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ARTICLE

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Plasticizing effect of biodegradable dipropylene glycol bibenzoate and epoxidized linseed oil on diglycidyl ether of bisphenol A based epoxy resin

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Abstract

Major limitation for use of epoxy thermosets in engineering applications is its sudden brittle failure. In the present study dipropylene glycol dibenzoate (DPGDB) based plasticizer is used to modify diglycidyl ether of bisphenol A (DEGEBA) based epoxy resin system via simple blending technique. Bio-based epoxidized linseed oil was also used to modify epoxy resin system and compared with DPGDB modified resin. For DPGDB modified resin storage modulus and loss modulus of the epoxy system modified with 10% plasticizer increased by 7.54% and 12.24%, respectively. The primary mechanism responsible for such behavior is improved crosslinking density. With 5% plasticizer loading, flexural strength increased by 21%. There was an improvement of 312.74% in strain at failure for 10% plasticizer loading, while preserving its mechanical strength. It was found that DPGDB based modification was better than epoxidized linseed oil modification.

KEYWORDS

mechanical properties, structure-property relationships, synthesis and processing techniques, thermal properties, viscosity and viscoelasticity

1 | INTRODUCTION

The use of composite materials has rapidly increased in recent years particularly for advanced engineering applications.[1–5] Thermosets and thermoplastics are used as matrix for fiber reinforced polymers. Among all the polymers, epoxy resins are the most widely used materials as matrix. Diglycidyl ether of bisphenol A (DGEBA) based resins are of particular importance as they are extensively being used for structural reinforcement,[6] electronics,[7] coatings,[8] and adhesives.[9] DGEBA based resins attract such high demand due to their outstanding mechanical and thermal properties, high chemical and corrosion resistance, outstanding adhesion to various substrates, low shrinkage upon cure, good electrical insulating properties, and ability to be processed under variety of conditions. However, due to high crosslinking during cure reaction they are very brittle and less resistant to cracks that makes them less suitable for mission critical applications.[10–12] To overcome this problem toughening mechanisms have been studied over the past few decades. Toughening of resin by means of adding suitable toughening agents or chemical modification is an indirect way to improve the energy absorption capacity of the material. Toughening of resin not only decreases brittleness of the material but also improves toughness, impact resistance, elongation (ductility) and resistance to crack propagation. However, care must be taken that strength and modulus of material are not compromised due to toughening.[13–16]

Various methods were used in past to modify epoxy resins to improve toughness and reduce brittleness.

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These methods include blending with inorganic particles, liquid elastomers, thermoplastics and biodegradable polymers. Toughening epoxies using elastomeric materials is very effective but it reduces mechanical properties and thermal stability of the system drastically. Toughening by particulate (micro/nano size) method can increase viscosity of the resin, which might lead to other problems during infusion and curing, as they are prone to agglomerations. Hence, the most viable approach could be incorporation of dispersed polymer in the cured epoxy matrix. By such approach, a synergy of orientation to improve flexibility of system and 3D network structure of epoxy can be achieved.[17–25]

Liquid polysufide polymer was used as plasticizing agent in bisphenol A and epichlorohydrin epoxy system hardened with triethylene tetramine by E. Sideridis et al.[26] Viscoelastic properties of the material were calculated using ultrasound based NDT methods. It was found that tensile modulus and strength decreased with plasticization whereas Poisson ratio increased. Storage modulus decreased with increasing plasticizer content and loss modulus increased with increasing plasticizer due to internal damping. Another approach to improve desirable elongation properties of epoxy is to modify the curing agent. S. Su et, al[27] used polyamide and the polyether amine based curing agents. Due to this approach cross-link density was adjusted by two-phase separation phenomena and resulted in high elongation at break (>100%) and still acceptable tensile strengths. Recently in another study, Baig et al[28] modified epoxy curing agent with two different amine terminated additives and studied its effects on mechanical and thermal properties of epoxy polymer. They have found that aromatic amine terminated polymers imparted better overall mechanical properties than aliphatic amide polymers.

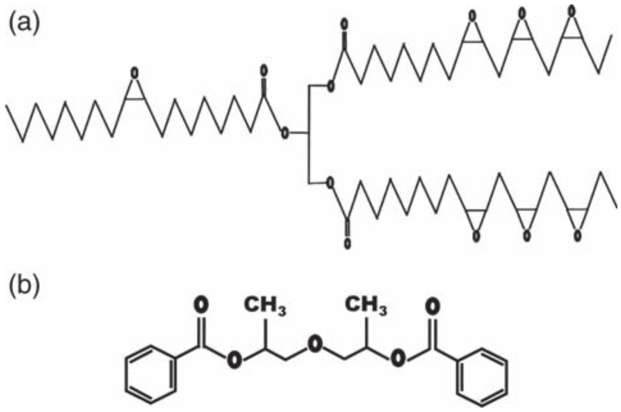

FIGURE 1 Block chemical structure of expoxidized linseed oil (ELO) and dipropylene glycol dibenzoate (DPGDB)

