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Synthesis ,Characterization and Curing of Vinyl ester resin

Jyoti Chaudhary

Department of Polymer Science,University College of Science, MLS University, Udaipur, Rajasthan

Abstract

The epoxy resin employed in this study is synthesized by using excess of epichlorohydrin and aniline. Further the prepared diglycidyl aniline epoxy resin (DGA) is treated by crotonic acid to form vinyl ester resin (VER).The resultant resin was characterized by FT-IR spectral studies. The curing study of this resin was monitored by Differential Scanning Calorimeter (DSC). The unreinforced cured resin was subjected to Thermogravimetric analysis (TGA).

Keywords*: Differential Scanning Calorimeter, Thermogravimetric analysis, FT-IR, Vinyl ester resin*

1.INTRODUCTION

Vinyl ester resins form a family of thermosetting resins which combine the excellent thermal and mechanical properties of epoxy resins with the ease of processing and rapid curing of polyester resins[1]. These resins are the additional product of Epoxy resin and unsaturated carboxylic acid and produced by the esterification of an epoxy resin with an unsaturated monocarboxylic acid. They have terminal reactive double bonds derived from the carboxylic acid used . They combine the best properties of epoxies and unsaturated polyesters [2]. The Vinyl ester resins were first introduced commercially in the early 1960,s. These are widely used in fabricating industrial equipment and structures such asabsorption towers, process vessels, storage tanks, pipes, hoods, ducts and exhaust stacks [3]. Vinyl ester resin are used in various typical application include structural structural material coating ,adhesives, molding compound ,electrical appliances ,dental material and aerospace application[4-6].These resins are widely used in marine Indus tries ,manufacturing FRP tanks and vessel due to its increased corrosion resistance and ability to withstand water absorption[7].

Jyoti Chaudhary
Email: jyotichaudhary13@gmail.com

Vinyl ester oligomers are also used to modify unsaturated polyester resin because of its good tensile strength and chemical resistance over epoxy resin [8].Vinyl ester resins are also found applicability in special domains, such as aeronautic, naval and auto vehicle, constructions towards biomedical science and in the construction of waste incineration gas cleaning units [9-10]. In thepresent paper, Characterization and Curing of synthesized Vinyl ester resin was discussed.

2. MATERIALS AND METHOD

All the chemicals used in the present work were of analytic grade or purified by literature method.

2.1 Synthesis of Diglycidyl aniline epoxy resin (DGA):

The DGA synthesized according to the method reported earlier [11]. Aniline was addednto a mixture of epichlorohydrin, 95% methanol and water ,were charged in a three necked flask equipped with mechanical stirrer . The temperature of the reaction maintained at 75^{0}C for 6-7 hours. Then to this solution 1N NaOH is added drop by drop , excess epichlorohydrin was removed under vacuum . Then 60 ml

benzene is added and the solution is washed fourtimes ,the organic layer is distilled under vacuum at 70°C and viscous DGA layer is obtained.

2.2 Synthesis of Vinyl ester resin (VER)

Diglycidyl aniline Epoxy Resin (DGA) was synthesized using Aniline and Epichlorohydrin [refe. Siddhr].This Synthesized epoxy resins and Crotonic acid in 1:2 ratio were charged in three necked flask equipped with a mechanical stirrer for 6-7 hours at 100°C . To this 0.1 mole of hydroquinone was added as a catalyst. Few drops of pyridine were used as an inhibitor. The resin was then discharged to give vinyl ester oligomer. These were in the form of pasty mass.

2.3 Characterization

The presence of unsaturation in VER was predicted by Bromate –Bromide titration method..FT-IR has been scanned in KBR pellet on Perkin-Elmer, RXZ IR spectrometer.

2.4 Curing

The curing study of VER was monitored on a Universal V3.0GTA instrument, using benzoyl peroxide as a catalyst and using diamino diphenyl methane(DDM) as a curing agent. The instrument was calibrated by the standard indium metal with the known heat of fusion. Curing was carried out at heating rate of 10oC min -1 .The sample weight was in the range of 4-5 mg.

Thermogravimetric analysis (TGA) of unreinforced cured sample was carried out on

Table 1. TGA of unreinforced cured sample

Sample Code	% Weight loss at various Temperature (°C) from TGA			
	150	300	450	600
VER	2.25	20.21	76.52	87.81

