Effect of Epoxidized Jatropha Oil on the Cure, Thermal, Morphological and Viscoelastic Properties of Epoxy Resins

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Received September 17, 2015; Accepted November 16, 2015

ABSTRACT: This article reports the effect of epoxidized jatropha oil (EJO) on the thermal, cure and viscoelastic properties of epoxy resins. Epoxidized jatropha oil with an oxirane value of 5.0 was prepared and epoxy formulations containing different concentrations of EJO were evaluated for cure, morphology, thermal and viscoelastic properties. The curing temperature of the formulations increased with increasing EJO content. The glass transition temperature of the cured films decreased from 56 °C for unmodified epoxy resin to 23 °C for the sample with 60 wt% EJO reactive diluent, suggesting good plasticizing action. The thermal decomposition was only marginally affected.

KEYWORDS: Jatropha oil, plasticizer, biobased epoxy, curing, viscoelastic properties, thermal properties, morphology

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1 INTRODUCTION

Vegetable oils are widely used as renewable raw materials for the preparation and modification (e.g., as plasticizers or stabilizers) of different polymers [1–9]. Utilization of vegetable oil-based monomers in industrial application offers several beneficial features, like renewability, biodegradability and reduced toxicity, over the synthetic additives currently used [2,8]. Epoxy polymers are a versatile class of materials that form network structure on curing and are used in many structural applications. They possess several advantageous properties like high wettability, good mechanical properties (viz., high modulus and tensile strength), high chemical resistance and a wider service temperature range [9–11]. However, when reacted with a stoichiometric amount of polyfunctional amines as curing agent, the high degree of crosslinking makes the cured epoxy network a very brittle material. Several properties, like peel and impact strength, come down and, therefore, limit its application. Hence, modification of epoxy resin has been the subject of immense research interest.

Several approaches have been known to improve the toughness of cured epoxy systems [12–18]. The toughening of epoxy resins with nitrile- and carboxyl-terminated acrylonitrile-butadiene liquid (CTBN) elastomers was reported to develop potential high-performance adhesives because of their durability and ability to prevent catastrophic failure of the structural joints [13,14]. The liquid rubber having functional end groups reacts with the epoxy resin during curing [13]. Also, in the initial stages, these rubbers will be compatible with epoxy-hardener mixture. When cured, a two-phase system having rubber particles dispersed and bonded to the epoxy matrix was obtained, which shows a very high crack resistance without significant deterioration of other thermomechanical properties. The use of vegetable oils or epoxidized vegetable oils for modification of epoxy resins has also been reported [19–21]. The evaluation of epoxidized soybean oil as a reactive diluent for epoxy resins has been reported [21]. They lead to the production of flexible materials with good mechanical properties like impact strength.

As compared to soybean oil, the nonedible oils such as karanja (*Pongamia glabra*) and jatropha (*Jatropha curcus*) are potential raw materials for the biodiesel industry in India. These oils can also serve as renewable feedstock for the polymer industry after appropriate chemical transformation methodologies are adopted in the preparation and modification (e.g., as plasticizers or stabilizers). Thus, in this work, we report the cure, thermal, morphological and viscoelastic properties of jatropha oil modified epoxy resins for the first time.

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DOI: 10.7569/JRM.2015.634118

2 EXPERIMENTAL

2.1 Materials

Jatropha oil was procured from a local industry in Hyderabad, India. Aqueous hydrogen peroxide (30%), formic acid (85%), sulphuric acid, acetic acid, acetic anhydride, sodium sulphate and sodium methoxide were purchased from SD Fine-Chem Ltd. (Mumbai, India) and used as such. Hexane, methanol and ethyl acetate were purchased from Industrial Solvents and Chemicals Pvt. Ltd. (Mumbai, India). A commercial sample of epoxy adhesive (Araldite)—consisting of standard epoxy resin (Araldite GY 250, a bisphenol-A-based epoxy resin) with polyamine hardener (Aradur 3224)—from Huntsman Advanced Materials (India) Pvt. Ltd., Mumbai, was used.

2.2 Preparation of Epoxidized Jatropha Oil

Jatropha oil (100 g, 0.12 mol), formic acid, 85% concentration (9.3 ml, 0.24 mol) and the catalyst sulphuric acid (1.3 ml, 2% of the weight of HCOOH) were taken in a three-necked round-bottom flask and the reaction temperature was maintained at 10 °C. Hydrogen peroxide solution, 30% concentration (98 ml, 0.312 mol), was added slowly to the flask under agitation over a period of 1 h. After addition, the contents were stirred at 60 °C for 6 h. Samples were taken periodically and analyzed for oxirane value to monitor the conversion. After complete conversion, ethyl acetate (120 mL) was added and the mixture washed with water until neutral. The product was dried over anhydrous sodium sulphate, then under reduced pressure, to yield the final product with oxirane value (OV) 5.0%.

