ISSN: 2277-9655
Impact Factor: 4.116
CODEN: IJESS7

IJESRT

INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY

PREPARATION, CHARACTERIZATION AND THERMAL BEHAVIOR OF ALKYL SUBSTITUTED PHENOLIC EPOXY RESIN

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DOI: 10.5281/zenodo.246801

ABSTRACT

The present article deals with the synthesis of phenolic epoxy resin by the reaction of phenolic resin and epichlorohydrin. The synthesis of phenolic resin was carried out by using p-ethylphenol, formaldehyde and naphthol. The structures of phenolic and epoxy resins were confirmed by spectroscopic analysis. The synthesized epoxy resin showed solubility in polar solvents like DMF, dioxane, acetone, DMSO, THF, ethyl acetate, and chloroform. Thermal characterization of epoxy resin was monitored by differential scanning calorimeter (DSC) using curing agent and thermo gravimetric analysis (TGA). Molecular weight of the resin was evaluated by gel permeation chromatography (GPC).

KEYWORDS: Phenolic resin, Epoxy resin, TGA, GPC, FT-IR Method.

INTRODUCTION

Epoxy resins have versatile industrial applications and used as an adhesives in the construction of aircrafts, boat, automobiles, and as structural matrix material in aerospace industry. Epoxy resins have found a large variety of applications in painting, surface coating, semiconductor devices because they show excellent resistivity to heat, weather and electrical insulation.[1-4] At present epoxy resins are widely used in various industrial applications such as solvent storage tanks, sewer pipes, concrete flooring and absorption towers.

Epoxy resins are applicable to join various materials like glass, plastics, wood and metals as glue, due to presence of strong bonding properties. Epoxy resins show good adhesion to many substrates and high resistivity to moisture chemical, heat and electrical so these resins are used as fiber reinforced materials.[5-6] Epoxy resins have favorable physical properties, toughness, low cost, mechanical stiffness, superior chemical and corrosion resistance. These resins can be employed in the construction of aircrafts, automobiles, boat as an adhesive. [7-9]

The applications of phenolic epoxy resin in high performance materials have been increasing since last few decades. Extensive work on the phenolic epoxy and their synthesis in the related industry have been reported but there are many scopes and demands in the modern civilization for the present conditions and to release the stress full environment and provide a better quality of composites and epoxy resins. In this sequence phenolic epoxy resins containing naphthalene ring have excellent thermal and mechanical properties because they have combine properties of epoxies and phenolic resins. Phenolic epoxy resins provide structural adhesives with excellent high temperature resistance as compared to other epoxy resins.[10] The present article reports on the synthesis, characterization and thermal studies of phenolic epoxy resin.

MATERIALS AND METHODS

(A)Materials

p-Ethylphenol, formaldehyde, ethanol, 1-naphthol, NaOH, methanol, epichlorohydrin were used of analytical grade without further purification. Glass cloth Satin (2/2) weave woven fabric, 0.25 thick, E-glass, 270 g/m^2 (obtained from Unnati Chemicals, Ahmadabad, India), was used for composite fabrication.

(B)Synthesis of Phenolic Resin

p-Ethylphenol and formaldehyde taken in 1:2 ratio with NaOH as a catalyst. All reactants were charged in three necked flask equipped with a mechanical stirrer, water condenser and thermometer. The reaction solution was stirred and allowed to reflux at about 65^{0}c for one hour. Then to this solution naphthol and ethanol as a solvent are added.