Currently, there has been lot of interest among material researchers to utilize natural source based materials to alter desirable properties of epoxy-based systems. Various bio-based additives like micro fibrillated cellulose,[29] bacterial celluloses,[30–31] silk fibroin,[32] and epoxidized natural rubber[33] have been used to modify epoxy systems. Vegetable oil based epoxies are another important class of materials that are being extensively explored for altering engineering properties of epoxy systems.[34] Yim et, al.[35] investigated the effects of epoxidized linseed oil (ELO) on mechanical and viscoelastic properties of epoxy

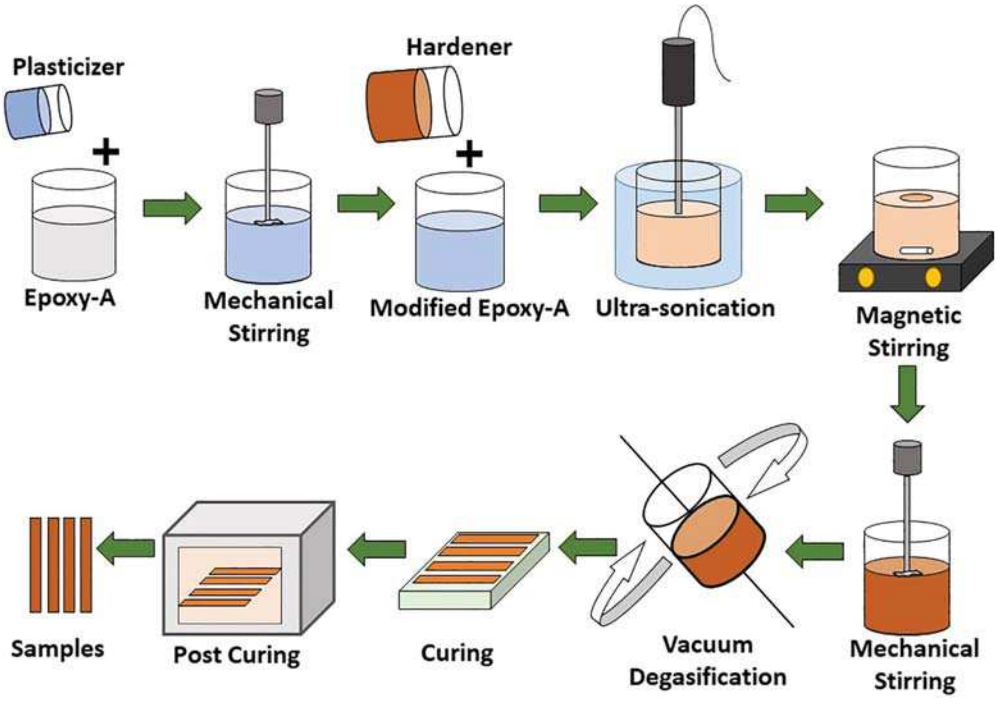

FIGURE 2 Schematic illustration showing fabrication of polymer samples [Color figure can be viewed at wileyonlinelibrary.com]

DGEBA based epoxy system. They found that cross-link density of epoxy decreased with addition of ELO (Figure 1), which resulted in enhanced impact strength of the system. Vu-Bach et al[36] investigated the effect of carboxyl-terminated poly (acrylonitrile-co-butadiene) grafted epoxidized linseed oil (CTBN-g-ELO) mechanical and fracture toughness properties of epoxy resin system. They have reported that the tensile strength, impact strength, and fracture toughness of the epoxy increased due to cavitation/debonding of CTBN-g-ELO from epoxy matrix.

Plasticizers are a class of materials generally added to polymer system to either ease the process ability of the polymer or increase flexibility of the overall material. They give opportunity to the researchers to develop new variants of materials from the few available polymers.[37] Plasticizers are traditionally used with thermoplastic materials. By lowering the transition temperature, they help to improve the flexibility.[38] Dipropylene glycol dibenzoate (DPGDB) (Figure 1) is a recent choice of plasticizer for polyvinyl chloride (PVC) polymer mainly because of its less toxicity and biodegradability.[39] PVC polymer system was modified using DPGDB based plasticizer, it was reported that they were effective in increasing process ability and flexibility of PVC.[40] Nowadays the scope for use of DPGDB plasticizer is explored for polymers other than PVC. Recently researchers have investigated use of DPGDB plasticizer to develop poly (vinylidene fluoride)/polymethyl methacrylate blend. It was found DPGDB severed as a good diluent, via solid–liquid (S-L) phase separation to develop a microporous membrane.[40] Another study where Wan et, al.[41] used DPGDB to plasticize Poly (lactic acid) (PLA) which is a biodegradable aliphatic thermoplastic. It was reported that PLA/DPGDB blend exhibited improved flexibility with good mechanical and thermal properties.

As it is can be established that biodegradable DPGDB can impart good plasticizing effect on thermoplastic materials while preserving its mechanical and thermal properties, its use for plasticization of epoxy has never been explored in the past to best of our knowledge. The aim of present study is to investigate for the first time the effect of DPGDB based plasticizer blending with DGEBA based epoxy resin system on its viscoelastic, thermal and mechanical properties. An epoxy resin system modified with ELO was also studied for comparison.