2.3 Preparation of Epoxy Formulations

Epoxy resin formulations were prepared with the standard epoxy resin, epoxidized jatropha oil (EJO) and amine hardener. The formulations were prepared by direct mixing of the epoxidized jatropha oil into the base resin in different weight ratios. The hardener content was kept constant and the weight ratio of epoxy resin to EJO was varied from 0 to 60 wt%. The formulations were mixed well, cast into a Teflon petri dish and then dried at 85 °C temperature for 3 h in a convection oven. The test specimens of required dimensions were then cut from the cured films for the thermomechanical tests.

2.4 Characterization

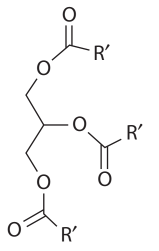
Structural characterization of the epoxidized jatropha oil was carried out with NMR spectroscopy. ^{1}H NMR spectra were obtained using a Bruker AR X 400 spectrometer (400 MHz). ^{13}C NMR spectra were recorded in $CDCl_3$ on a Varian spectrometer (700 MHz). IR spectra were recorded on a Perkin Elmer (Model: Spectrum BX; Connecticut, USA) FT-IR spectrometer neat as thin film. The spectra were measured at a resolution of 4 cm^{-1} at room temperature in the transmission mode and a minimum 8 scans were signal averaged. The hydroxyl value was determined by AOCS Official Method Cd 13–60, iodine value by Cd 1–25 and oxirane value by Cd 9–57. The curing temperature was measured on a Bohlin rheometer (model MAL1030829) from Malvern Instruments, U.K., in parallel-plate geometry. The heating rate used was 5 °C/min.

2.5 Thermomechanical Characterization of Cured Films

The DSC analysis was carried out with a Q100 machine from TA Instruments, USA. The heating rate used was 10 °C/min in N_2 atmosphere. Thermogravimetric analysis of the sample was conducted on a TA Q500 instrument at a heating rate of 10 °C/min in N_2 atmosphere to study the effect of adding EJO on the thermal stability. The viscoelastic properties of the cured system were evaluated using a dynamic mechanical thermal analyzer (DMTA model, Mark IV, Rheometric Scientific) at a fixed frequency of 1 Hz. The heating rate used was 3 °C/min using liquid nitrogen for subambient conditions. Dynamic storage modulus, loss modulus and loss factor were obtained in rectangular tension mode for the sample size of 10 × 8 × 0.25 mm.

3 RESULTS AND DISCUSSION

The triglyceride oil from Jatropha is composed of the unsaturated fatty acids, oleic acid (45.7%) and linoleic acid (33%), along with the saturated fatty acids, palmitic (14%) and stearic acid (7%), and the structure is schematically represented in Figure 1 [22].



R (x:y) =	16:0	18:0	18:1	18:2	20:0
Jatropha oil	14	7	45.7	33	0.3

Figure 1 Chemical structure of triglyceride and typical composition of jatropha oil in % (R (x:y) = composition of the fatty acids; x = chain length of carbon atoms; y = number of double bonds); $R \neq R^1$.

Table 1 Composition and other details of the EJO modified epoxy resin samples.

Sample code	Resin (wt%)	Hardener[a] (wt%)	EJO[b] (wt%)
RE011	100	100	0
RE145	80	100	20
RE235	60	100	40
RE325	40	100	60

[a] The resin and hardener were taken on equal part by weight.
[b] Based on total resin wt%.

Epoxidized vegetable oils like soybean are widely used as plasticizers in epoxy resins. We performed epoxidation of jatropha oil with peracid generated *in situ*. The epoxidized oil was then used as a reactive diluent in standard epoxy resin formulations. The saturated fatty acids cannot take part in the polymer network formation. The preparation of EJO is described first, followed by its evaluation as a reactive plasticizer in epoxy resins. Epoxy resin formulations containing different weight percentages of EJO, namely 0, 20, 40 and 60 wt%, were prepared and characterized. The formulations used for the study as well as the sample codes are summarized in Table 1.

3.1 Synthesis and Characterization of Epoxidized Jatropha Oil (EJO)

Jatropha oil with iodine value 92 was epoxidized using performic acid generated *in situ* through reaction of formic acid and hydrogen peroxide using sulphuric acid catalyst. The epoxidized oil was obtained in 96 wt% yield with oxirane value 5.0 and characterized by IR, ^{1}H NMR and ^{13}C-NMR analysis. The infrared spectrum of jatropha oil before and after epoxidation showed characteristic aliphatic C-H stretching at 2926 cm^{-1}. The ester carbonyl absorption appeared at 1746 cm^{-1} and the characteristic triglyceride ester triplet at 1238, 1162 and 1110 cm^{-1} was seen. In addition, the epoxidized oil showed distinct absorption bands at 823 cm^{-1}, characteristic of the C-O-C oxirane stretch.

