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GLASS FIBER REINFORCED EPOXY RESIN-m-AMINO BENZOIC ACID COMPOSITES

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ABSTRACT

The curing behaviour of epoxy resin-m-amino benzoic acid, the condensation product of epoxy resin namely; diglycidyl ether of bisphenol-A (DGEBA) and m-amino benzoic acid (m-ABA) was studied by differential scanning calaorimetry (DSC). The resultant neat products of DGEBA-m-ABA were characterised by infrared (IR) spectral studies and thermogravimetric analysis (TGA). The glass fiber reinforced composites were prepared and evaluated for their chemical and mechanical properties.

INTRODUCTION

Epoxy resin matrices cured with amines have gained major importance in the fabrication of composites for their extensive application in aeronautic and supresonic appliances.[1,2] The kinetics

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of the curing reaction of epoxy resin with different diamines using the differential scaning calorimetry (DSC) method has been studied by several researcher[3,4]. The reaction of oxirane ring with amine and carboxylic group has been well established, but no systemetic study of the cured reaction of epoxy resin with the compounds having both amino and carboxylic groups (e.g. amino benzoic acid) has been carried out.

The polymer materials based on such reaction have been reported elsewhere[5,6] and a report about such system is confined to patent literature[7]. The present paper comprises the study of the curing reaction betweeen DGEBA and m-ABA at varying ratio by DSC. Glass fiber reinforced composites prepared have been evaluated for their mechanical properties and chemical resistancy.

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EXPERIMENTAL

Materials

All the chemical used were of laboratory grade. Commercial epoxy resin diglycidyl of bisphenol-A (DGEBA) was obtained from Sympol Products Pvt. Ltd., Ahmedabad, India. Specification of epoxy resin are: epoxy equivalent weight: 190 - 210; viscosity at 25°C: 10 P; density at 25°C : 1.17 d/cm^3. E type fiber glass woven fabric (Epoxy Compitible) of 0.25 mm thick (Unnati Chemicals, India) of arral weight 270 gm/m^2 was used for fabrication of composites.

Measurments

Infrared spectra (IR) of the epoxy-resin systems was taken on a Perkin Elmer 983-spectrophotometer.

Thermogravimetry of all the cured samples was carried out on a Linseis thermobalance at heating rate of 10°C/min.

Curing of DGEBA epoxy resin for all the resin system was carried out by Differential Scanning Calorimetry. A Dupont 900 DSC was used for this study. Curing was carried out using single heating rate, 10°C/min. All the chemical, mechanaical and electrical tests of the prepared composites were conducted according to ASTM methods.

Composite Fabrication

A suspension mixture of varying proportions of epoxy resin, DGEBA to m-aminobenzoic acid (m-ABA) (with and without TEA catalayst) was prepared and applied with a brush on a 150 mm x 150 mm epoxy compatible fiber glass cloth. Once dried, the ten plies of prepreg were stacked one on top of another, pressed between steelplates coated with a teflon film release and compressed in a flat plates press under 100 psi pressure, at 120°C for 4 hrs. The post curing of the composite obtained was cooled to 35°C before the pressure was released.

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RESULTS AND DISCUSSION

The IR spectra for all the neat products of DGEBA-m-ABA show no peak corresponding to 990 cm^{-1} of oxirane ring. This indicated that curing reaction exhibit between DGEBA AND m-ABA.

The cure reaction of DGEBA-m-ABA was studied for three different stoichiometric ratios namely 1:1, 1:1.3 and 1:1.5 at a heating rate of 10°C/min. The DSC thermograms showed that all the DGEBA-m-ABA gave single exothermic peaks in the range of 150 to 190°C. The temperature at which curing starts (Ti), peak exotherm temperature (Tp) and completion temperature (Tf) are reported in Table 1. The data (Table 1)reveal that the curing temperature of the epoxy resin depends on the nature of the curing system (presence or absence of catalyst). The amino and carboxylic groups in the m-aminobenzoic acid were responsible for curing of the epoxy resin. The broad exotherm may be attributed to reaction of oxirane ring with amino and carboxylic group exhibit simultaneously.

The varying ratios of DGEBA and m-ABA show no appriciable changes in Ti, Tp and Tf, but, incorporation of triethylamine (TEA) lower the curing temperatures. These may be ascribed to a higher catalytic character of tertiary nitrogen present in the TEA.

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The activation energy (Ea) (Table 1) for such systems have no wide variation. The kinetic parameters Ea and order of reaction (n) were calculated by assuming that the curing reactions obeys Arrhenius type kinetics and that the peak maximum represents a point of constant conversion at a heating rate of 10°C/min. The data indicate that the system in which TEA used require less energy of activation, which also reflect the enhanced catalytic effect of triethylamine.