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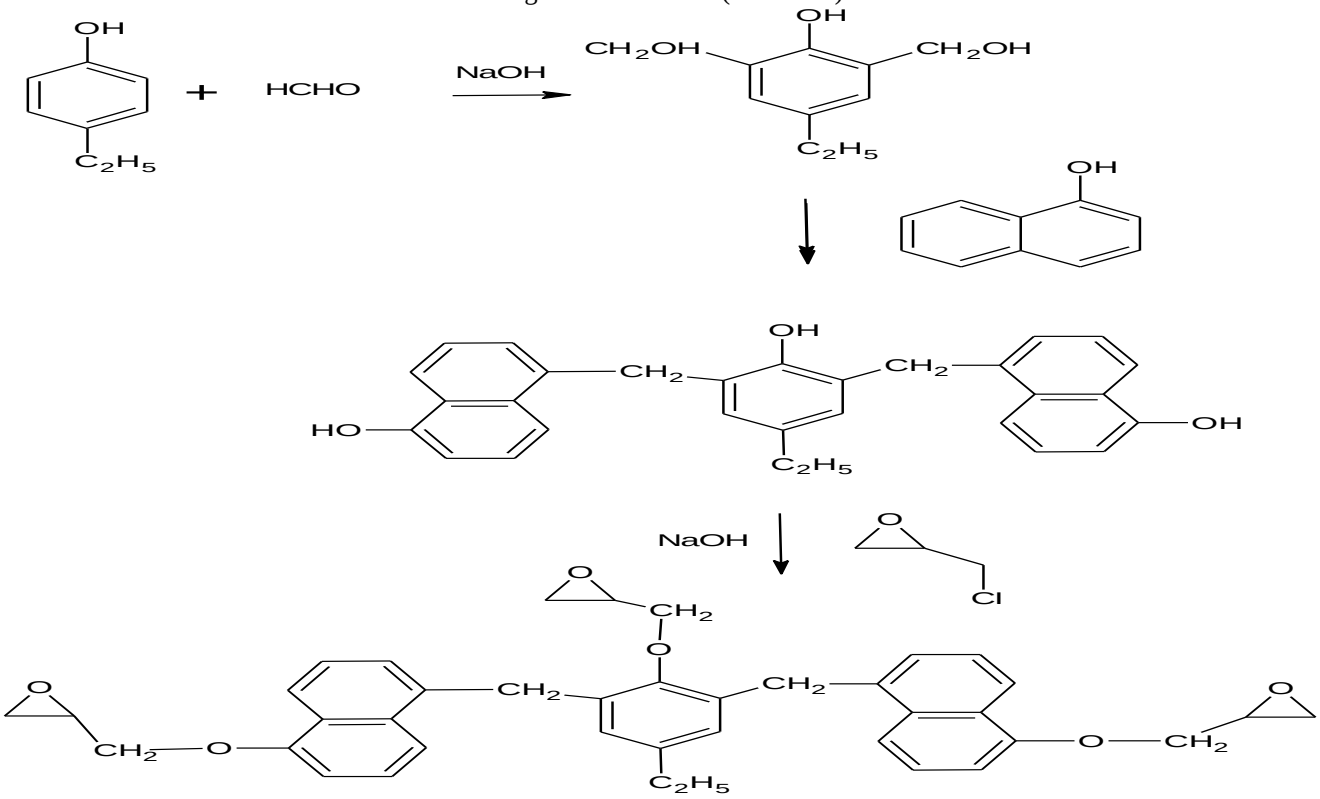
The solution was continuous stirred for 16 hrs at 70^0c temp. The resulting solution was obtained in the form of viscous liquid as 2, 6-dimethylol-4-ethylphenol.

(C) Synthesis of Epoxy resin

The synthesized phenolic resin, epichlorohydrin and methanol were taken in three necked flask equipped with a reflux condenser and mechanical stirrer. The reaction mixture was stirred and reflux at 90^0C for 7 hours. 10 % NaOH solution is added to the reaction flask with stirring as shown below (Scheme 1).



Scheme 1. Reaction pathway for the synthesis of phenolic epoxy resin

RESULT AND DISCUSSION

Infrared spectra and ^{1}H-NMR were recorded in the form of KBr pellets on a FT-IR Perkin-Elmer spectrometer and BRUKER AVENCE II 400 MHz NMR spectrophotometer respectively. The chemical structures of resultant phenolic and epoxy resins were confirmed on the basis of fourier transform infra-red (FT-IR). A peak at 3334 cm^{-1} was observed due to the O-H group stretching of hydroxyl group in phenolic resin. The presence of oxirane ring in epoxy resin was confirmed by the appearance of a strong band at 913 cm^{-1}. A band at 1273 cm^{-1}due to symmetrical C-O stretching in epoxides. Other specific bands at different position gave more evidence to confirm the structure of synthesized epoxy resin. FT-IR spectra analysis data of epoxy resin is examined and given in **Table 1**.

Further characterization was done with ^{1}H-NMR analysis to identify the structure of resin in DMSO. Important chemical shift values of the epoxy resin were summarized in **Table 2**.

Table1. FT-IR Absorption Bands of Epoxy resin

Characteristics	**Signals (cm^{-})**
Oxirane ring str	913
C-H str of aromatic CH=CH	3191
C=C str of aromatic CH=CH	1460, 1506, 1589,1627
C-H str of CH_2	2937
Ortho substituted	763
Para substituted	829
C-O str of epoxide	1273
C-H def of CH_3	1388

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Table 2. ^{1}H-NMR data of Epoxy resin

Characteristics	Chemical shift (ppm)
protons of phenyl ring	7.2-8.1
–CH_2 protons of oxirane ring attached to phenyl ring	2.7
–CH_2 protons of oxirane ring attached to naphthalene ring	2.5
O–CH_2 protons attached to phenyl ring	3.8
O–CH_2 protons attached to naphthalene ring	3.6-3.7
Protons of -CH_2	4.1
–CH_2 protons of ethyl group	3.2
–CH_3 protons of ethyl group	1.3

Thermal Studies:

Unreinforced cured samples were subjected to thermogravimetric analysis (TGA) on Du Pont 950 thermogravimetric analyzer at a heating rate of 10 K min^{-1} and analyzed data are represented in **Table 3**. Experimental results showed that unreinforced epoxy resins have degradation at about 150 °C. Initial weight loss of sample is about 0.69% and the decomposition rate was increased rapidly between 300 to 500 °C as shown in **Figure 1**. Universal VG03 differential scanning calorimeter (DSC) was used to investigate the curing study of epoxy resins at heating rate of 10 °C/min. The DSC data of epoxy resins are shown in **Table 4** revealed that cured samples give a single exothermic peak shown as **Figure 2**.