The ^{1}H-NMR spectrum of epoxidized oil, shown in Figure 2a, indicates complete conversion of carbon–carbon double bonds. The characteristic peaks due to olefinic and allylic hydrogen atoms of jatropha oil at chemical shift values 5.3–5.4 and 2.6–2.8 ppm, respectively, are absent. New peaks appear in the region of 2.8–3.1 ppm, suggesting the presence of epoxy group protons.

The epoxide formation was also confirmed by ^{13}C-NMR (cf. Figure 2b). The characteristic peaks of epoxy carbons appear at 54–58 ppm and the olefinic carbon peaks at 128–130 ppm disappeared. The obtained results are in agreement with the structure of epoxidized jatropha oil reported earlier by Akbar *et al.* [22]. The important physicochemical characteristics of EJO are summarized in Table 2.

In order to study the effect of epoxidized jatropha oil (EJO) on the curing behavior, epoxy formulations with varying wt% EJO were studied. The viscosity-temperature plot given in Figure 3 shows that the curing temperature increases from ~ 103 °C to 110 °C and then to 120 °C as the EJO content was increased from 20 wt% to 40 wt% and then to 60 wt%, respectively. Correspondingly, the viscosity also decreased with incorporation of excess EJO. The viscosity for 20 and 40 wt% mix was about 30000 Pa s but decreased to ~ 8000 Pa s for the sample with 60 wt% EJO (see Figure 3C). Thus, the results show that higher concentrations of EJO affect the properties badly, even though the reactive diluent takes part in the curing process.

The reduction in viscosity also influenced the mechanical properties like modulus, as seen from the dynamic mechanical studies.

3.2 Morphological Characterization of the Cured Films

The cured films were characterized for morphology using scanning electron microscopy (SEM). Figure 4 shows the SEM images of EJO modified and unmodified samples. No phase separation was noticed on addition of EJO to the epoxy resin, which suggests the formation of a homogeneous network. In the unmodified sample a brittle fractured surface is observed (the film also appeared brittle) with needle-like inclusions, whereas the epoxidized jatropha oil modified samples suggested a toughened and soft film. The effect of triglyceride oils, like epoxidized soybean oil, has also been reported by Czub, who observed very good miscibility when incorporated in the epoxy matrix [21].

3.3 Thermomechanical Properties of EJO Modified Epoxy Networks

Thermal properties of the epoxy composites play an important role, but few studies on the thermal property-composition behavior of conventional epoxy resin/epoxidized vegetable oil (EVO systems) have been reported [23]. In order to study the effect of the modifier concentration on the properties of the cured network we prepared epoxy networks containing different concentrations of EJO and studied its thermal and dynamic mechanical properties. The DSC curves of the samples cured with 0, 20 and 40 wt% EJO are shown in Figure 5.

No exothermic peaks, characteristic of the epoxy curing reactions, were detected on the DSC heating

