

Mesua ferrea L. Seed Oil-Based Epoxy Resins

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ABSTRACT: Exhaustion of fossil fuels, tremendous increase of materials demand, and unpredictable prices of petroleum based products urge upon the sustainable development. Three different epoxy resins have been synthesized from monoglyceride of *Mesua ferrea* L. seed oil and epichlorohydrin with and without other dihydroxy compound like tetrabromobisphenol-A (TBPA) and bisphenol-A (BPA). The synthesized epoxy resin were characterized by measurement of physical properties like epoxy equivalent, viscosity, hydroxyl value, saponification value, acid value, etc., and spectroscopic techniques like FTIR and ^1H NMR. High thermostability with initial decompositions temperature of 225–265°C was observed for the cured resins and 75 mol % BPA based resin exhib-

its the highest thermostability. Newtonian flow behavior was observed for all resins as indicated by the rheometric study (CVO 100). The flame retardancy rating of TBPA based epoxy was found to be V1 as tested by UL 94. The performance characteristics as coating materials were studied by the measurement of gloss, impact resistance, scratch hardness, tensile strength, elongation at break, adhesive strength, and chemical resistance. The results indicate the suitability of the synthesized resins as coating materials. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 118: 128–134, 2010

Key words: synthesis; *Mesua ferrea* L. seed oil; renewable resources; high performance polymers; thermal properties

INTRODUCTION

The demand for new materials has instigated tremendous exploration of renewable resources in both academics and industries. Depletion of petroleum feed stocks has directed research in polymers to hunt for alternative renewable sources. Vegetable oil is one of the best resources amongst other renewable alternatives.¹ Research and development are being now focused for use of traditional and nontraditional vegetable oils for surface coating applications without jeopardizing the needs of human consumption.² These materials have a number of advantages like physical and chemical stability, aptitude to facile chemical modification, reduced toxicity, possibility of recycling, renewable, and biodegradable and at the same time available in large quantities in low and stable prices. Above all, the plantation will reduce pollution, which is most important for today's society. The use of vegetable oil in production of various industrial polymers significantly reduces green house gas generation that is the prime attraction for the utilization of these materials in the production of

value added products. There are other significance also, like employment of people in cultivation of naturally growing plants, collection of oil seeds, and isolation of oil from the seeds.^{3–5} Seeds like cashew nut,⁶ karanj,⁷ annona squamosa,⁸ and natural rubber,⁹ along with the traditional seeds like linseed, soyabean, coconut, and castor oil have been used successfully for the preparation of various resins. Some recent reviews showed the potential availability of some seed oils and their current utilization.^{10,11} The high oil content (70 wt %) of *Mesua ferrea* L. seeds and suitable fatty acid composition of this oil put forward its utilization for the preparation of various industrial resins such as polyesters,¹² poly (ester amide)s,¹³ polyurethanes.¹⁴ Although there are few reports on synthesis of vegetable oil based epoxy resin including halogenated one,¹⁵ but no report has been found so far on the preparation of epoxy resin (except epoxidized oil) by employing this oil, to our knowledge. So this is the first report on utilization of low cost *M. ferrea* L. seed oil in synthesis of curable epoxy resins using condensation technique, though epoxidized oil of the same oil as a reactive diluent has already been reported.³

For many decades tough and high strength epoxy resin has been one of the prime attractions for application in different fields as binder for adhesives, printed circuit boards, coatings and paints, engineering composites, etc.¹⁶ Hence with this unique combination of properties coupled with outstanding

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formulating versatility and reasonable costs have gained epoxy resins as a material of choice for many applications.^{17,18} However the flammability of this material is one of the major drawbacks, flame retardant epoxy has been reported by incorporation of flame retardant additives like trichlorophenylphosphine, molybdenum oxide, etc.^{19,20} Brominated epoxy resin is one of the most widely used flame-retardant epoxy resins, it is widely used in electronics equipment, computer equipment, paints, etc. The incorporation of tetrabromobisphenol-A (TBPA) offers high performance flame retardant epoxy resins (FRE).^{19,20}