2 | EXPERIMENTAL

2.1 | Materials

Polymer used for this study is a SC-15 supplied by Applied Poleramics Inc (API), It is a low-viscosity two-phased toughened epoxy resin system consisting of part A (resin mixture of diglycidylether of bisphenol-A, aliphatic diglycidylether epoxy toughener) and part B (hardener mixture of cycloaliphatic amine and polyoxylalkylamine). Synthetic DPGDB based liquid plasticizing agent EP9009 was purchased from Eager Plastics, Chicago, IL, USA. ELO based biodegradable plasticizer VikoFlex 7190 with epoxy equivalent weight 176 was purchased from Arkema, Colombes, France.

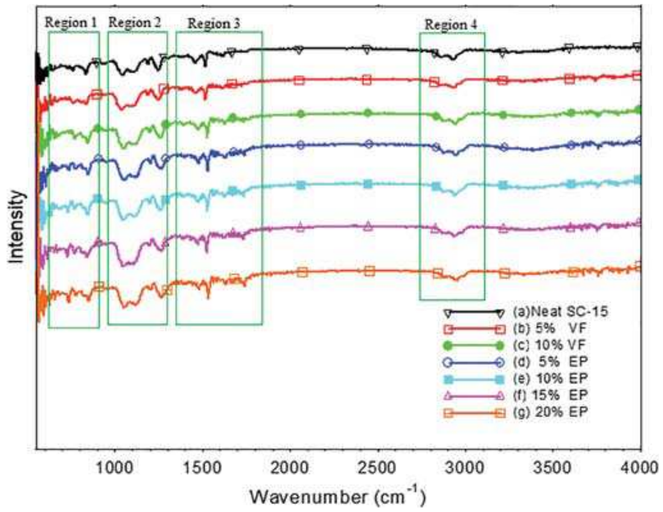

FIGURE 3 Fourier transform infrared (FTIR) spectrum full of neat and modified epoxy system [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 1 FTIR wavelengths indicating prominent bonds

Wavelength (cm^{-1})	Bonding type
710	C—H aromatic
1000–1125	C—N aliphatic
1415–1455	C—C aromatic
2850–2950	Alkyl polyamine

Abbreviation: FTIR, Fourier transform infrared.

2.2 | Preparation of epoxy/plasticizer blend system

SC-15A was mixed with measured amount of plasticizer EP9009 or VF7190. The plasticizer loading was varied from zero to 20 wt%. The mixture was mechanically mixed for 5 min. Then hardener (part B) in suggested stoichiometric ratio part A/B (100:30) was added. The mixture was ultra-sonicated for about 20 min using a one fourth inch probe. The solution was then further mixed by mechanical stirring for about