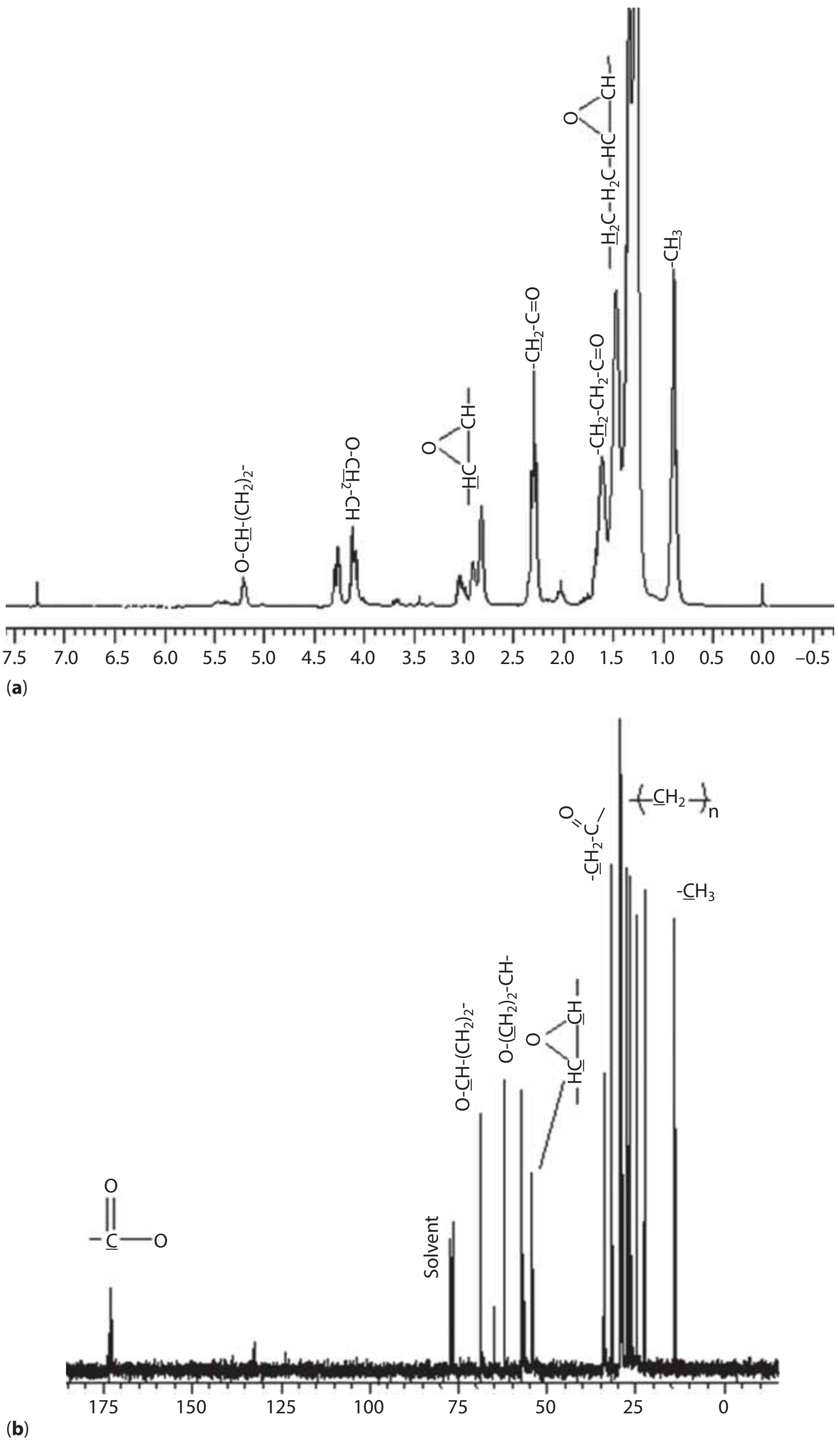


Figure 2 ^{1}H- (**a**) and ^{13}C-NMR (**b**) spectra of epoxidized jatropha oil (EJO) in $CDCl_3$.

Table 2 Physicochemical properties of jatropha oil and epoxidized jatropha oil.

Properties	Density at 15 °C (g/cm³)	Kinematic viscosity (cSt) at 40 °C	Refractive index at 40 °C	Oxirane value, %
Jatropha oil (JO)	0.9188	40.69	1.4744	-
Epoxidized jatropha oil (EJO)	0.9761	146.52	1.4720	5.0