Table 3. TGA data of Epoxy Resin

Comp.	% Wt. loss at various temperatures					
	150°C	200°C	300°C	400°C	500°C	600°C
Epoxy Resin	0.71	2.65	21.98	47.91	68.89	84.95

Table 4. DSC Curing data of Epoxy Resin

Comp.	Curing Temperature °C		
	Initial Temp. (T_i)	Peak Temp. (T_p)	Final Temp. (T_f)
Epoxy Resin	115	145	171

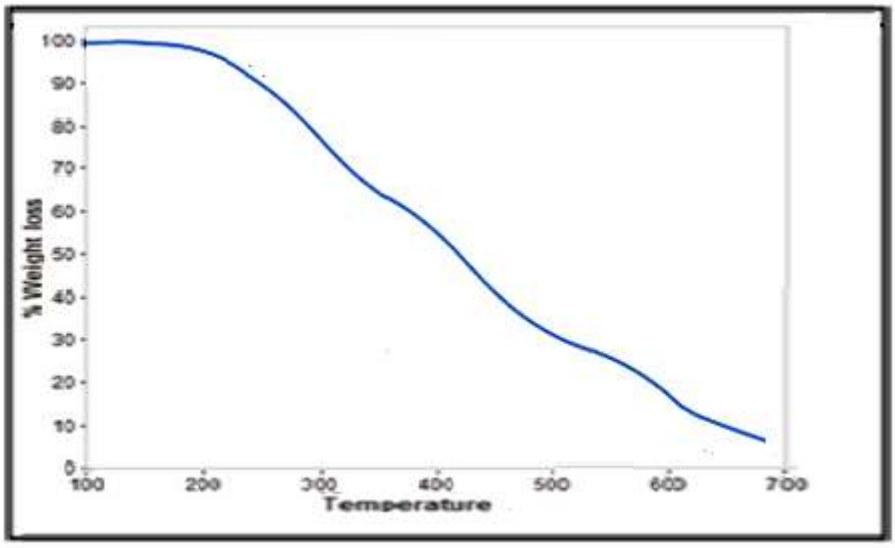

Figure-1: TGA spectrum of Epoxy Resins

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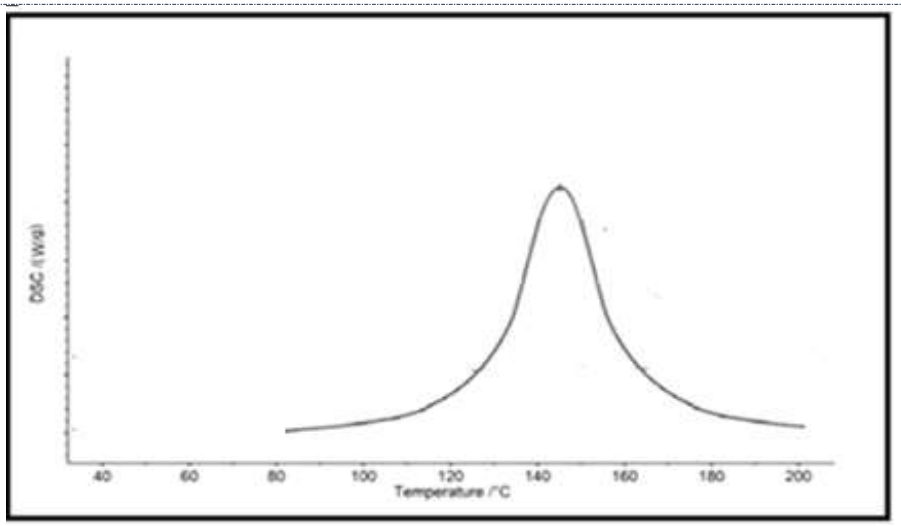

Figure-2: DSC spectrum of Epoxy Resins

The molecular weight of the synthesized epoxy resin was measured by gel permeation chromatography (GPC) analysis. GPC is a reliable and fast technique to determine the polydispersity index (PDI) and molar mass averages of polymers. The number average, weight average molecular weights (Mn, Mw) and polydispersity index of epoxy resin were summarizes in **Table 5**.

Table 5. Molecular Weight and PDI of resin

Resin	**Mn**	**Mw**	**PDI(Mw/Mn)**
Epoxy resin	2348	3833	1.632

CONCLUSION

This work focused on the synthesis of epoxy resin with thermal studies. The epoxy resin was synthesized by condensation reaction of phenolic resin with epichlorohydrin. The structure of resin is characterized by their FT-IR and ^{1}H-NMR spectrum. Curing of resin was investigated on a DSC by using diaminodiphenyl methane (DDM) as curing agent. The synthesized epoxy resin having naphthalene ring demonstrated good thermal stability property. As a result, the resin is used as adhesive and coating materials in industry.

ACKNOWLEDGEMENT

The authors wish to thank Head, Department of Polymer Science, M. L. S. University Udaipur, SICART, Vallabh Vidhyanagar, Gujarat, and SAIF Chandigarh for their help in present work.

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