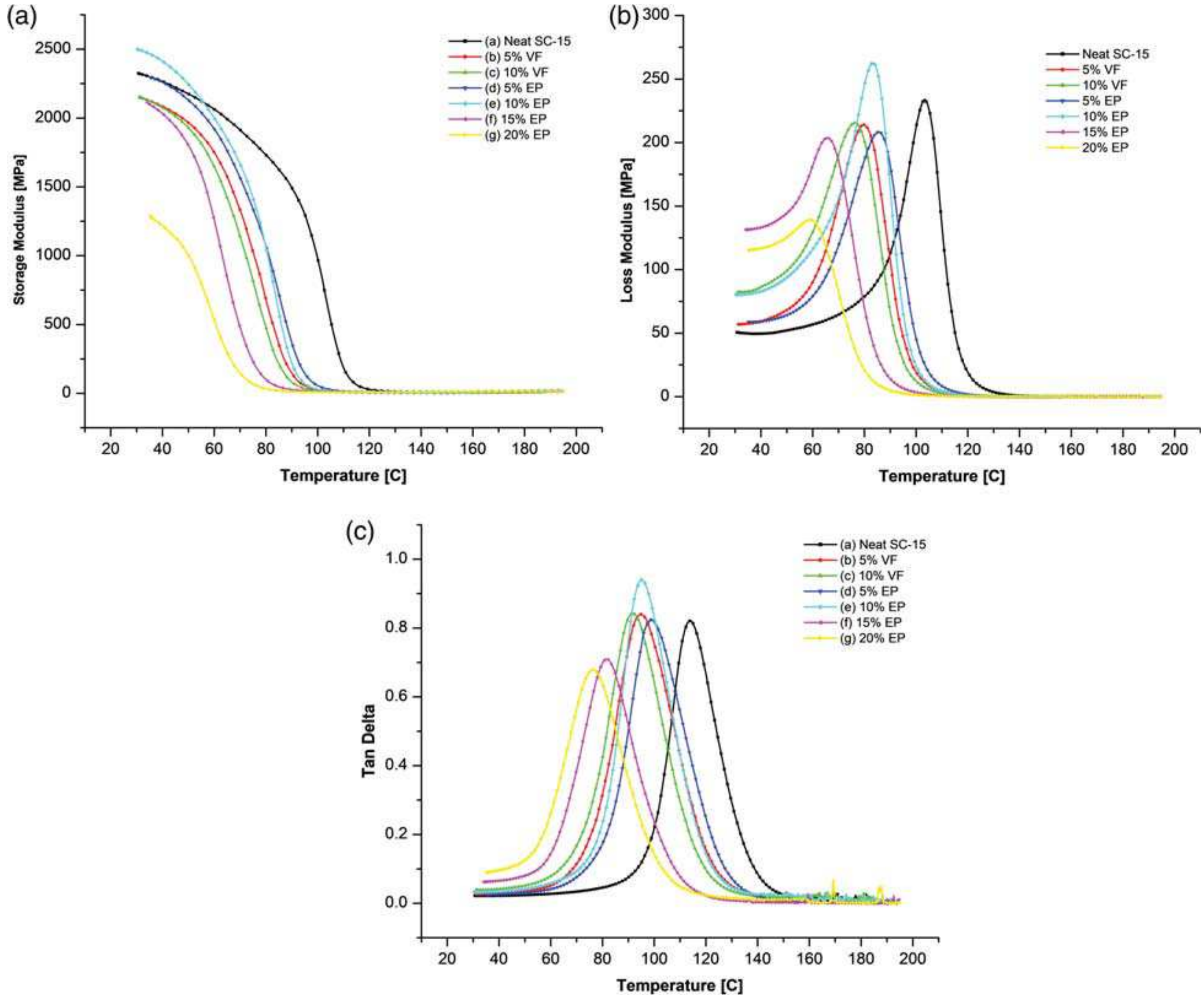

FIGURE 4 Storage modulus, loss modulus and Tan Delta curves of neat and modified epoxy system [Color figure can be viewed at wileyonlinelibrary.com]

5 min. The polymer blend was then vacuum degasified for 30 min to remove any entrapped air bubbles. The mixture was then poured into silicone mounds of desired shape and allowed to cure at 60°C for 2 h and then post cured at 100°C for another 2 h. (Figure 2) Samples were then removed from molds and modified according to ASTM standards. Epoxy was blended with 5 and 10% epoxidized linseed oil VikoFlex 7190 (VF) and 5, 10, 15 and 20 wt% of synthetic plasticizer EP9009 (EP). Samples are denoted by (a) neat SC-15 (b) 5% VF (c) 10% VF (d) 5% EP (e) 10% EP (f) 15% EP (g) 20% EP.

2.3 | Characterization

Chemical analysis on epoxy systems was performed by Fourier transform infrared-attenuated total reflectance (FTIR-ATR) (FTIR-8400S Shimadzu spectrophotometer) using 50 averaged scans and 4 cm^{-1} resolutions over a range of 4000–650 cm^{-1}. FTIR studies were conducted to investigate effect of oxygen treatment for various duration on functional group changes on the surface of pellets. Background spectra were taken in the empty chamber before measurements to eliminate the influence of water moisture and CO_2 in air.[42]

Viscoelastic properties of polymer blend system were investigated using Dynamic Mechanical Analysis (DMA) on a TA Instruments' Q800 operated in dual cantilever mode according to ASTM 4065. Samples were tested from 30 to 200°C at a ramp rate of 5°C/min. Characteristic storage and loss moduli, and tan delta curves were obtained as function of temperature for each sample. Three samples from each set were tested and average glass transition temperature (Tg) determined from peak of loss modulus curve were obtained for comparison. Dimensional stability of polymer system was investigated using Thermo-mechanical analysis (TMA) on a TA Instrument Q400. The temperature was ramped from

TABLE 2 Dynamic mechanical properties of neat and modified epoxy system

Sample	Storage modulus (MPa)	Loss modulus (MPa)	Tan Delta	T_g (°C)
Neat SC-15	2324.8 ± 144.14	234.13 ± 10.97	0.82 ± 0.00	114.7 ± 1.31
5% VF7150	2150.28 ± 253.43	214.05 ± 21.14	0.84 ± 0.03	94.87 ± 0.95
10% VF 7150	2152.72 ± 076.92	215.39 ± 04.85	0.84 ± 0.03	91.9 ± 1.02
5% EP9009	2296.09 ± 164.61	208.33 ± 19.20	0.82 ± 0.05	98.7 ± 1.31
10% EP9009	2500.21 ± 043.22	262.78 ± 06.69	0.94 ± 0.02	95.0 ± 0.85
15% EP9009	2113.54 ± 019.68	203.58 ± 02.46	0.71 ± 0.01	81.6 ± 0.49
20% EP9009	1288.29 ± 010.52	139.38 ± 05.37	0.68 ± 0.01	76.7 ± 0.75

TABLE 3 Crosslink density of neat and modified epoxy system

Sample	Rubbery plateau modulus E_r (Mpa)	Temperature T_r (K)	Crosslink density ν_t (mol/m^3)
Neat SC-15	9.77	459.15	853.12
5% VF7150	5.47	427.15	513.42
10% VF 7150	6.91	459.15	603.38
5% EP9009	7.38	427.15	692.7
10% EP9009	18.29	459.15	1597.08
15% EP9009	12.39	459.15	1081.9
20% EP9009	13.83	459.15	1207.64

ambient temperature to 250°C at a rate of 5°C/min. Coefficients of thermal expansion before and after glass transition were determined from each sample set according to ASTM D696 standards. Specimens of size $10 \times 10 \times 5$ mm^3 were subjected to constant force (0.2 N) and coefficient of thermal expansion (CTEs) were determined in the ranges (30–55°C) and (120–170°C). The cross-link density was calculated using the rubber plateau region of the storage modulus curve.[43]

$$E_r = 3RT_r\nu_t,$$

where E_r is storage modulus (MPa), R is Avogadro's number (m^{3}Pa Kmol$^{-1)}$, T_r is temperature (°K) and ν_t is cross link density (mol m^3).