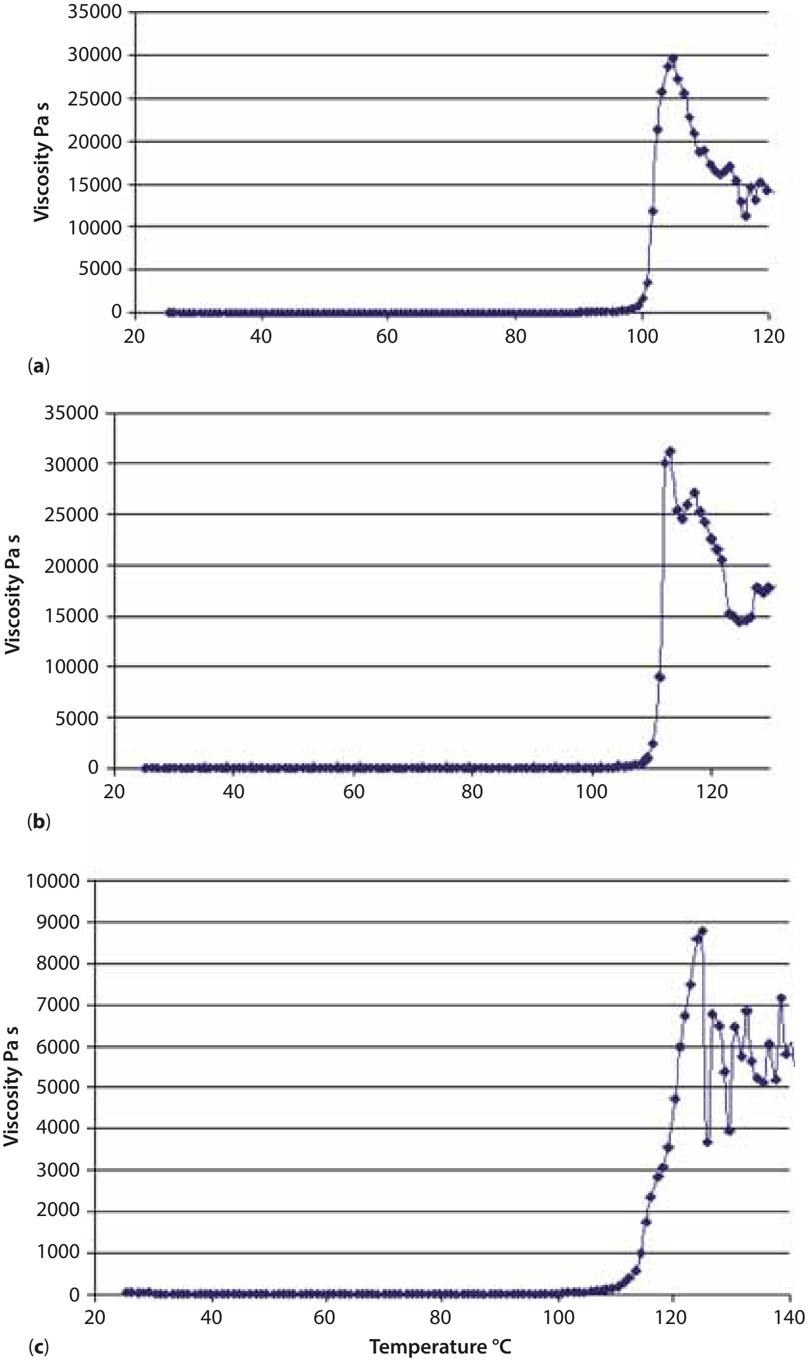


Figure 3 Viscosity-temperature plots showing effect of EJO content on the curing temperature of epoxy formulations. (**a**) 20 wt% EJO content, (**b**) 40 wt% EJO content and (**c**) 60 wt% EJO content.

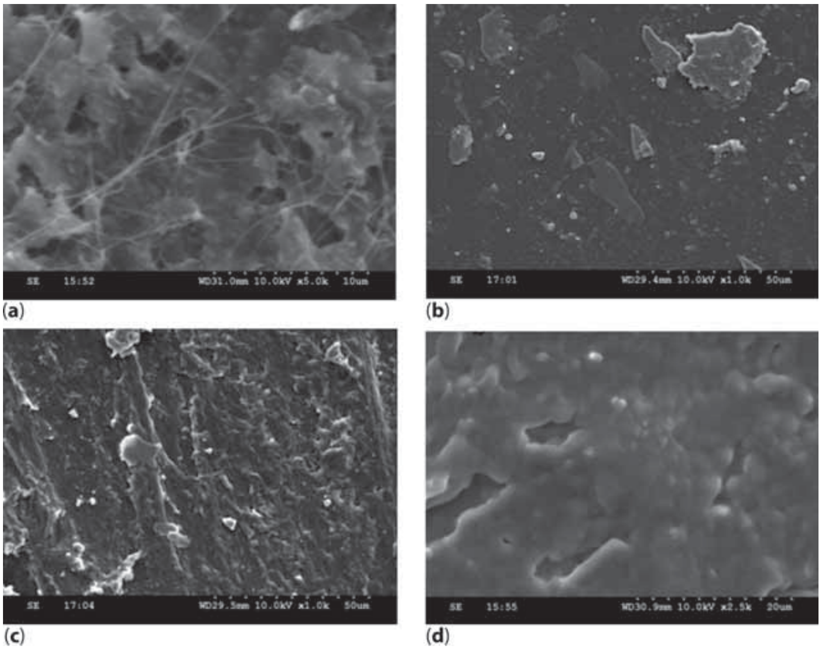


Figure 4 SEM photographs of the fracture surfaces of (**a**) unmodified epoxy, (**b**) modified network with 20 wt% EJO, (**c**) modified network with 40 wt% EJO and (**d**) modified network with 60 wt% EJO.

