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Epoxy Based Vinyl Ester Resins: Synthesis and Characterization

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Abstract

Epoxy based vinyl ester resins were synthesized by reacting with methyl acrylate (MA) / ethyl acrylate (EA). The estrification reaction of epoxy resin was carried out using triethylamine (1 percent of total weight of epoxy resin) as a catalyst and 0.03% of hydroquinone as an inhibitor at 90^{o}C-100^{o}C. The synthesized resins are structurally characterized by FT-IR and ^{1}H- NMR spectroscopic methods and their resultants show good solubility in polar solvent.

Keyword: Epoxy resin, Methyl acrylate, Ethyl acrylate, NMR, FT-IR Method

1. INTRODUCTION

Vinyl ester resins are widely used for building and construction, automobile structural parts, chemically resistant surface coatings like solvent storage tank and flue stack linings etc., printed circuit board coatings and ultraviolet cured inks because of their low cost, superior chemical and corrosion resistance, excellent heat performance and good mechanical properties[1-3]. Vinyl ester resins are also employed as the matrix in the formation of fiberglass reinforced plastic (FRP) which are often used for such diverse product as boat hulls, automotive parts, building panels, housing, bathroom components, pipes, and pressure vessels, appliances, and electronic and electrical applications by using various lay-up, spray-up, filament winding, compression, and resin transfer moulding techniques[4-6]. The development of simple and efficient method for the synthesis of resin from readily available reagents is the major challenge. In the present work, we report the estrification of epoxy resin based on naphthalene group with different type of unsaturated acrylates in the presence of alkali catalyst.

2. MATERIALS AND METHODS

(A)Materials

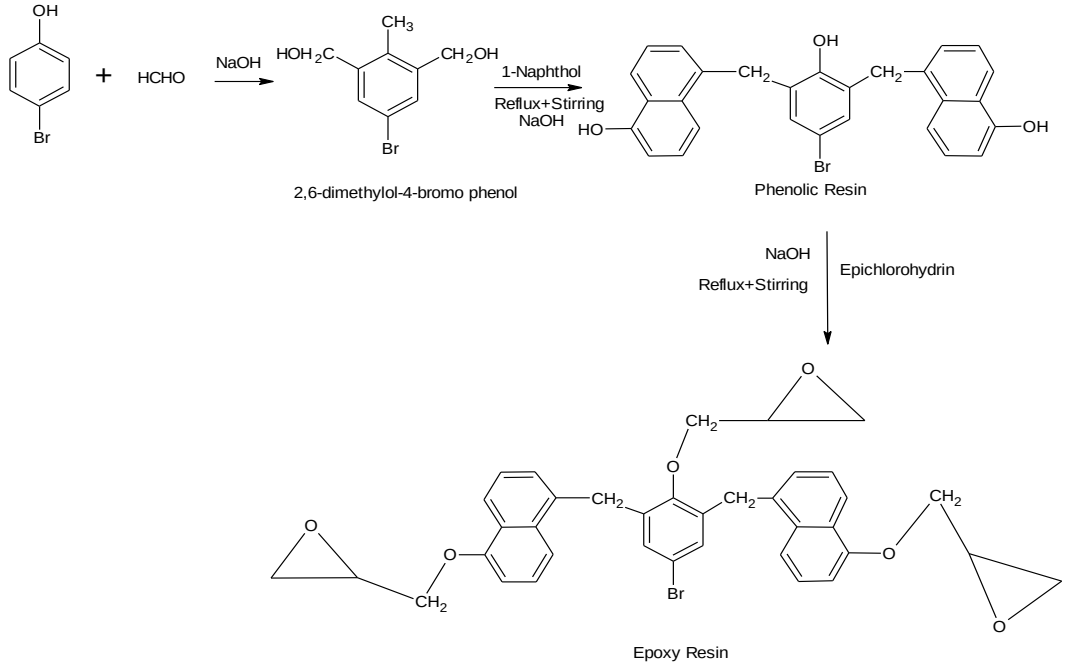
4-bromophenol, formaldehyde, 1-naphthol, epichlorohydrin, NaOH pellets, methyl acrylate and ethyl acrylate etc.