Vegetable oil based epoxy resins have been reported from sunflower oil,²¹ cottonseed oil,²² linseed oil,²³ etc., directly. However, most of the epoxy resins obtained from vegetable oils exhibit poor mechanical properties hence limit their applications.²⁴

Thus in the present study, authors wish to report the synthesis, characterization, and properties of vegetable oil modified epoxy resins. These epoxy resins have been synthesized by using three different composition of diols viz. vegetable oil based monoglyceride, bisphenol-A (BPA), and TBPA. The performance characteristics of these resins were evaluated as coating materials.

EXPERIMENTAL

Materials

M. ferrea L. seeds (Jamugurihat, Assam) were utilized for the collection of the oil. The oil was isolated from the matured seeds, and purified by distillation, degumming, followed by alkali refining and vacuum drying techniques, as reported earlier.¹² Epichlorohydrin (Merck, Mumbai, India) and TBPA (Aldrich, Steinheim, Germany) were used as received. BPA (BG & Co., Mumbai, India) was used after purification by recrystallization from toluene. Glycerol (Merck, Mumbai, India) was used after drying under vacuum at 45–50°C for 12 h. All other reagents used in the present investigation are reagent grade. The purity of the materials varies from 98–100%.

Synthesis of epoxy resin from monoglyceride

Monoglyceride of the oil was prepared by the conventional method as reported earlier.¹²

Monoglyceride based epoxy resin

Eight grams (0.0230 mol) of monoglyceride and 8.51 g (0.092 mol) of epichlorohydrin were taken in a three necked round bottom flask fitted with a thermometer pocket, a condenser, and a nitrogen inlet. The mixture was mixed together and refluxed for 1 h. The reaction temperature was maintained at the

temperature (115°C ± 5°C), and to this reaction mixture, 2.76 g of NaOH in the form of 30% aqueous solution was added at slow rate such that it is completed within 1 h. The reaction was then continued for 14–16 h. After this period of time of the reaction, the product was separated by a separating funnel, washed with brine solution and followed by distilled water. Then the content was vacuum dried to obtain the product with 80–90% yield.

Monoglyceride and bisphenol-A based epoxy resin (MGBP AE)

Similar resinification reaction was carried out with required amount of BPA along with monoglyceride by maintaining the mole ratio at 75 : 25, while the mole ratio of epichlorohydrin has been kept same as above. The reaction was carried out for 14 h keeping all other conditions same as mentioned above.

Flame retardant epoxy resin

A flame retardant epoxy resin (FRE) was prepared from monoglyceride of the oil, BPA, and TBPA with epichlorohydrin. Similar resinification reaction was carried out with required amount of BPA, TBPA along with monoglyceride by maintaining the mole ratio at 50 : 25 : 25, with epichlorohydrin mole ratio kept as same as above. All other conditions remain the same, as above.

Curing of the resins

A homogenous mixture of the resin (FRE, Monoglyceride based epoxy resin (MGE), and Monoglyceride and bisphenol-A based epoxy resin (MGBP AE) separately) with 50 phr (parts per hundred grams of resin) of poly(amido amine) hardener was prepared in a glass beaker at room temperature by hand stirring for 20 min. The thin film of the mixture was casted on a glass plate, degassed for 15 min under vacuum, and then heated at 100°C in a muffle furnace to determine the touch free time (minimum time, when no impression will appear on touching the film) and hard dry time of the resin. The mixtures were also uniformly casted on mild steel plates (150 mm × 50 mm × 1.60 mm), tin plates (150 mm × 50 mm × 0.40 mm), and glass plates (75 mm × 25 mm × 1.75 mm) for impact resistance, gloss, and chemical resistance tests. The plates were cured at 100°C for specified period of time as mentioned in Table I. The dry film thickness was measured by a pen tester (Sheen Instrument, UK, Table I).

Measurements

FTIR spectra of resins were recorded by FTIR spectroscopy (Impact-410, Nicolet) using KBr pellet.