Therefore, $\nu_t = \frac{E_r}{3RT_r}$.

Thermal decomposition behavior of the epoxy system was investigated via Thermo-gravimetric analysis (TGA) on a TA Q500 instrument. Samples were scanned from ambient temperature to 600°C at a heating rate of 10°C/min. Onset of Thermal decomposition, maximum decomposition temperature and residue were determined for each scan and average data compared.[43]

Flexural tests were performed according to ASTM D790-10 under a three-point configuration to find ultimate strength and young modulus of the polymer blend system. The tests were conducted using a Zwick/Roell 2.5 Test System equipped with a load cell of 2.5 kN. All tests were performed at room temperature and displacement control mode. Fracture surface analysis of neat and polymer blend failed samples were examined under a field emission scanning electron microscope (JEOL JSM 5800). Samples were sputter coated with a thin conductive layer for better visualization under the microscope.

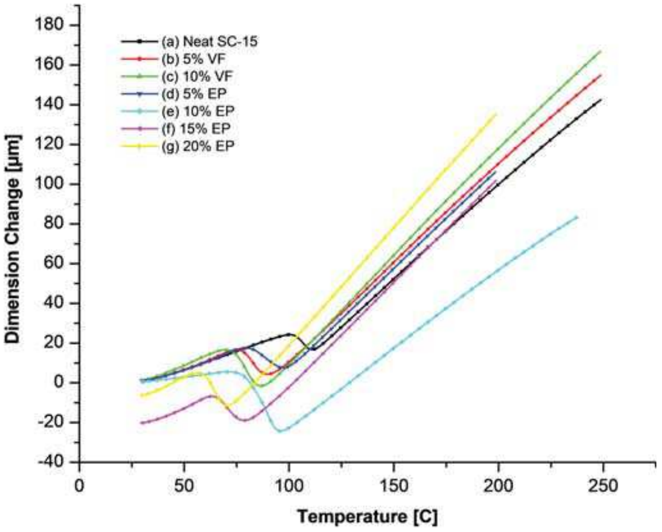

FIGURE 5 Thermomechanical curves of neat and modified epoxy system [Color figure can be viewed at wileyonlinelibrary.com]

3 | RESULTS AND DISCUSSION

Figure 3 shows the FTIR spectra of neat and plasticizer modified epoxy system. Major peaks are observed in four regions. All the major peaks observed are tabulated in Table 1. Region 1 at around 710 cm^{-1} shows the peaks of C—H aromatic mono saturate benzene bands which are indicative of DPGDB plasticizer. It can be observed that with increasing plasticizer EP9009 content the intensity of peak is increasing. The C—N aliphatic amine peaks between 1000 and 1125 cm^{-1} seen in Region 2 (Supplementary Information) are the characteristic FTIR vibrational bands of aliphatic epoxy polymers. In Region 3 it can be observed that the peaks around 1415 and 1455 cm^{-1} corresponds to C—C aromatic bonds, it can be observed that the peak intensities are higher with blends modified with DPGDB plasticizer due to aromatic benzene rings present in them. In Region 4 (Supplementary Information) C—-H alkyl methylene peaks are observed between 2850 and 2950 cm^{-1} this can be attributed to the alkyl polyamine hardener.[36,44]

TABLE 4 Thermomechanical properties of neat and modified epoxy system

Sample	CTE pre T_g (1/°C)	CTE post T_g (1/°C)
Neat SC-15	0.060 ± 0.035	0.195 ± 0.012
5% VF7190	0.079 ± 0.005	0.201 ± 0.003
10% VF7190	0.080 ± 0.027	0.204 ± 0.005
5% EP9009	0.080 ± 0.008	0.188 ± 0.003
10% EP9009	0.070 ± 0.014	0.192 ± 0.004
15% EP9009	0.096 ± 0.011	0.199 ± 0.005
20% EP9009	0.096 ± 0.013	0.202 ± 0.001

3.1 | DMA of neat and modified epoxy system

DMA was performed on neat and modified epoxy systems to evaluate storage modulus at 30°C, loss modulus using peak of loss modulus curve and glass transition temperature using the peak of tan delta curve. It was observed

