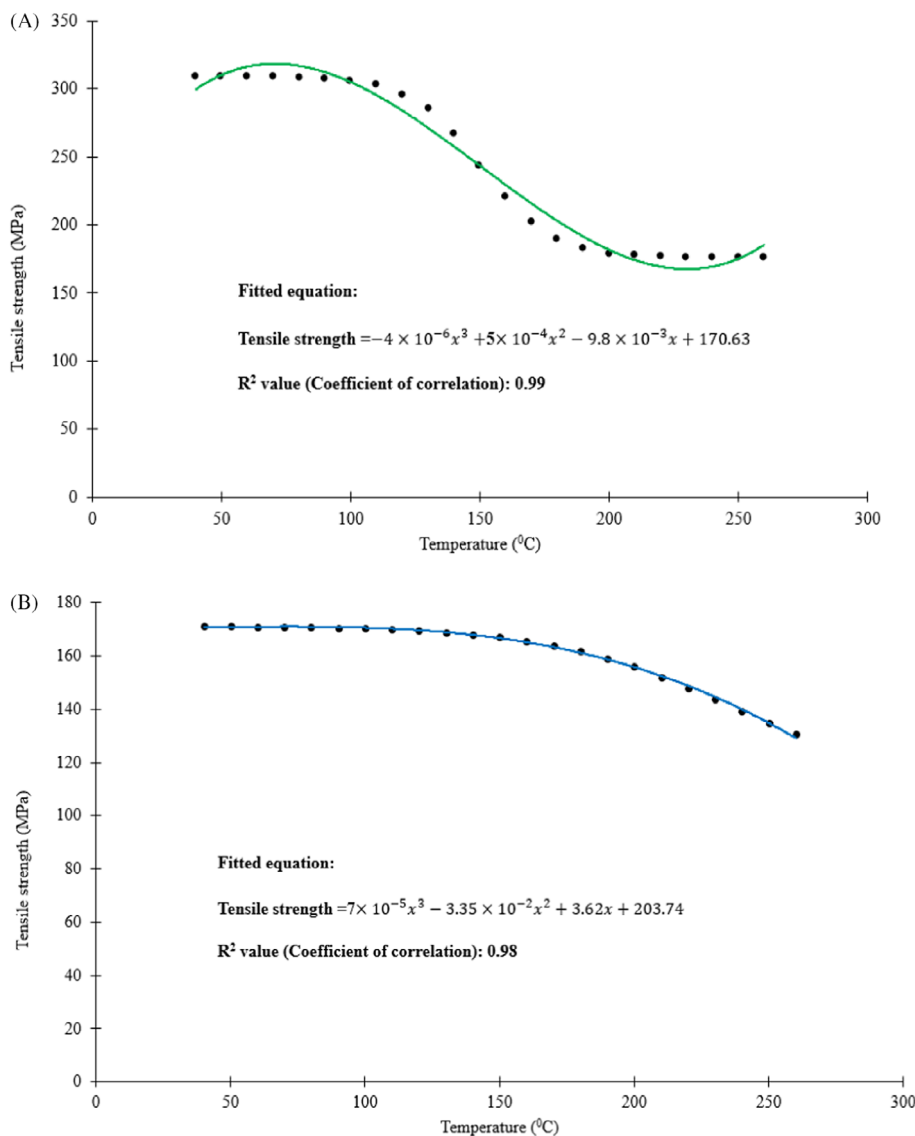


FIGURE 13 Fitted curve for tensile strength versus temperature (A) T25, (B) T75.

while T25 has shown around 8% strain. T0 shows extremely poor elastic behavior at 250°C as anticipated.

3.4.2 | Tensile strength at elevated temperatures

The applicability and accuracy of the model were tested using the experimental results at elevated temperatures. The tensile strengths at two different temperatures (70 and 130°C) were experimentally evaluated for T25 and compared with the values estimated with the corresponding model parameters, as illustrated in Figure 12. Deviations between the estimated values and experimental values (Table 2) are less than 2%. Therefore, the model's prediction of tensile strength versus temperature is acceptable for the developed composite. The estimated values are shown in Figure 13 with curve fitting equations.

4 | CONCLUSION

This research has focused on assessing the performance of carbon fiber composites prepared using an epoxy matrix system consisting of two epoxy monomers: di-functional and tri-functional epoxy monomers. In particular, this research investigates the effect of blending di-functional bisphenol A epoxy (Araldite GY 191) and tri-functional Tactix 742 epoxy monomers on thermal stability and mechanical properties of its fiber reinforced composites. From the DMA result, it was found that the thermo-mechanical property of the composite is considerably improved by Tactix 742, where the T_g rises from 123 to 229°C when Tactix 742 is increased from 25% to 75% in the epoxy mixture. The thermal stability of the composite was attributed to the formation of three-dimensional networks in the Tactix/DGEBA/DDS mixture. According to FTIR analyses, Tactix behaves as a crosslinking agent, attaching to the epoxide and carboxyl groups of DGEBA and forming a three-dimensional network during the curing. Notably, it was observed that the higher percentage of Tactix led to a reduction in strength, possibly due to decreased crystallinity, and formed amorphous phase materials. T25's mechanical properties at room temperature were: a tensile strength of 310 MPa, flexural strength of 371 MPa, compression strength of 251 MPa, and impact energy of 0.15 J/mm, respectively. In addition, the tensile strength model for elevated temperatures revealed that the T25 composite has tensile strength of 309 and 285 MPa at 70 and 130°C, respectively. These findings have proven the stability of mechanical properties of T25 composite at temperatures higher than 100°C. Considering the obtained results, it is meaningful to blend di-functional bisphenol A epoxy (Araldite GY 191) and tri-functional Tactix 742 epoxy

monomers to develop a thermal stable composite with sufficient mechanical properties. Thus, this study has contributed to develop a cost effective novel polymeric material which is suitable for general structural applications at elevated temperatures.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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