TABLE I
Physical Properties of the Resins

Property	FRE	MGBPAAE	MGE
Acid value (mg KOH/g)	20	18	15
Saponification value (mg KOH/g)	220	240	268
Hydroxyl value (mg KOH/g)	242	254	266
Epoxy equivalent (g/eq. epoxy group)	225	463	784
Specific gravity at 25°C	1.4	0.85	0.8125
Touch free (min)	30	60	840
Hard dry (min)	60	120	300

^1H NMR spectra of the resins were recorded with 400 MHz FT NMR (Varian) spectrometer by using CDCl_3 as the solvent and TMS as the internal standard. The viscosities of the resins were measured by rheometer (CVO100, Malvern, UK) using parallel plate (pp 20). The relationships between viscosity and time at constant stress and single shear, viscosity with temperature gradient at constant stress of the resin were determined by using the above instrument.

The bromine content of FRE was determined by Schoniger Oxygen Combustion method (S.C. Dey & Co). The bromine content was calculated by the following equation

$$N_{\text{Br}\%} = (V_1 - V_2)N \times 79.92 \times 100 / (w \times 1000)$$

Where V_1 is the volume of AgNO_3 solution for titration of polymer and V_2 is volume of AgNO_3 solution for blank titration, N is the concentration of AgNO_3 solution, and ' w ' is the amount of polymer used in gram.

The mechanical properties of the cured films of the resins such as tensile strength and elongation at break (as per the ASTM D 412-51 T) and adhesive strength (lap shear adhesion method by using plywood as the substrate) were measured with the help of universal testing machine (Zwick Z010, Germany). The scratch hardness by using scratch hardness tester (Sheen instrument, UK) and impact resistance by impact tester (S.C. Dey Co., Kolkata, India) of the cured films were measured. The gloss characteristic of the cured films was found out by using mini glossmeter (Sheen instrument, U.K.), over resin coated mild steel plate at an angle of incidence, 60° . All these tests were done according to Indian Standard, Methods of Sampling and Test for Paints, Varnishes and Related Products 101, 1988.

The flame retardancy of the polymers was done by UL 94 test method.²⁰ The samples were cut into $5'' \times 12'' \times 0.037''$ then the specimens were mounted vertically such that the gap between a surgical cotton placed directly below the specimen is $12''$. A pilot flame was introduced to the specimen at an

angle of 45° for 10 s. A total of 5 tests were done for each specimen and average result was taken.

Thermogravimetric (TG) analysis was carried out in Shimadzu TG 50 using the nitrogen flow rate of 30 mL/min and at the heat rate of $10^\circ\text{C}/\text{min}$.

The chemical resistance of the cured films was performed in different chemical environments. Glass plates coated with films were kept in 250 mL beakers containing 150 mL of different chemicals for 10 days and then observed for any change on the films.