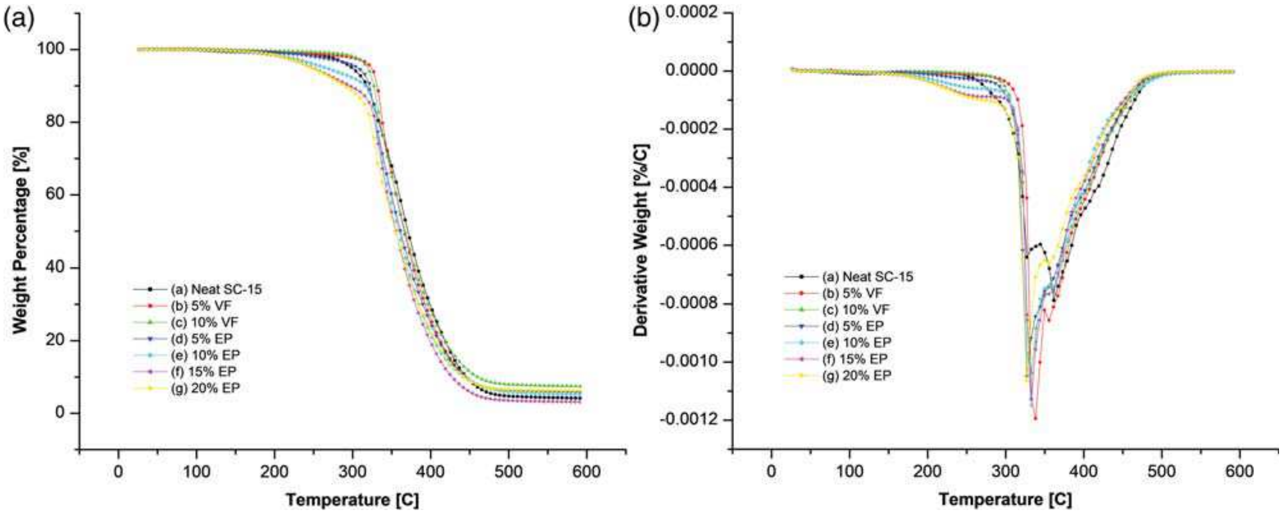

FIGURE 6 Thermal decomposition curves and derivative weight loss curves of neat and modified epoxy system [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 5 Thermal decomposition properties of neat and modified epoxy system

Sample	Onset of decomposition (°C)	50% decomposition (°C)	Residue (%)
Neat SC-15	304.00 ± 5.66	365.00 ± 0.00	5
5% VF7190	324.00 ± 1.41	339.00 ± 0.14	8
10% VF7190	318.00 ± 3.54	334.00 ± 1.41	10
5% EP9009	313.00 ± 3.54	327.00 ± 0.00	8
10% EP9009	270.00 ± 7.07	338.00 ± 0.00	8
15% EP9009	253.00 ± 3.54	331.00 ± 0.00	5

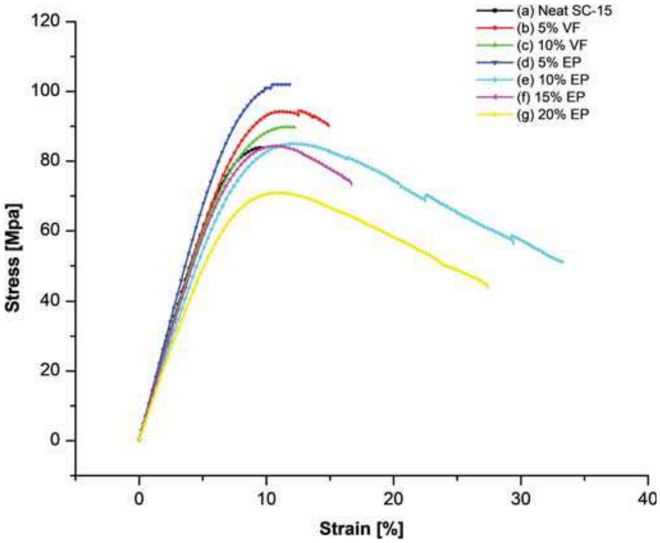

FIGURE 7 Flexural stress vs strain graphs of neat and modified epoxy system [Color figure can be viewed at wileyonlinelibrary.com]

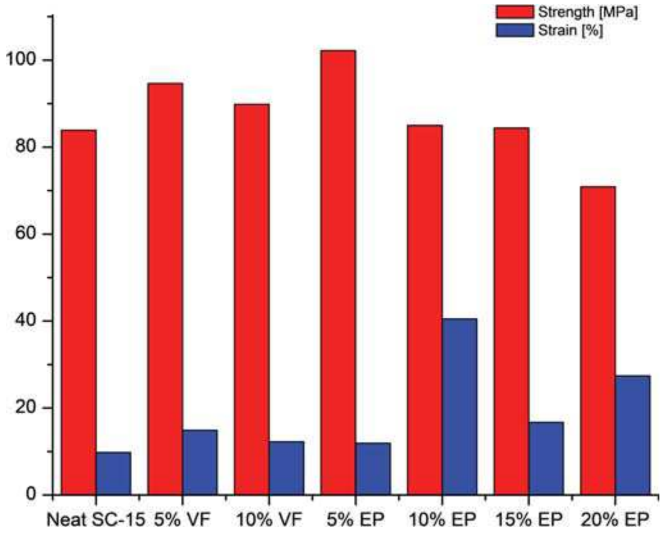

FIGURE 8 Flexural strength and strain comparison of neat and modified epoxy system [Color figure can be viewed at wileyonlinelibrary.com]

that the storage and loss modulus of epoxy modified with epoxidized linseed oil based Vikoflex reduced as shown in Figure 4. The reduction was primarily due to decrease in crosslink density of the epoxy system shown in Table 2. Even though epoxidized linseed oil modification reduces strength and modulus of the material, they are helpful in improving strain and toughness properties of the material.[35,36]

With DPGDB based EP9009 plasticization of epoxy it was observed that storage modulus was almost same as neat for 5 wt% modification; however with 10 wt% EP modification storage modulus increased from 2324.8 MPa for neat to 2500 MPa at 30°C. The increase in storage modulus can be attributed primarily to cross link density which was improved for 10% loading due to good interaction between DPGDB molecules and epoxy chains which may be possible due to homogeneous network formed through primary and secondary amine cross linkage between that of alkyl polyamine and the weight fraction mixture cycloaliphatic amine and DPGDB (Table 3).[45–51] Even loss modulus of 10 wt% modified samples was improved from 234.13 MPa for neat to 262.78 MPa which is an increase of about 12%. Increase in loss modulus indicates that there is some internal damping or energy dissipation effect within the system due to presence of plasticizer. However, with further increase in EP9009 plasticizer loading storage modulus and loss modulus both decreased drastically which can be due to detrimental effect caused by huge phase separations caused by plasticization as shown in Table 2. So it can be inferred that the optimum loading of 10 wt% of plasticizer helps to improve storage and loss modulus of DGEBA based epoxy system.

