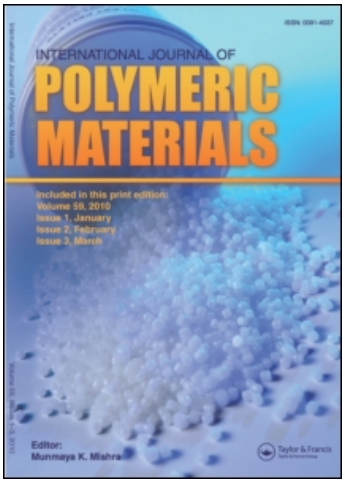

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Epoxy Resin-4-Amino Benzoic Acid Curing Reaction

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The reaction between epoxy resin namely; diglycidyl ether of bisphenol-A (DGEBA) and 4-amino-benzoic acid (4-ABA) at varying ratio was studied kinetically by differential scanning calorimetry (DSC). The resultant neat polymeric products of DGEBA-4-ABA were characterized by infrared (IR) spectral studies and thermogravimetric analysis (TGA). Glass fiber reinforced composites have also been prepared and characterized mechanically.

KEY WORDS Diglycidyl ether of bisphenol-A (DGEBA), 4-aminobenzoic acid, differential scanning calorimetry, glass fiber reinforced composites, mechanical properties.

INTRODUCTION

Epoxy resins have been widely used as an engineering materials because of their extraordinary toughness, good mechanical properties, better chemical and electrical resistance.[1,2] Considerable amount of research work has been done on the relation between the properties of cured resins and structure of epoxy compounds. These studies were mostly based on bisphenol-A type epoxy resin with various types of hardeners like amines, anhydrides[1,2] etc.

Since the reaction of oxirane ring (i.e. epoxy group) with amine and carboxylic[3] group has been well established, it was interesting to study the curing reaction of epoxy resin with the compounds such as aminobenzoic acid having both amino and carboxylic groups. However, the studies of polymeric materials based on such reactions were so far confined to patient literature[4–6] or additives for coatings.[7,8] A systematic study of epoxy resin-4-amino benzoic acid curing reaction and its reinforcing potential has not been reported. The present paper comprises: 1) a kinetic study of the reaction between DGEBA and 4-ABA at varying ratio by DSC, and 2) the characterization of glass fiber reinforced composites (i.e. laminates).

RESULTS AND DISCUSSION

The IR spectra (not shown) for all the neat products of DGEBA-4-ABA show the disappearance of the band at $-910\ cm^{-1}$ associated with oxirane ring. This indicated that curing reaction between DGEBA and 4-ABA has taken place.

The curing reaction of DGEBA-4-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-4-ABA have a single exothermic peaks in the range between 150 to 190°C. The temperature at which curing starts (*Ti*), peak exotherm temperature (*Tp*) and temperature of completion of curing (*Tf*) are reported in Table I. The data (Table I) reveal that the curing temperature of the epoxy resin depends on the nature of the curing system (say, presence or absence of catalyst). The amino and carboxylic groups in the 4-aminobenzoic acid were responsible for curing of the epoxy resin. The broad exotherm may be attributed to the simultaneous reaction of oxirane ring with amino and carboxylic group exhibit.

The varying ratios of DGEBA and 4-ABA show no appreciable changes in *Ti*, *Tp* and *Tf*, but the incorporation of triethylamine (TEA) lowers the curing temperatures. These may be attributed to a higher catalytic effect of tertiary nitrogen present in the TEA.

The values of activation energy (*Ea*) (Table I) for such systems do not vary much. The kinetic parameters *Ea* and order of reaction (*n*) were calculated assuming that 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 is used exhibit lower energy of activation, which also reflects the enhanced catalytic effect of triethylamine.

In order to investigate the thermal stability of the DGEBA-4-ABA resin system, the cured samples were studied by dynamic thermogravimetric analysis (TGA) at a heating rate of 10°C/min. The thermograms obtained (not shown) for the varying DGEBA-4-ABA compositions were similar. 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 samples lose 6–11% weight at 300°C. The degradation rate maximum is around 500°C and degradation is completed around 600°C. The aromatic character of 4-ABA may play a significant role in the thermal stability of DGEBA-4-ABA system.