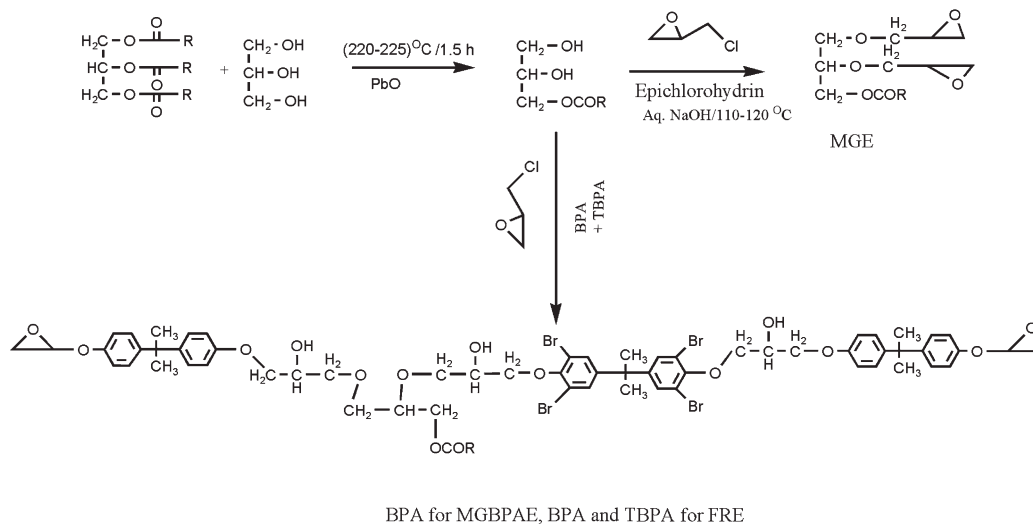
RESULTS AND DISCUSSION

Resinification

Synthesis of epoxy resin from the monoglyceride of *M. ferrea* L. seed oil has been carried out by using monoglyceride of the oil as one of the components to utilize a low cost product to a value added industrial resin. Three epoxy resins of the oil were prepared by condensation reaction of diol like monoglyceride, combination of monoglyceride and BPA, and combination of monoglyceride, BPA, and TBPA with epichlorohydrin separately (Scheme 1). The addition of aqueous NaOH was done in such a manner that the color of phenolphthalein should not disappear, which otherwise forms undesirable products.²⁵ The hydroxylation reaction of the epoxy group accelerated with increase of the reaction time from the optimum, so the reactions should not be performed for over time (>14 – 16 h). It was also observed that with the increase of the reaction time the viscosity of the resin increase, so the reactions were not carried out for less than 14–16 h. By using this time period adequate viscosity with good epoxy equivalent of the resins was found. Thus the resins were obtained with relatively good yield (80–90%) under the used reaction conditions. No noticeable difference was observed during resinification reaction compared to the conventional BPA based epoxy resin due to the use of monoglyceride.^{16,17}

Structural analysis of FRE, MGE, and MGBPAAE

The FTIR spectra of FRE, MGE, and MGBPAAE are presented in Figure 1. The bands for asymmetric vibrations for epoxy ring appeared at 912 – 834 cm^{-1} for FRE, 928 – 832 cm^{-1} for MGBPAAE, and 926 – 831 cm^{-1} for MGE.²⁶ The appearance of sharp bands at 740 cm^{-1} and 660 cm^{-1} indicates the stretching vibrations of C–Br at the aromatic ring for FRE. The presence of a strong absorption band at 1038 cm^{-1} for FRE and 1042 cm^{-1} for MGBPAAE confirmed the presence of alkyl-aryl ether group in the molecule. Similarly, a strong absorption band at 1242 cm^{-1} for FRE and 1243 cm^{-1} for MGBPAAE confirmed the



R=Hydrocarbon part of mixture of oleic, linoleic, stearic and palmitic acids

Scheme 1 Synthesis of epoxy resins.

presence of aryl ether linkage in the structure of the resins.¹⁶ The C=C stretching vibrations for FRE, MGBPAAE, and MGE were observed at around 1609–1458 cm^{-1} , 1609–1461 cm^{-1} , and 1607–1457 cm^{-1} , respectively. Bands at 1734 cm^{-1} for FRE, 1733 cm^{-1} for MGBPAAE, and 1730 cm^{-1} for MGE in the spectra were observed due to the C=O stretching vibrations of triglyceride esters in the resins. The presence of bands at 3437 cm^{-1} , 3421 cm^{-1} , and 3415 cm^{-1} for FRE, MGBPAAE, and MGE, respectively are due to the O–H stretching vibrations.²⁵