Glass transition temperature (T_g) was calculated using peak of tan delta curve as shown in Figure 4. It was found that T_g decreased for both ELO modified system and plasticizer modified system. The reason for reduction of T_g with epoxidized linseed oil modified system was due to decrease in cross linking density. In case of DPGDB plasticized system reduction in T_g was mainly due to presence of phases of plasticizer. With increasing temperature, the macromolecules of plasticizer present within the system allows for easy movement of polymer chains resulting in early transition of material from glassy to rubbery.

3.2 | TMA of neat and modified epoxy system

TMA was performed to understand the coefficient of thermal expansion of the modified polymers systems pre and post glass transition. CTE was calculated using slope of the graph before and after transition (Figure 5). It can be observed that for 10% DPGDB plasticized epoxy CTE before transition changed by minimum amount 16%. This could be mainly attributed to crosslinking density. Even from TMA analysis it seems that there is an optimum interaction between plasticizer and epoxy system for 10% EP9009 loading (Table 4). It has been reported in literature that with favorable optimum interaction, there will be increase in transition temperatures (Tg) and decrease in expansion coefficients.[45–51]

TABLE 6 Flexural properties of neat and modified epoxy system

Sample	Flexural modulus (GPa)	Percentage change	Flexural strength (MPa)	Percentage change	Strain at failure (%)	Percentage change
Neat SC-15	2.65 ± 0.06	—	83.89 ± 3.50	—	9.81 ± 2.99	—
5% VF7150	2.75 ± 0.09	3.77	94.64 ± 2.72	12.81	14.91 ± 1.06	51.98
10% VF7150	2.55 ± 0.07	−3.77	89.86 ± 1.36	7.12	12.26 ± 2.84	24.97
5% EP9009	2.63 ± 0.19	−0.75	102.19 ± 0.42	21.81	11.94 ± 3.08	21.71
10% EP9009	2.68 ± 0.16	1.13	84.99 ± 2.33	1.31	40.49 ± 10.38	312.74
15% EP9009	2.48 ± 0.06	−6.42	84.39 ± 1.13	0.58	16.72 ± 3.03	70.44
20% EP9009	2.07 ± 0.07	−21.89	70.91 ± 1.26	−16.33	27.40 ± 2.83	179.31