(B) Synthesis of epoxy resin

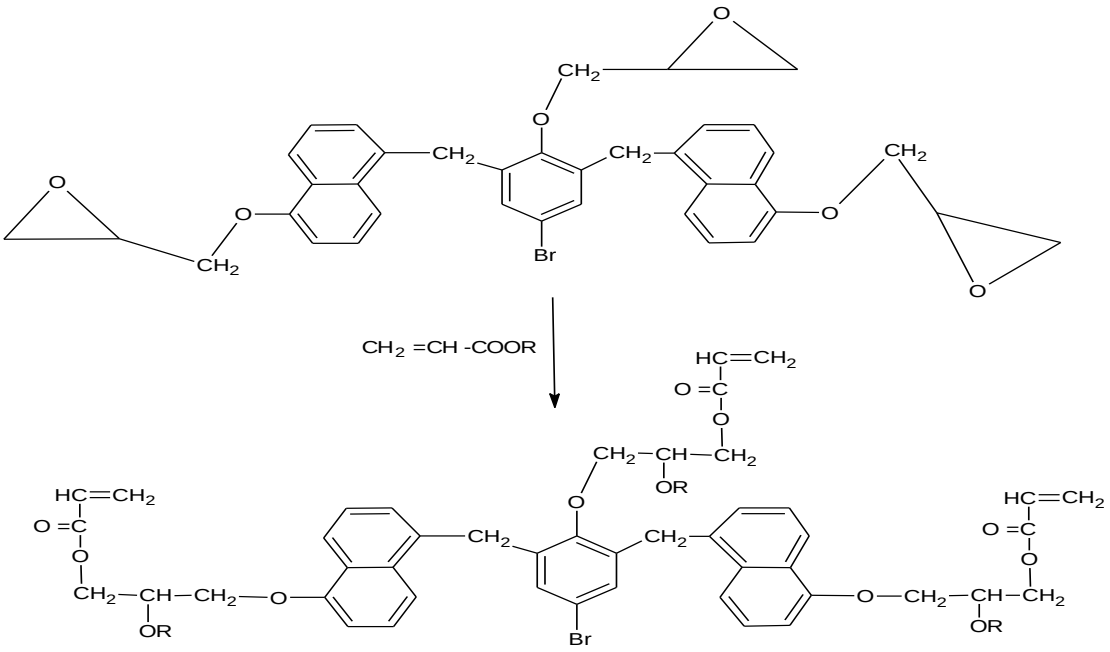
The epoxy resin was synthesized by using method reported method [4]. In three necked flask 4-bromophenol and formaldehyde in 1:2 ratio were charged in alkali medium. The solution was heated with constant stirring to form dimethylol resin at 70°C-80°C. Then add to reaction mixture 1-naphthol to prepare phenolic resin. Epoxidation of phenolic resin was carried out with epichlorohydrin in presence of NaOH for 8-10 hours. The reaction scheme is shown in Scheme 1.

(C) Synthesis of vinyl ester resin

The epoxy resin and methyl acrylate/ethyl acrylate were charged in two necked flask equipped. The mixture was heated at 90°C -100°C in presence of triethylamine used as a base catalyst and hydroquinone as an inhibitor. The estrification reaction was done for 6 hours. The synthesized resin was dissolved in toluene and filtered using whatman filter paper to remove salt. Toluene was distilled off under reduce pressure and the product was dried in the oven at 60°C. Vinyl ester was formed in viscous form. The reaction scheme is shown in Scheme 2.



Scheme 1: Reaction pathway for the synthesis of epoxy resin



Scheme 2: Reaction pathway for the synthesis of VER resin

3. RESULT AND DISCUSSION

FT-IR and ^{1}H-NMR spectroscopy were used to structural characterization of the resultant resins. FT-IR spectra were recorded on a Shimadhzu – 8201 FT-IR Spectrometer with KBr pallet technique. ^{1}H-NMR spectra were measured on BrukerAvances II 400 NMR Spectrometer by using TMS as an internal reference. Elemental analysis was evaluated on Carlo-Ebra NA – 500 auto-micro analyzer. The C, H, N contents of both the epoxy resin and the vinyl ester resins are summarized in Table 1.

