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Ageing Studies on Diglycidyl Ether of Bis-phenol A Resin Modified by Epoxy Cresol Novolacs

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ABSTRACT

In this study, novolacs were prepared from para cresol and ortho cresol. The Cresol-formaldehyde ratio in the novolacs was maintained at 1:0.8 for maximum property enhancement. Novolac resins are epoxidised through the phenolic hydroxyl groups by treatment with epichlorohydrin. Due to the presence of epoxide groups in epoxidised novolacs it is compactable with the DGEBA (diglycidyl ether of bis-phenol A) resin. Epoxidised novolac synthesised from p-cresol (p-ECN) and Epoxidised novolac synthesised from o-cresol(o-ECN) were used to blend with DGEBA. These Blends were subjected to ageing studies and the results compared with those of the neat resin. The post-cured samples of the neat DGEBA, DGEBA/p-ECN blend (15 wt %) and DGEBA/o-ECN blend (15 wt%) were aged in a temperature controlled air oven kept at 100 ^{0}C for 24, 48, 72, 96 and 120 hours successively. The aged samples were tested for tensile properties, impact strength and water absorption

KEYWORDS: DGEBA; Resin; Novolac , Ageing

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INTRODUCTION

Epoxy resins are characterised by the presence of more than one 1, 2 epoxide groups per molecule. The first and still the most important class of commercial epoxy resins is the reaction product of bis-phenol A (BPA) and epichlorohydrin in the presence of sodium hydroxide. It is called the diglycidyl ether of bis-phenol A (DGEBA) [1, 2] . Epoxy resin has excellent mechanical, electrical and adhesion properties and is widely used as a high performance thermosetting material in many industrial and engineering fields[3,4,5]. Epoxy resins based on bisphenol A and epichlorohydrin (DGEBA) exhibit brittleness and low elongation after cure. This leads to low resistance to crack initiation and propagation. The usefulness of epoxy resins in many engineering applications is often limited by these disadvantages[6,7]. Epoxies are modified by different methods. Novolac resins are epoxidised through the phenolic hydroxyl groups by treatment with epichlorohydrin.. . In this study first novolac was prepared from ortho cresols and p-cresol. The cresol-formaldehyde ratio in the novolacs was maintained at 1:0.8 for maximum property enhancement. Cresol Novolac resins were epoxidised through the phenolic hydroxyl groups by treatment with epichlorohydrin. These epoxidised novolac was used for blending with DGEBA resin. Then the thermal mechanical and water absorption characteristics are studied. Epoxy resins are generally exposed to harsh environment and this leads to aging of epoxy resin [8,9]. Epoxidised novolac synthesised from p-cresol(p-ECN) and epoxidised novolac synthesised from o-cresol(o-ECN) were used to blend with DGEBA. These Blends were subjected to ageing studies and the results compared with those of the neat resin.

EXPERIMENTAL SECTION

Materials

Commercial grade epoxy resin GY 250, and the room temperature amine hardener HY 951 (polyamine) were supplied by Petro Araldite Pvt. Ltd. in Chennai. . Phenol, o-cresol, p-cresol, formaldehyde, epichlorohydrin, benzene, NaOH, oxalic acid , sodium sulphate were supplied by Merck India Ltd.

Synthesis of Epoxidised Cresol Novolacs

The novolacs were prepared by reacting Cresol with formaldehyde in the molar ratio 1:0.8 in presence of oxalic acid catalyst in a 3-necked flask fitted with a mechanical stirrer, water condenser and thermometer. The reaction mixture was heated and allowed to reflux at about 100°C for 2-3 hours. When the resin separated from the aqueous phase the reaction was stopped. The resin was neutralised with sodium hydroxide, filtered, washed with water and vacuum dried. 1 mole of the novolac resin (1:0.8) was dissolved in 6 moles of epichlorohydrin and the mixture heated in a boiling water bath. The reaction mixture was stirred continuously for 16 hours while 3 moles of sodium hydroxide in the form of 30 %

aqueous solution was added drop wise. The rate of addition was maintained such that the reaction mixture remains at a pH insufficient to colour phenolphthalein. The resulting organic layer was separated, dried with sodium sulphate and then fractionally distilled under vacuum.

Modification of DGEBA with Epoxidised Cresol Novolacs

DGEBA is mixed with 15 weight % Epoxidised cresol novolac resins prepared from o-cresol and p-cresol and was cured at room temperature by adding 10 weight % of the amine hardener and stirring the mixture. The resin was then poured into appropriate moulds coated with a releasing agentCuring was done at room temperature for 24 h, followed by post curing at 120°C for 4 hours.