The ^1H NMR spectra of FRE, MGBPAAE, and MGE are shown in the Figure 2. Chemical shifts values at $\delta = 2.7\text{--}2.9$ ppm, 2.7–2.8 ppm, and 2.5 ppm are due to epoxy proton for FRE, MGBPAAE, and MGE, respectively.¹⁶ In the desired resins the shift values

at $\delta = 6.82$ and 6.80 ppm for FRE and 6.8 and 7.1 ppm for MGBPAAE are due to the presence of two types of proton in the BPA ring.^{16,25} The higher value is due to ortho proton to isopropylidene and lower value is due to ortho proton to ether linkage in both the cases. But it is seen that the peak values are absent in MGE which suggests the presence of BPA moiety in both FRE and MGBPAAE. The CH_2 protons attached with ether linkages of aliphatic and aromatic unit in the resins were observed at $\delta = 3.3\text{--}3.38$, 3.34, 3.5–3.7 ppm and $\delta = 3.5\text{--}3.6$, 3.7–3.8, 3.8–3.9 ppm for FRE, MGBPAAE, and MGE, respectively. All the protons of monoglyceride are also observed in the ^1H NMR spectra. The peaks at $\delta = 0.85\text{--}0.88$

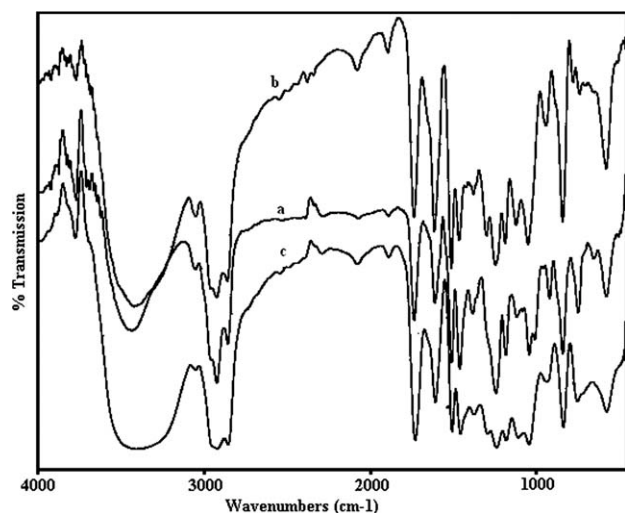


Figure 1 FTIR spectra for (a) FRE, (b) MGE, and (c) MGBPAAE.

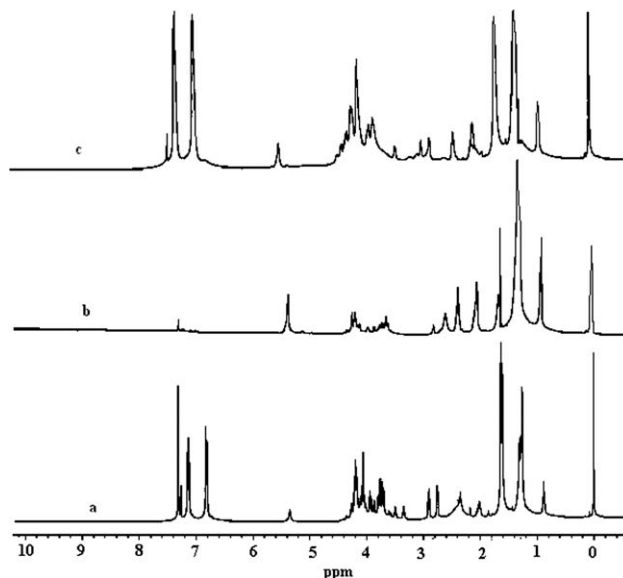


Figure 2 ^1H NMR spectra for (a) FRE, (b) MGE, and (c) MGBPAAE.

ppm are for terminal methyl group of the fatty acids chains. The peaks at $\delta = 1.25\text{--}1.30$ ppm are for the protons of all the internal CH_2 groups present in the fatty acid chains. The peak for protons of unsaturated moiety appear at $\delta = 5.3\text{--}5.4$ ppm. The CH_2 protons attached with the double bonds are found at $\delta = 1.6\text{--}1.62$ ppm, whereas the protons for CH_2 attached with ester groups were observed at $\delta = 4.0\text{--}4.3$ ppm.^{12,13}

Physical properties of resins and their solubility

The physical properties like acid value, saponification value, hydroxyl value, and epoxy equivalent of the resins are given in Table I. The saponification value decreases with the decrease of amount of monoglyceride content in the resins (Table I) as it is the only component containing ester linkage. FRE has the lowest epoxy equivalent value, whereas MGE has the highest. The results are again due to the variation of monoglyceride content. As the monoglyceride may also contain some other components like triglyceride, diglyceride, etc.,²⁷ so the presence of epoxy groups is less in the structure (Table I).