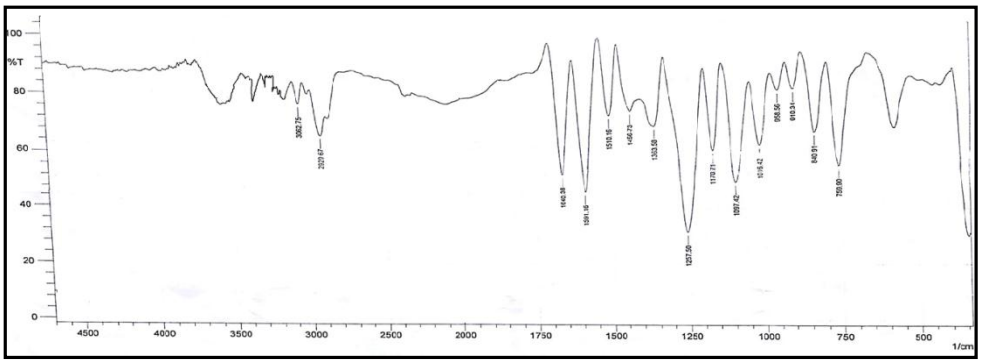
Table 1: Preliminary Characterization of Resins

Resin code	Molecular Formula	Elemental Analysis	
		%C (Calc. / Found)	%H(Calc. / Found)
EPOXY	$C_{37}H_{33}O_6Br$	56.66/57.12	5.05/5.98
VER I	$C_{49}H_{51}O_{12}Br$	64.54/66.01	5.59 /6.67
VER II	$C_{52}H_{57}O_{12}Br$	65.47/64.52	5.98/5.15

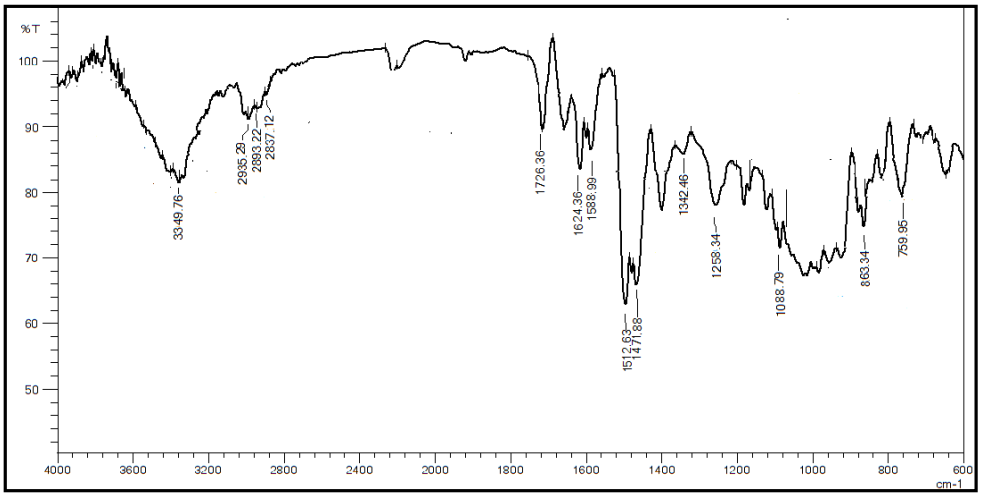
The structure of the prepared epoxy resin and vinyl ester resin is confirmed by FT-IR. Furthermore, the structure of VER is also confirmed by NMR techniques.

The FT-IR spectrum of epoxy resin is shown in fig.1 (a). The 910 cm^{-1} absorption band in epoxy resin is due to oxirane ring and the absorption band near 1456 cm^{-1}, 1510 cm^{-1}, 1591 cm^{-1} and 1640 cm^{-1} etc. may be due to aromatic C=C bond. Figure 1 (b) and (c) show the FT-IR spectrum of vinyl ester resin. The absence of absorption band due to oxirane ring in vinyl ester resins spectra indicate that epoxy groups are completely used during the reaction. The bands at 1627 cm^{-1} and 1624 cm^{-1} indicate the presence of olefinic band which confirms the formation of vinyl. The band at 1721 cm^{-1} and 1726 cm^{-1} are due to the –C=O stretching vibration of ester group. The absorption band at 1258 cm^{-1} and 1273 cm^{-1} is due to the C-O-C stretching for ethers.