Glass reinforced composites based on DGEBA-4-ABA resin system show the specific gravity in the range 1.6 to 1.8. Chemical resistance studies reveal that the glass fiber composites are not affected by immersion in organic solvent (ketones, alcohols, THF etc.). No change in weight or thickness was observed. It was also noted that concentrated hydrochloric acid (25% w/v) did not affect the composites. However, exposure to concentrated alkali (25% w/v, NaOH) resulted in change in thickness and weight (Table III). 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 use of high proportions of epoxy resin. The dielectric strength of all the composites is in the range 2.1–2.4 kv/mm. The low values of dielectric strength of the composites can be attributed to the presence of a charred path, over which subsequent discharges could take place more and more readily.

Studies with curing reagents having two or more epoxy curing groups with wide structural variations are in progress.

TABLE I

Curing characteristics of DGEBA-4-ABA system

Resin System	Heating rate 10°C/min.	DGEBA to 4-ABA ratio	Kick off temp. °C	Peak temp. Tp (°C)	Final temp. Tf(°C)	Activation Energy (Ea) Kcal/mol.	Order of reaction
DGEBA-4-ABA	10	1:1	159	189	205	16.8	1.1
DGEBA-4-ABA	10	1:1.3	155	183	196	16.3	1.2
DGEBA-4-ABA	10	1:1.5	150	178	190	16.1	1.0
DGEBA-4-ABA-TEA*	10	1:1	86	97	109	15.5	1.0
DGEBA-4-ABA-TEA*	10	1:1.3	83	90	101	15.5	1.0
DGEBA-4-ABA-TEA*	10	1:1.5	77	87	93	15.1	1.1

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

TABLE II

TGA of unreinforced DGEBA-4-ABA cured materials

Resin system	DGEBA to 4-ABA ratio	% wt. loss at °C from TGA				
		300	350	400	500	600
DGEBA-4-ABA	1:1	11	35	68	81	96
DGEBA-4-ABA	1:1.3	10	33	64	78	95
DGEBA-4-ABA	1:1.5	8	30	60	75	94
DGEBA-4-ABA-TEA	1:1	9	31	63	78	94
DGEBA-4-ABA-TEA	1:1.3	7	29	59	71	93
DGEBA-4-ABA-TEA	1:1.5	6	26	56	67	92

EXPERIMENTAL

Materials

All the chemicals used were of laboratory grade. Commercial epoxy resin diglycidyl either 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:4–10 P; density at 25°C:1.16–1.17 g/cm^3. E type fiber glass woven fabric (Epoxy Compitible) of 0.25 mm thick (Unnati Chemicals, India) of areal weight 270 gm/m^2 was used for fabrication of composites.

Measurements

Infrared spectra (IR) of the epoxy-resin systems were obtained using a Perkin Elmer spectrophotometer.

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

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

Composite Fabrication

A mixture of varying proportions of epoxy resin, DGEBA to 4-aminobenzoic acid (ABA) (with and without TEA catalyst) was prepared and applied with a brush on a 150 mm × 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 about

TABLE III

Mechanical and electrical properties of glass reinforced composites based on varying epoxy systems

Resin System	DGEBA to 4-ABA ratio	% change on exposure to 25% (WN) NaOH		Specific Gravity	Flexural Strength (mPa)	Impact Strength (mPa)	Hardness (Rockwell)	Electrical Strength (Kv/mm)
		Thickness	Width					
DGEBA-4-ABA	1:1	1.1	1.3	1.8	200	218	193	2.1
DGEBA-4-ABA	1:1.3	1.2	1.2	1.8	209	222	199	2.2
DGEBA-4-ABA	1:1.5	1.2	1.3	1.7	215	227	203	2.4
DGEBA-4-ABA-TEA	1:1	1.2	1.2	1.7	218	230	208	2.6
DGEBA-4-ABA-TEA	1:1.3	1.1	1.1	1.8	222	233	211	2.7
DGEBA-4-ABA-TEA	1:1.5	1.2	1.1	1.8	226	236	215	2.8

100 psi pressure, at 120°C for first 4 hrs. The post curing of the compositest obtained was cooled to 35°C before the pressure was released.

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