The resins are soluble in common polar solvents such as THF, CHCl_3 , xylene, toluene, etc., as well in highly polar aprotic solvents like DMF, DMAc, etc. The solubility of the resins in polar solvent can be attributed to the presence of large numbers of polar functionality in the structures.

Curing studies of the FRE, MGE, and MGBP AE

In the drying process, the liquid resins are converted to solid form by chemical crosslinking reaction with hardener. The epoxy group (oxirane ring) of the resins is highly strain and is readily open up in the presence of active or labile atom like proton of amine or phenol/thiol. The hardener, poly(amido amine) contains labile amino protons, which can readily react with epoxy groups under heat to form crosslink three dimensional network structures. As in all the above resin systems, the amount of hardener and temperature are the same, so the rate of cross linking or drying is dependent on the reactivity and amount of epoxy groups present in the resin. FRE required the lowest curing time, while MGE took the highest time for curing (Table I). For FRE, more number of epoxy groups (lowest epoxy equivalent, Table I) took part in the curing reaction; also the reactivity of epoxy groups is the highest due to the presence of electronegative bromine atom in the structure, and hence cross linking reaction accelerated in this case. On the other hand, MGE shows the slowest cure rate, which can be attributed to the lowest amount of functionality (highest epoxy equivalent, Table I) and also the epoxy groups are less re-

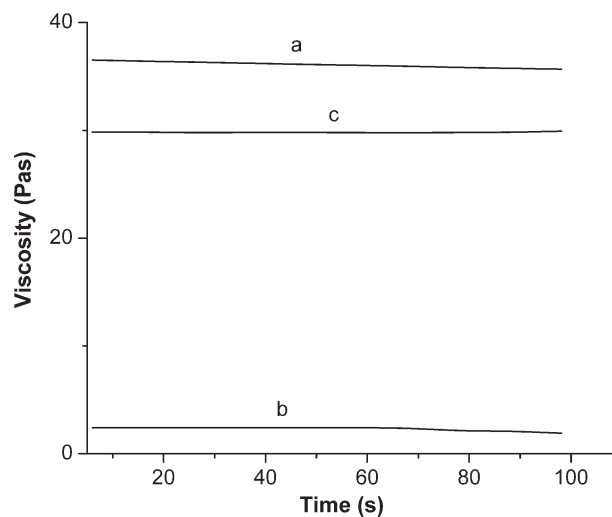


Figure 3 Variation of viscosity against time at constant stress under isothermal condition for (a) FRE, (b) MGE, and (c) MGBP AE.

active as they are not freely assessable because of the long hydrocarbon chains that are entangled and hence epoxy groups at interior are not taking part in crosslinking reaction.²⁴

Rheological studies

The rheological characteristics such as variation of viscosity against shear stress, temperature at constant stress, and time of the resins were studied. Under a constant shear stress of 100 Pa, the viscosity remains almost constant; this indicates that all the resins exhibit Newtonian like behavior (Fig. 3). The average viscosity values are given in Table II. FRE shows the highest viscosity, whereas MGE shows the lowest value. This difference may result from the increase of the number of epoxy group, which may form hydrogen bonding. The viscosity decreases when a temperature gradient is applied (Fig. 4). This decrease may be due to the increase in the molecular mobility of the chains as kinetic energy of the system increases.