Testing of Cast Samples

The samples after post curing were tested for tensile strength, modulus, elongation-at-break, toughness, impact strength and water absorption taking six trials in each case. Morphology of the blends studied using scanning electron micrographs of fractured surfaces

Ageing studies on DGEBA Resin Modified by Epoxy Cresol Novolacs

Cured samples of neat epoxy resin and epoxy resins modified by o-ECN (15%) and p-ECN(15%) were prepared. The samples were aged in a temperature controlled air oven kept at 100^0C for 24,48,72,96 and 120 hours successively. The aged samples were subjected to tensile strength, Impact strength and water absorption

RESULT AND DISCUSSIONS

Modification with Epoxidised Cresol Novolacs

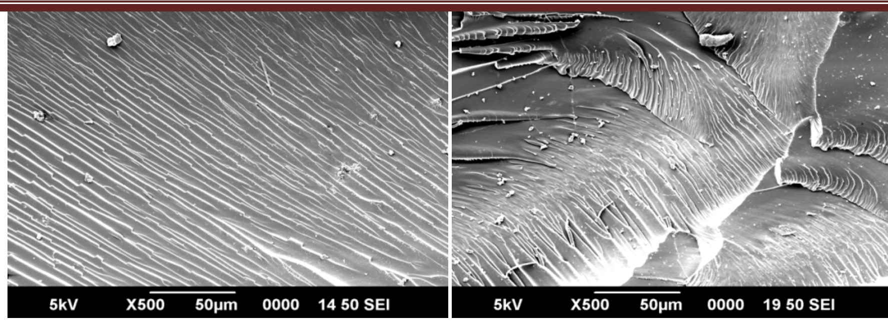
Table.1 showing the properties of epoxy ECN blends. The DGEBA/epoxy novolac blends showed improved mechanical properties and improved water resistance. p-ECN, with a more linear structure result in the absorption of a larger amount of energy . So p-ECN/DGEBA shows better mechanical properties than o-ECN/DGEBA.

Table 1. Properties of epoxy ECN blends

Properties	**Values for Neat DGEBA**	**% Improvement in Properties**	
		p-ECN /DGEBA	**O-ECN /DGEBA**
Tensile Strength (MPa)	55.5	36	20.5
Elongation at break (%)	3.01	46	16.6
Energy absorbed (.J/mm^2)	4.05	75.5	38
Impact strength (J/m)	111.04	98	67
Water absorption (%)	0.2003	-15.12	-10.08

Morphological studies

Fig 1. (a) is a SEM micrograph of the unmodified DGEBA resin. It is a typical case of brittle fracture. Fracture paths show river markings and are mostly straight.



(a) **(b)**

Fig. 1 SEM micrographs of a) DGEBA b) DGEBA/p-ECN

Fig.1 (b) shows the fractured surface of an DGEBA-p-ECN blend.. Multilevel fracture paths with feathery texture indicate energy absorption on a large scale during failure.

Ageing studies on DGEBA Resin Modified by Epoxy Cresol Novolacs

Tensile properties and impact strength

The effect **of** variation of ageing time with tensile strength is shown in Fig.2 Tensile strength decreases during ageing due to the stiffening and thermal degradation of polymer chains. After ageing for 120 hrs the neat resin shows a reduction of 37% in tensile strength while the reduction is 32% in the case of DGEBA/o-ECN and 28% in DGEBA/p-ECN blends.