Table 2. DSC Curing Data

Sample Code	Initial Temp. (°C)	Peak Temp. (°C)	Final Temp. (°C)
VER	96	122	146

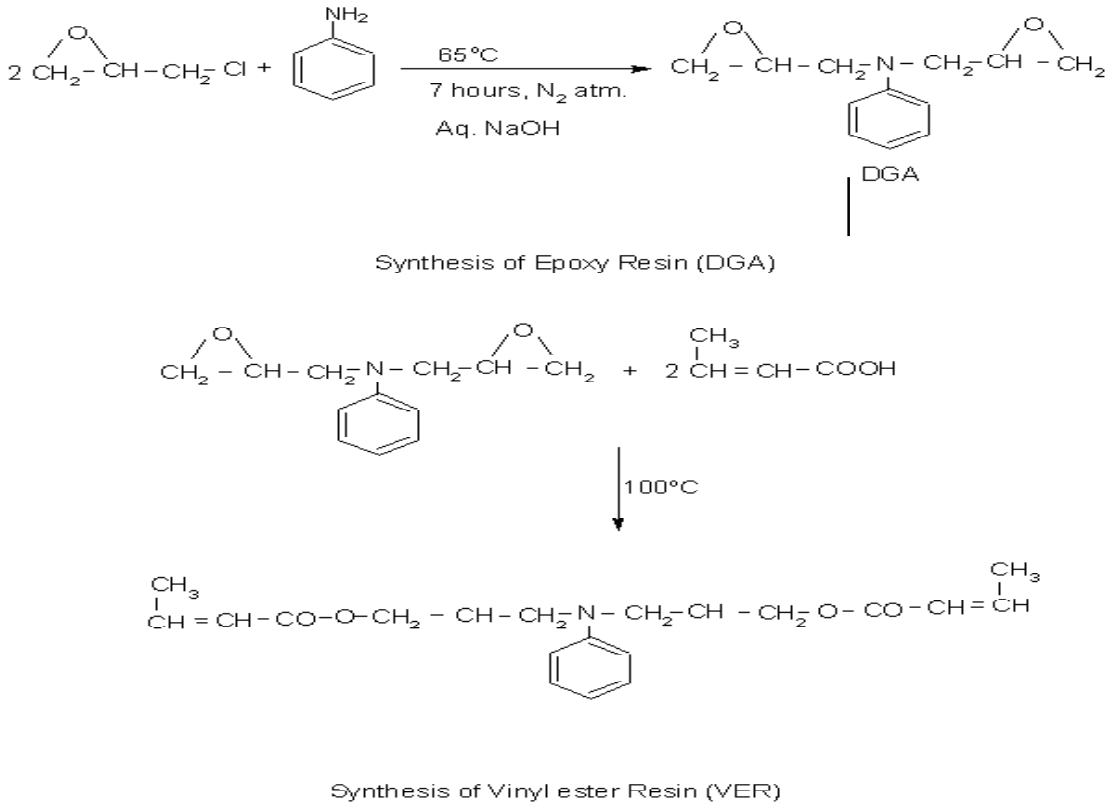
"Universal V3.0G TA Instrument Thermo gravimetric analyzer" .Heating rate was 10o C/ minute.

3. RESULT AND DISCUSSION

The structure of synthesized epoxy resin and vinyl ester resin is confirmed by their FT-IR spectra. The FT-IR spectra of Epoxy resin (DGA) show a characteristics absorption frequencies at 3060 cm-1 (Aromatic C-H)1590 cm-1,1571 cm-1,1495 cm-1 (C=C),1270 cm-1 (Ar-N),905 cm-1 (epoxy group),1103 cm-1 (ether group) .

Synthesized Vinyl ester resin show band at 1720 cm-1 is due to carbonyl group of ester. The absorption band due to stretching and bending vibration of vinyl group were present at 1630 cm-1 and 1036 cm-1 which confirm the vinyl group in the resin. Lacks of ether absorption band at 1103 cm-1 also confirm the completion of epoxy esterification reaction . The peak at 905 cm-1 representing the epoxy group was no longer visible (Fig-1). Curing of the synthesized vinyl ester resin was done on differential scanning calorimeter (DSC) by using benzoyl peroxide as a catalyst and DDM as a curing agent .The data of DSC thermogram of the resin are shown in Table-1.

Thermogravemetric analysis (TGA) was used to analyze the unreinforced cured vinyl ester resin sample.TGA data of the cured sample are furnished in Table-2. The results reveal that the cured sample starts its degradation at about 150OC and initial weight loss of is about 2.25%.At 300OC the weight loss of about 20.21% is found. The maximum weight loss was found between 300OC



Scheme: 1 Synthesis of epoxy resin and vinyl ester resin

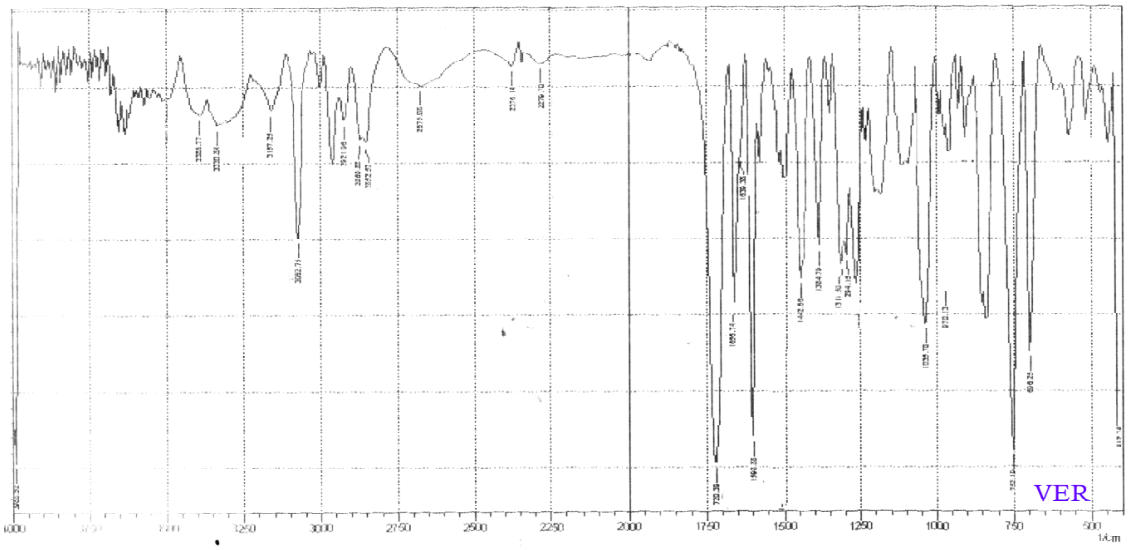


Fig. 1 : FT-IR Spectra of Vinyl ester resin (VER)

to 450OC temperature. About 87.81% weight loss was observed at 600 OC.

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