Performance studies of FRE, MGE, and MGBP AE cured films

The performance characteristics such as impact resistance, scratch resistance, gloss, tensile strength,

TABLE II
Viscosities of FRE, MGE, and MGBP AE
as Measured by CVO 100

Samples	Stress applied (Pa)	Viscosity (Pas)
FRE	100	36.08
MGBP AE	100	27.33
MGE	100	2.12

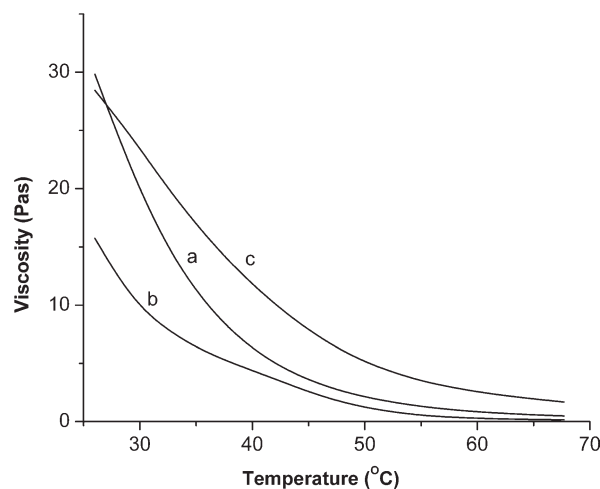


Figure 4 Variation of viscosity against temperature under constant stress for (a) FRE, (b) MGE, and (c) MGBPAAE.

and adhesive strength values of the cured resins are tabulated in Table III. From this table, it has been found that the impact strength increases with the increase of the epoxy content of the resins. The variation of impact strength with epoxy content of the resin may be explained from the angle of toughness of the films, which is the ability to absorb the applied external energy. Thus the impact strength increases with the increase of mechanical strength and flexibility of the films (Table III). However for MGE the impact strength is the lowest, which may be due to its low strength. The presence of long hydrocarbon chains in the side group of MGE causes enhancement on flexibility, which in turn makes the cured resin too soft to peel out. Thus as the film of cured resin of MGE is not possible to obtain, so mechanical tests were not possible to perform. Scratch resistance of FRE shows the highest value. This property can be related to the surface morphology and the toughness of the film. Highly crosslinked film with good toughness shows high scratch resistance (Table III). The high crosslink density is supported by the swelling values (Table III). Thus the film with good toughness shows resistance to any plastic deformation. Most of the reflected light from a coated surface is diffused. Gloss refers to specular reflection of the light reflected at the same angle of incidence. The gloss generally depends on the quantity of light absorbed or transmitted by the coating

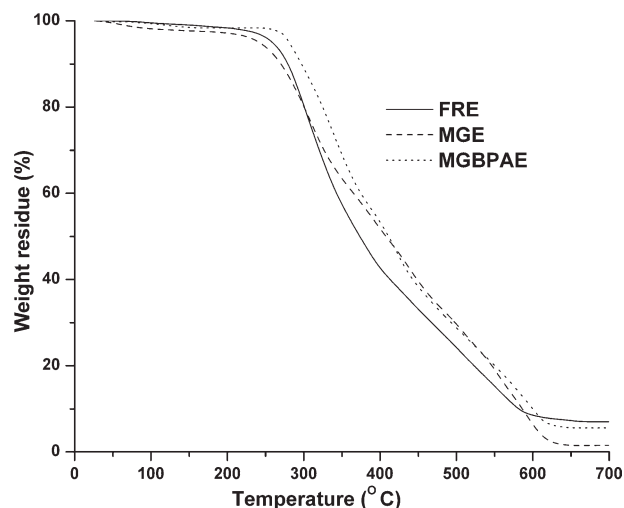


Figure 5 TGA thermogram for FRE, MGE, and MGBPAAE.

material, which is determined by the smoothness of the texture of the surface. All of the resins show moderate gloss characteristic. FRE has good smooth film surface and hence shows the highest gloss value, whereas MGE film surface is not so smooth due to low dimensional stability and hence results in low gloss value. FRE also exhibits good adhesive strength. The adhesive strength of the resins on wood substrate depends on a wide range of variables such as surface smoothness of the substrate, presence of wood extractives, pH, etc. The good adhesive strength in this case is due to the formation of H-bonding, polar-polar, and polar-induced polar interactions between hydroxyl, epoxy, ester, ether, etc., groups of the resins and the hydroxyl groups of the cellulose plywood substrate.