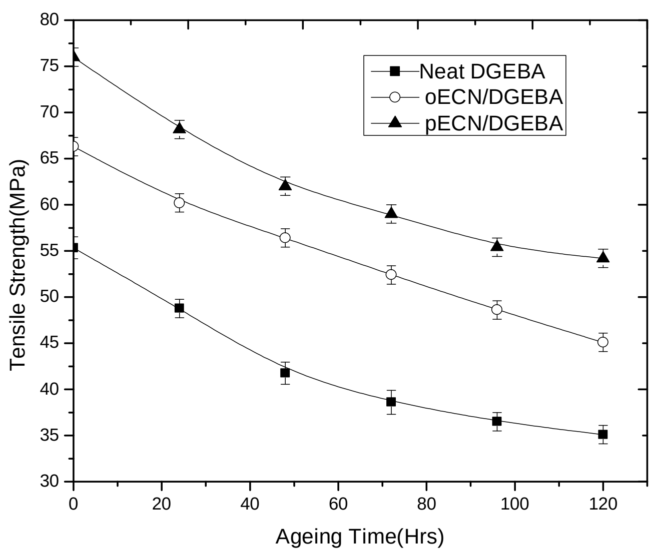


Fig. 2. Tensile strength of modified resin Vs ageing time

The variation in toughness of cured resin (measured as the energy absorbed to break) with ageing time is given in Fig.3. The energy absorbed (to break) of the blends decreases with ageing time mainly due to reduced flexibility of the chains. While the neat resin shows a reduction of 41% in energy absorption at break, the o-ECN and p- ECN blends show a reduction of only 32-27%. This suggests the superiority of these epoxy cresol resins in improving the ageing characteristics of DGEBA resin.

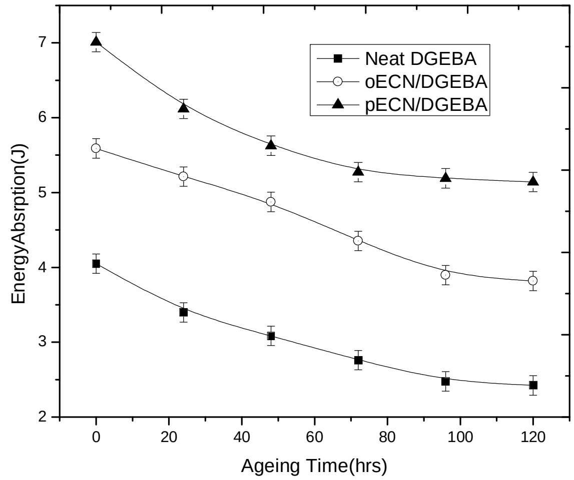


Fig.3 Energy absorbed (to break) of modified resin Vs ageing time

The variation in impact strength of the modified resin during ageing is given in Fig.4. Impact strength decreases sharply during ageing due to stiffening of the polymer chains. However the extent of decrease is less in the blends (p-ECN 27% and o-ECN 29%) compared to the unmodified sample (33%). This confirms the ability of epoxy novolacs to improve the ageing properties.

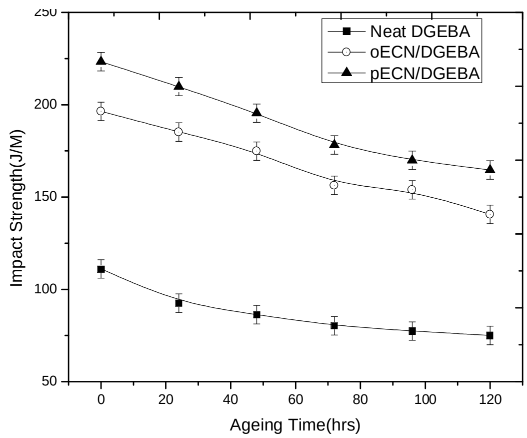


Fig. 4. Impact strength of modified resin Vs ageing time

Water absorption

DGEBA/p-ECN and DGEBA/o-ECN blends show better water resistance than DGEBA resin. This is also due to additional cross-linking accompanying the ageing process.(Fig.5)

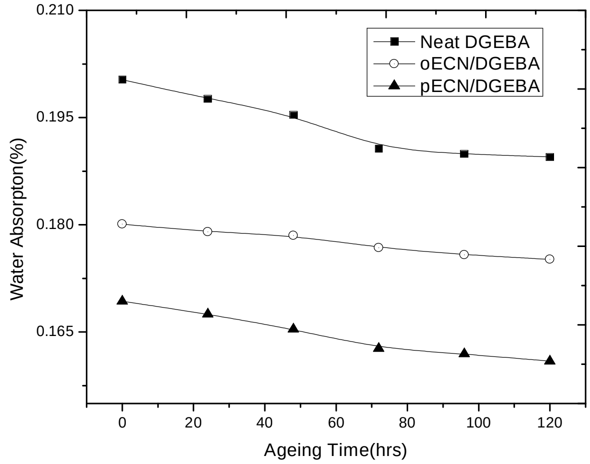


Fig. 5. Water absorption of modified resin Vs ageing time

CONCLUSION

The study reveals that modification using epoxy cresol novolacs not only improves the mechanical properties but also improves the ageing behaviour of the DGEBA resin. The modified resin retains the mechanical properties to a greater extent than the unmodified resin**.** p-ECN modified DGEBA shows better ageing behaviour than o-ECN modified DGEBA.

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