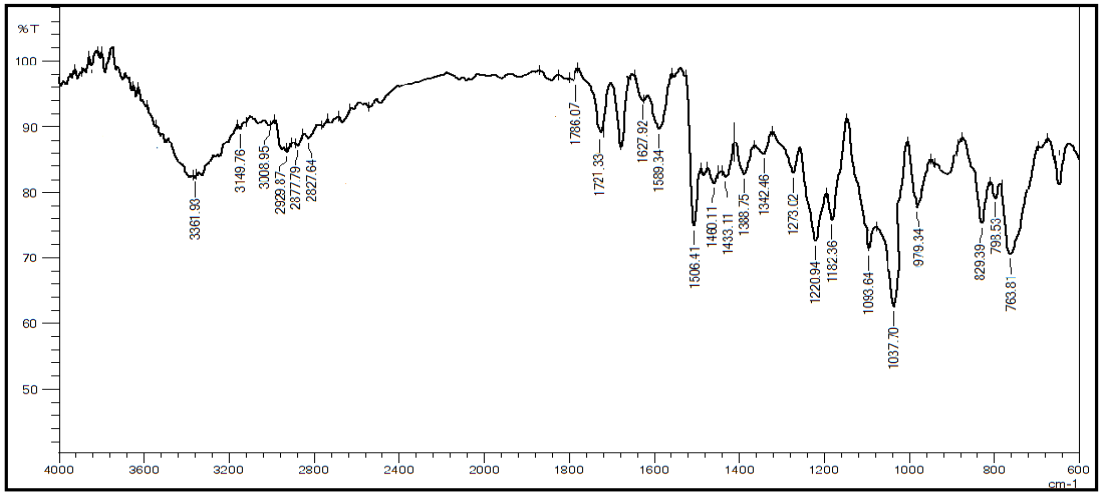
^{1}H-NMR spectra of vinyl ester resins (figure-2) showed the chemical shift of vinylic protons of methyl and ethyl acrylate show in the range of 5.3 to 6.2 ppm. Aromatic protons show the signal in the region of 6.8-7.9 ppm. Signals at 3.2- 4.4 ppm were observed due to methylene protons.



(a)

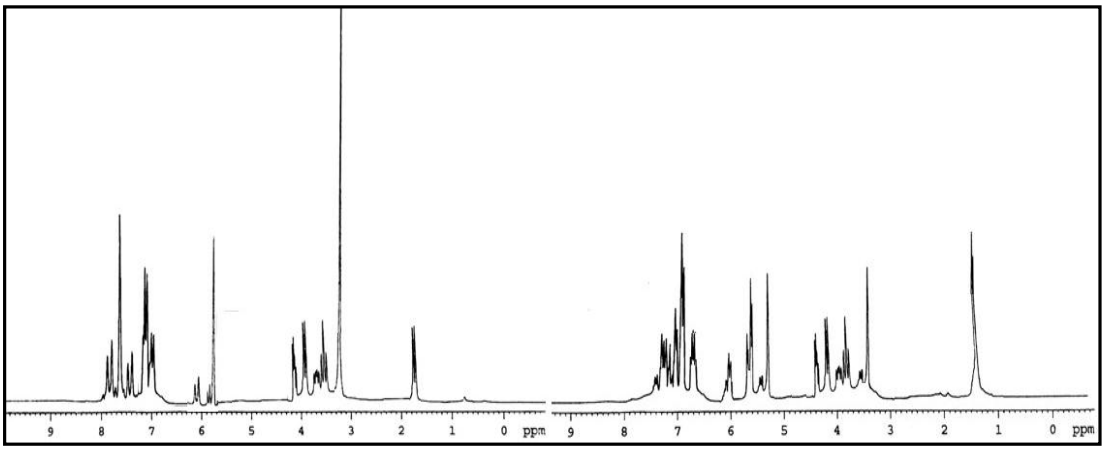


(b)



(c)

Figure 1. FT-IR Spectra of (a) Epoxy resin; (b) Vinyl ester resin of bromo phenol with methyl acrylate (VER I); and (c) Vinyl ester resin of bromo phenol with ethyl acrylate (VER II)



(a) **(b)**

Figure 2. ^{1}H-NMR Spectra of (a) VER I; and (b) VER II

4. CONCLUSION

This work focused on the synthesis of vinyl ester resins by the reaction of epoxy resin based on 2, 6- dimethylol-4-bromonaphthol with methyl acrylate and ethyl acrylate in presence of triethylamine and hydroquinone. The CHN data of resultant samples are consistent with the predicted structure of the vinyl ester resins. The structures of synthesized epoxy as well as vinyl ester resins are supported by their IR analyses results and further confirmed by NMR spectral studies.

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