Thermal studies

The relative thermal stability of all the cured resins was compared from TG analyzes (Fig. 5). From these curves, it can be seen that all the resins undergo one-step degradation process. The onset temperature of degradation for FRE is 242°C and for MGBPAAE 265°C, whereas for MGE it is 225°C. For FRE, due to the presence of the bromine containing moiety in the structure, degradation occurs at a lower temperature than MGBPAAE. Since bromine is labile, the major initial mass loss may be due to the loss of HBr from

TABLE III
Mechanical Properties of FRE, MGE, and MGBPAAE

Samples	Impact resistance (cm)	Scratch hardness (Kg)	Gloss (°)	Tensile strength (N/mm ²)	Adhesive strength (N/m)	Elongation (%)	Percentage of swelling (%)
FRE	>100	3.5	67.02	5.9	750.56	83.60	10.18
MGBPAAE	75	1	60.57	2.3	230.34	25.7	22.03
MGE	15	0.5	55.90	–	–	–	40.2

TABLE IV
Chemical Resistance of FRE, MGE, and MGBPAAE Cured Films

Types of media	FRE	MGBPAAE	MGE
Alkali (0.5%)	Fair	Fair	Very poor
HCl (10%)	Excellent	Excellent	Poor
NaCl (10%)	Excellent	Good	Good
Distilled water	Excellent	Excellent	Good
Ethanol (20%)	Excellent	Excellent	Fair

the structure. However, the enhance stability of FRE over MGE may be due to the greater crosslinking density of the former than the latter because of the lower epoxy equivalent that is higher number of epoxy groups present in the FRE resin. Also in MGE, the constituents are mainly aliphatic hydrocarbon, whereas in FRE sufficient amount of aromatic moieties are present. The highest thermal stability of MGBPAAE compared to other two resins can be attributed to the presence of the aromatic group of BPA and absence of thermolabile bromine atoms in the structure.

Flame retardancy

The flame retardancy rating of FRE was found out to be V1 as tested by UL 94. However for the MGE and MGBPAAE, it was found that they were combustible, i.e., they burnt out completely on application of the flame. This may be due to the presence of highly combustible hydrocarbon moiety, where C/H ratio is low. For FRE good flame retardant property can be attributed to the presence of TBPA moiety in the resin. TBPA moiety acts by hindering the spread of the fire, and gives precious extra time in the initial stages of a fire when it is much easier to escape. They could be involved to remove high-energy $\cdot\text{OH}$, $\cdot\text{H}$, $\cdot\text{O}$ and the like radicals generated during the burning process by the suppressions via bromine.²⁸ Also it can produce the noncombustible HBr (25% Br content in the resin as measured by Schoniger method), which helps the inhibition of flame during the burning process though vapor phase mechanism.

Chemical resistance test

The chemical resistance of FRE to aqueous HCl, NaCl, and ethanolic solution is very good; however, to aqueous alkali the film remains intact though there is an adhesion failure (as the film comes out from the surface of the glass plate). However, MGE shows very poor resistance to alkali solution, which may be due to presence of large number of hydrolyzable ester group of the fatty acids. Thus the overall chemical resistance of the cured resins is good though the alkali resistance decreases with the increase of monoglyceride content due to the presence of hydrolysable ester group in it (Table IV).

CONCLUSIONS

M. ferrea L. seed oil based three different epoxy resins with varying compositions of diols were synthesized successfully. The resins were well characterized by using different analytical and spectroscopic techniques. FTIR and NMR studies support the formation of the epoxy resin. The resin (FRE) obtained from the combination of monoglyceride, BPA, TBPA, and epichlorohydrin exhibits the highest mechanical strength and good thermal stability. The flame retardant FRE resin is the best resin among the studied resins.

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