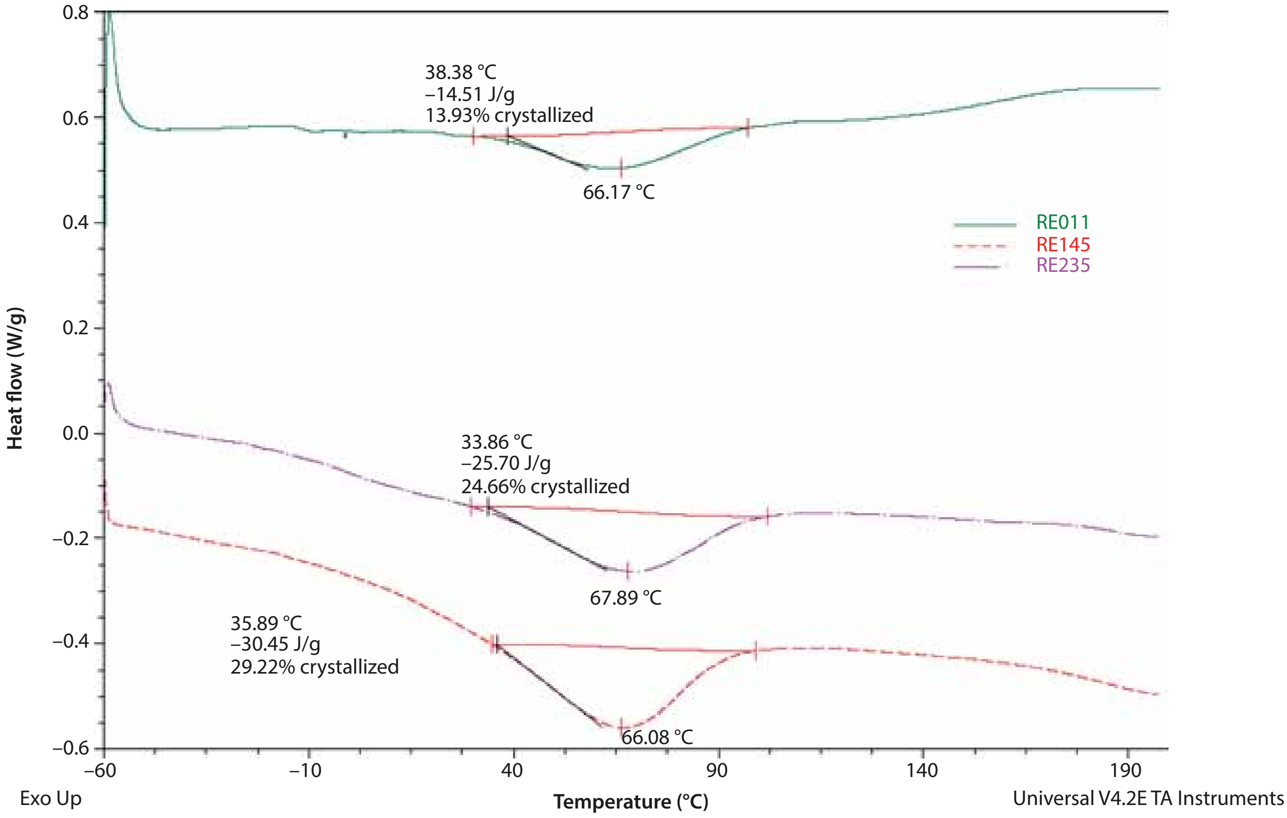


Figure 5 The DSC thermograms showing the non-isothermal thermal profile of EJO-cured epoxy resin samples.

curves of the films. This suggested complete curing of the film samples. Instead, an endothermic peak was observed, which broadened on increasing the concentration of the EJO. This may be due to the melting of the crystalline phases formed from the aliphatic chain segments as the crystalline nature of the aliphatic segments in vegetable oils has been earlier reported [24]. The enthalpy values also showed differences. The EJO modified sample cured with higher oil content shows a lower enthalpy value. No multiple transitions are observed in DSC, suggesting the absence of EJO homopolymerization. Another finding was that the peak temperature of standard epoxy/EJO modified system becomes broader or the transition temperature is broadened by the addition of EJO.

The dynamic mechanical properties of oil modified epoxy resins have also been widely reported [25,26]. The utilization of epoxidized soybean oil (ESO) as reactive diluent in bisphenol-A-based epoxy resin was studied by Piotr Czub [21]. He observed excellent plasticization and improvement of impact strength when the ESO content was 60 wt%. Tan and Chow [25] used DMTA technique to study the curing of ESO using tetraethyl ammonium bromide (TEAB). They observed that ESO with higher TEAB possesses higher storage modulus, glass transition temperature and thermal stability. Gerbase *et al.* [26] have studied the dynamic mechanical and thermal behavior of epoxy resins based on soybean oil and found that the cured resins exhibited thermal stability up to 300 °C. The effect of different curing agents on the properties has also been reported [27]. Film samples containing varying amounts of EJO were analyzed by DMTA analysis from −50 to 200 °C. The viscoelastic properties E′, E″ and tan δ are recorded as a function of temperature. The loss factor (tan δ) variation is shown in Figure 6 and all the samples show only one transition attributed to the α-relaxation of the epoxy network. The transition peak temperature shifted successively to lower temperatures as the amount of EJO was increased. The width of the relaxation in all the cases was found to be broad and transition temperature extending to lower temperatures as the EJO content is increased.

Thus, as the EJO content of the sample increases, the glass transition temperature decreases from 57 °C for unmodified epoxy to 23 °C for epoxy with 60 wt% of EJO. The magnitude of the tan δ peak is indicative of the damping properties. The higher the tan δ value, the better the damping properties. Thus, the damping properties increase at the same time as the T_g is decreased. The effect of EJO content on the modulus characteristics are presented in Figure 7.