In order to investigate the thermal stability of the DGEBA-m-ABA resin system, the cured samaples were studied by dynamic thermogravimetric analysis (TGA) at a heating rate of 10°C/m. The TGA data (Table II) show that all the cured resin samples degrade in one step. The percentage weight loss at different temperatures shows that all the resin samaples loses 6-11% weight at 300°C. The degradation rate is maximum at 495°C and degrade completely at 590°C. The aromatic character of m-ABA may play a significant role in the thermal stability of DGEBA-m-ABA system.

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TABLE 1 ; Curing characteristics of DGEBA-m-ABA system

Resin system	DGEBA to m-ABA ratio	Kick off temp. (°C)	Peak temp Tp (°C)	Final temp. TF (°C)	Activation Energy (Ea) Kcal/mol.	Orde of rec-tion
DGEBA-m-ABA	1:1	167	201	218	18.6	1.2
DGEBA-m-ABA	1:1.3	161	192	209	18.4	1.0
DGEBA-m-ABA	1:1.5	154	184	196	18.2	1.1
DGEBA-m-ABA-TEA*	1:1	95	105	117	16.1	1.1
DGEBA-m-ABA-TEA*	1:1.3	88	98	104	16.3	1.2
DGEBA-m-ABA-TEA*	1:1.5	81	91	96	162	1.1

* (Triethyl amine (TEA) added is 1% on the bases of DGEBA taken in the system.)

TABLE 2 ; TGA of unreinforced DGEBA-m-ABA cured materials

Resin system	DGEBA to m-ABA ratio	% wt. loss at °C from TGA				
		300	350	400	500	600
DGEBA-m-ABA	1:1	16	44	72	86	98
DGEBA-m-ABA	1:1.3	12	36	68	82	97
DGEBA-m-ABA	1:1.5	9	33	62	78	95
DGEBA-m-ABA-TEA	1:1	11	34	67	83	97
DGEBA-m-ABA-TEA	1:1.3	9	31	62	73	94
DGEBA-m-ABA-TEA	1:1.5	7	30	61	73	93

TABLE 3 ; Mechanical and electrical properties of glass rein forced composites based on varying epoxy systems.

Resin system	DGEBA to m-ABA ratio	Specific Gravity	Flexural strength (mPa)	Impact strength (mPa)	Hardness (Rock well	Electrical strength (kv/mm)
DGEBA-4-ABA	1:1	1.7	184	203	182	1.8
DGEBA-4-ABA	1:1.3	1.7	190	213	187	1.9
DGEBA-4-ABA	1:1.5	1.7	196	219	193	2.1
DGEBA-4-ABA-TEA	1:1	1.8	201	215	201	2.3
DGEBA-4-ABA-TEA	1:1.3	1.8	212	221	205	2.5
DGEBA-4-ABA-TEA	1:1.5	1.8	219	221	210	2.7

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Glass reinforced composites based on DGEBA-m-ABA resin system show the specific gravity in the range 1.7 to 1.8. The flexural strength, impact strength and hardness (Rockwell) data (Table III) of the composite reflects its mechanical properties, which may be accounted for the aromatic and cross linking character of the cured resin systems. The slight increase in the impact strength of produced systems may be due to high proportions of epoxy resin. The dielectric strength of all the composites is in the range 1.8 - 2.7 kv/mm. The values of electrical strength of the composites are low. This could result in the presence of **a charred path, over which subsequent discharge could take place more and more readily.**

TABLE 4 ; **Weight change (%) in glass fiber reinforced composites on exposure to the chemical reagents at room temperatures.**

Resin system	DGEBA to m-ABA ratio	Wt. change (%) on exposure for 7 days			
		H_2O	CH_3COOH	10% NaOH	10% HCl
DGEBA-m-ABA	1:1	1.12	1.18	3.45	2.32
DGEBA-m-ABA	1:1.3	1.14	1.20	3.47	2.36
DGEBA-m-ABA	1:1.5	1.15	1.21	3.50	2.39
DGEBA-m-ABA-TEA	1:1	1.07	1.09	3.32	2.21
DGEBA-m-ABA-TEA	1:1.3	1.10	1.11	3.21	2.18
DGEBA-m-ABA-TEA	1:1.5	1.7	1.14	3.19	2.15

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Chemical resistance study reveals that the glass fiber composites were not affected by immersion in organic solvents (ketons, alcohols THF etc.). Negligible change in weight or thickness was observed. It was also noted that the exposure to concentrated alkali (10% w/v NaOH) for 7 days resulted in 3.19 to 3.45 percentage weight change of the composite specimen (Table-4). The change in weight may be accounted for the water absorption nature of the composites which may be due to the moisture absorbing residual epoxide, amino and carboxylic acid functional groups present in the DGEBA-m-ABA cured network.

CONCLUSIONS

The reaction of oxirane ring (present in epoxy resin) with amine and carboxylic group plays important role in the curing reaction. All the epoxy resin-m-Aminobenzoic acid resin system were observed to follow a first order Arrhenius type kinetics for thermal degradation having activation energy in the range of 16.1 - 18.6 Kj/mole. The catalytic effect of triethylamine improves the curing rate, as well as increase the mechanical strength and chemical resistivity of the composites.

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