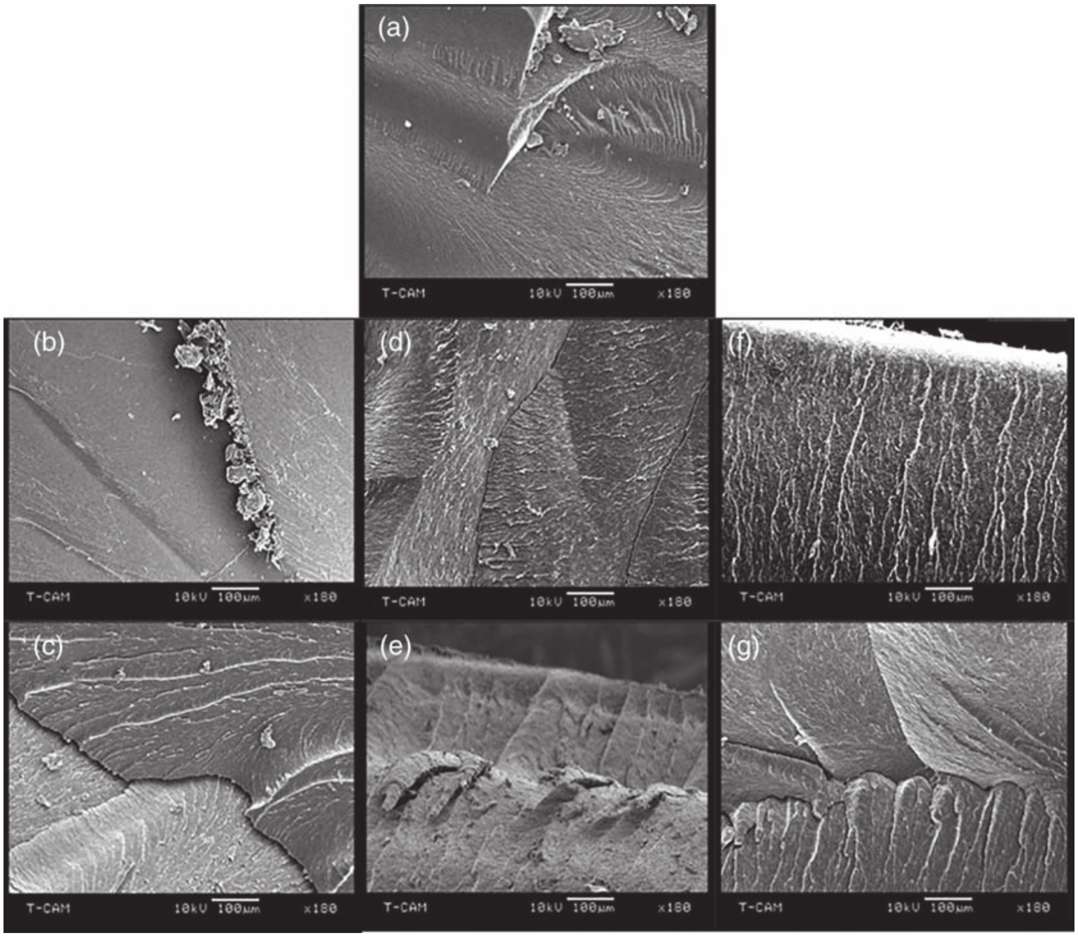

FIGURE 9 SEM micrographs of neat and modified epoxy system (a) neat SC-15 (b) 5% VF (c) 10% VF (d) 5% EP (e) 10% EP (f) 15% EP (g) 20% EP modified epoxy

3.3 | TGA of neat and modified epoxy system

TGA was performed to understand the decomposition behavior of neat and modified epoxy system. Onset of decomposition was calculated from 5% weight reduction. It was found that initial decomposition was slightly delayed with epoxidized linseed oil modified epoxy system by upto 20°C (Figure 6). Whereas for DPGDB plasticized epoxy system for 5 wt% loading onset of decomposition by about 13°C; however, with further increase in plasticizer loading initial decomposition temperature was reduced which means poor thermal stability of the epoxy system. The reason for such thermal properties of plasticized epoxy could be due to inherent poor thermal stability of the plasticizer. Rate of maximum decomposition temperature calculated from derivative weight loss curve for all the samples occurred around 330–350°C as shown in Figure 6. 50% decomposition temperature for all the modified epoxy system was lower than that of neat epoxy. Thus it can be inferred from the TGA results that overall thermal stability of the plasticized epoxy system was reduced by some extent (Table 5).

3.4 | Flexural properties of neat and epoxy blend system

Flexural properties of neat and modified epoxy samples were determined by three-point bending tests. Neat DGEBA samples show a very brittle failure behavior. It was observed that strain (flexural) increased for all the modified samples when compared with neat system as shown in Figure 7. The primary reason for such improvement may be due to reduction in 3D crosslinking density and effective bonding between epoxidized linseed oil and amine hardener of the epoxy system. For DPGDB plasticized epoxy system improvement in strain values are primarily due to phase separation induced due to dispersed plasticizer. The plasticizer which is relatively less rigid when compared to epoxy cross-linked chain allow themselves to be stretched under application of load thus showing plastic behavior before failure. Such behavior not only increases the ductility of the material but sometimes can also increase strength for optimum loading. It was found that modulus reduced for all the modified epoxy system except for 10%EP9009 samples which is primarily due to effect of crosslink density. Flexural strength increased by about 21% for 5%EP9009 samples. It can be concluded from flexural studies that various flexural properties like strength and strain can be achieved from different percentage of plasticization depending on the application. Figure 8 shows effect of different plasticization on strength and strain properties of epoxy system (Table 6).

3.5 | Fracture analysis of neat and epoxy system

Scanning electron micrographs were studied to examine the failure behavior of neat and modified epoxy system. It can be observed from SEM images that for neat (Figure 9(a)) there was little or no resistance to failure with a smooth surface indicating a brittle failure. Whereas for epoxidized linseed modified samples (Figure 9(b),(c)) there appears to be some kind of resistance to failure, the main reason for this resistance could be bonding between epoxidized linseed oil and cycloaliphatic amine groups which leads to increased strain rate shown in flexural results. For DPGDB plasticized epoxy it can be observed from micrographs that all of them show some kind of resistance to failure which translates to improved strain behavior (Figure 9 (d)–(g)). The maximum amount of resistance can be observed for 10%EP9009 sample which has demostrated maximum strain behavior for flexural tests.

4 | CONCLUSIONS

DGEBA based epoxy resin was modified with DPGDB based plasticizer to overcome its sudden brittle failure problem. It was found that optimum amount of plasticization with DPGDB could result not only in ductility improvement but also in strength and modulus improvement which is not possible through traditional toughening mechanisms. Improvement in crosslinking density was the main mechanism which led to such enhanced properties. With 10%EP9009 plasticizer loading improvement in storage and loss modulus of modified epoxy system was observed. Flexural strength increased by 21% at 5%EP9009 loading, and strain% improved by 300% at 10% EP9009 plasticizer loading. It can be concluded from the present study DPGDB based plasticizer can be used to improve strain properties of the material without compromising its other mechanical properties. DPGDB based plasticizer are better when compared to epoxidized linseed oil for mechanical performance of the material.

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CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

AUTHOR CONTRIBUTION

Chinedu Okoro: Synthesis, testing, analysis and writing-original draft. Zaheeruddin Mohammed: Writing-reviewing and editing. Shaik Jeelani: Supervision. Vijaya Rangari: Conceptualization, Supervision, Writing- review and editing.

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