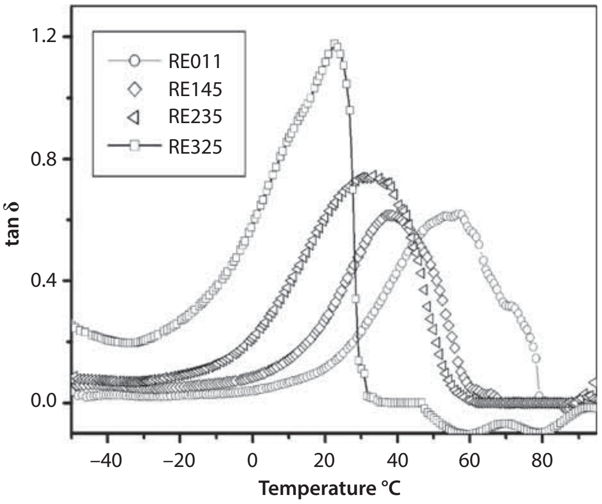


Figure 6 Variation in the glass transition temperature of the epoxy network modified with EJO.

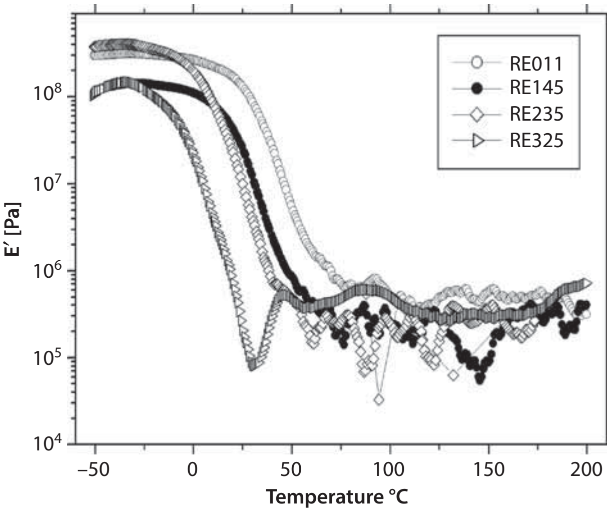


Figure 7 Variation in the elastic modulus of the epoxy network cured with varying concentrations of epoxidized jatropha oil (EJO) diluent.

The elastic storage modulus in the glassy region (at about −30 °C) decreases from 3.13×10^8 Pa for unmodified epoxy to 1.43×10^8 Pa on addition of 60 wt% EJO, a decrease of about 50%. As shown in Figure 7, the maximum in the *E′-T* curve shifts to lower temperature as the EJO concentration in the epoxy thermoset increases from 20–60 wt%. The decrease in modulus was the maximum for the sample with 60 wt% EJO.

3.5 Thermogravimetric Analysis

The thermal stability of anhydride-cured epoxidized soybean oil (ESO) was studied by Liu *et al.* and found to be thermally stable up to 300 °C, followed by decomposition in one step [28,29]. The two-step decomposition

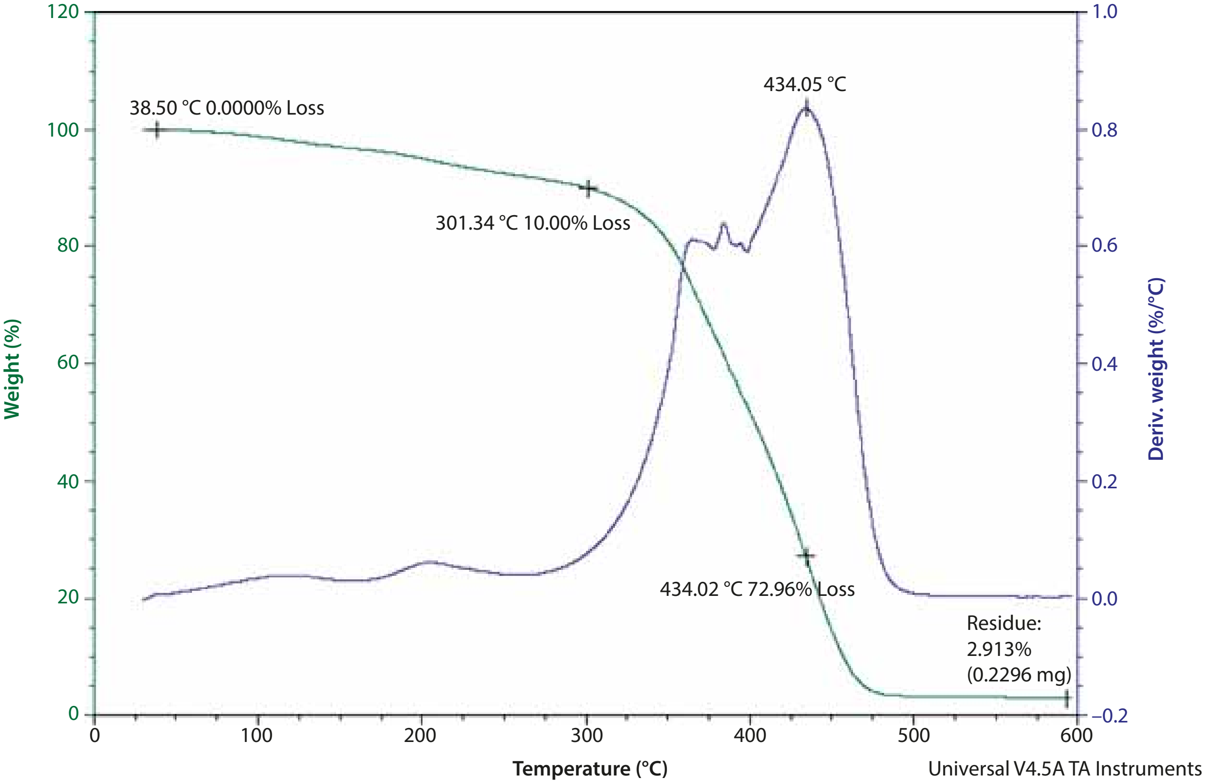


Figure 8 TGA plot showing weight loss characteristics and the derivative plot with the peak decomposition temperatures of epoxy resin modified with 20 wt% epoxidized jatropha oil (EJO).

Table 3 Thermal decomposition temperatures and char yield of epoxy jatropha oil (EJO) modified epoxy compositions as deduced from TGA data.

Composition	IDT1, °C	IDT2, °C	T10%, °C	T max., °C	Char residue, %
RE011	375	431	331	431	5.27
RE145	380	434	301	434	2.90
RE235	375	435	296	435	2.42
RE325	375	441	308	441	1.72

IDT1 = decomposition temperature 1; IDT2 = decomposition temperature 2; T10% = temperature at 10% weight loss; T_{max} = temperature at maximum weight loss.

was attributed to the side-chain decomposition of the anhydride curing agent used. The thermal decomposition pattern of EJO modified epoxy thermosets are studied by thermogravimetry. A typical plot showing the thermogravimetric and derivative curve as a function of temperature for the sample modified with 20 wt% EJO is shown in Figure 8.

It is seen that EJO modified samples experience a two-stage decomposition when being heated up to 600 °C. For detailed analysis, the TGA results for all the samples are summarized in Table 3.

The TGA data shows several features. Maximum weight loss of up to 69–77% decomposition was observed during the second stage of decomposition. The initial decomposition temperature (IDT1) was more or less constant for all the samples. This may be due to the epoxy network with the bisphenol-A-based starting materials. A marginal increase of the second decomposition temperature was observed with increasing the EJO content. The percentage of sample decomposed at the T_{max} shows an increase with the EJO content, from 69% for unmodified epoxy to 77% for epoxy with 60 wt% EJO. Correspondingly, the char yield decreased as the EJO content was increased, suggesting near complete decomposition of the network, unlike conventional epoxy system. The increase in decomposition rate may be due to the reduced thermal stability, suggesting that EJO reduces the thermal stability. The second stage of the decomposition took place in the temperature range 375–441 °C.

In general, as indicated by the T10 values (cf. Table 3), the initial decomposition of cured epoxy resins studied starts around 300 °C (varying from 296–336 °C), followed by decomposition in two steps, the first step (IDT1) at 375 °C and a second one (IDT2) at 441 °C for 60 wt% EJO content. This observation is different from earlier reports, which showed that the thermal decomposition of DGEBA epoxy resin/epoxidized oil mixtures and other epoxy resins [30,31] took place in a single step. However, reports on fish [32] and tung oil [33] modified epoxy polymers, had shown up to three different decomposition stages in air in the temperature range (400–560 °C).

4 CONCLUSIONS

In this work the effect of epoxidized jatropha oil (EJO) on the cure, thermal, morphological and viscoelastic properties of standard epoxy resin has been studied. The epoxidized oil was characterized for its structure and oxirane content. The cure temperature of the epoxy resin increases with increase of EJO content. The formation of a cured network without any phase separation was observed from the SEM micrographs and dynamic mechanical studies. The glass transition temperature of the cured films decreases from 57 °C for unmodified epoxy to 23 °C as the concentration of EJO is increased to 60 wt%, but a decrease of the modulus is observed at higher concentrations. The damping characteristic of the cured sample increases on addition of the EJO. The TGA results show a two-step thermal decomposition pattern and the char yield decreases with increasing EJO content. No major changes were observed in the thermal decomposition characteristics. The study shows that EJO is an economically viable and environmentally friendly toughening agent for epoxy resins. Due to the advances in oleochemistry technology and sustained availability of vegetable oils, the non-food applications of epoxidized vegetable oils will eventually gain popularity.

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