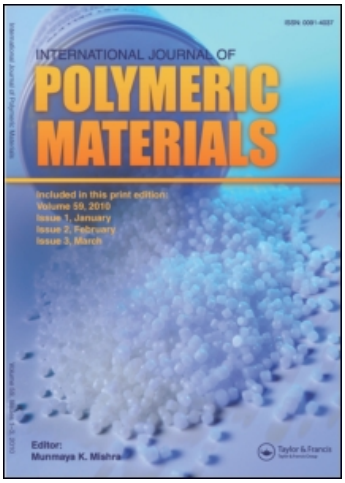

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Novel Epoxy Resin—I

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Novel epoxy resin namely diglycidyl ether of 2,4-dihydroxyacetophenone (DGE-RAP) was prepared and characterized. The curing of DGE-RAP by various diamines was studied kinetically by differential scanning calorimetry (DSC). The cured neat products have been characterized by IR spectral studies and thermogravimetric analysis (TGA). The glass reinforced composites based on such novel epoxy resin-diamine system have also been prepared and characterized.

KEY WORDS 2,4-Dihydroxyacetophenone, [Trivial name: Resacetophenone (RAP)], epoxy resin, DSC, IR spectroscopy, TGA glass fibre reinforcement.

INTRODUCTION

Epoxy resins have been widely used as an engineering materials because of their extraordinary toughness, good mechanical properties, and outstanding chemical and electrical resistance.[1] Considerable amount of research have been made on the relation between the properties of cured epoxy resins and the structure of epoxy compounds. However, these studies were mostly based on bisphenol-A type epoxy resins with various kinds of hardners.[1] Till now, the area in which synthesis and utilization of epoxy resins based on 2,4-dihydroxyacetophenone [i.e. resacetophenone (RAP)] has not been reported so far. Such RAP based epoxy resin may have interesting properties. Hence it was aimed to synthesis and characterized epoxy resin based on RAP.

The present paper comprises the synthesis, characterization, curing by diamines and glass reinforcement of novel RAP based epoxy resin.

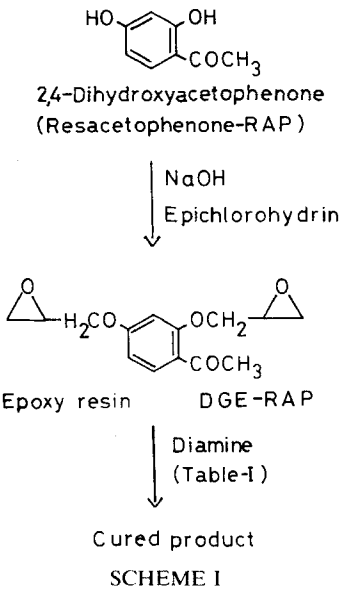
RESULTS AND DISCUSSION

The produced novel epoxy resin based on RAP designated by DGE-RAP was in form of highly viscous material. The C, H contents of DGE-RAP are consistent to its predicted structure (Scheme I). The epoxy equivalent weight of this novel epoxy resin was found to be 140 g/eq. IR spectrum (not shown) of DGE-RAP show discernible band at 910 cm^{-1} due to oxirane ring.

The cure reaction of DGE-RAP-diamine was studied at their two different molar ratios of 1:1 and 1.2:1. The data obtained from DSC scans (not shown) are given in Table I. From the DSC thermograms obtained it was seen that all the epoxy cured system with different ratios of epoxy-diamine gave a single exothermic peak in the range at 65–160°C. The dynamic DSC thermograms for 10°C/min shows that the rate of reaction of major primary and secondary H atoms with epoxide occurs simultaneously.[2] From the thermograms the kick off temperature (T_i) peak, exothermic temperature (T_p) and temperature at completion of curve (T_f) were obtained.

The data reveals that the curing temperature and activation energy of DGE-RAP resin is depending upon the nature of diamine. The diaminodiphenyl methane give cured epoxy resin at lower temperature than other diamines employed in the present work. The kinetic parameters such as E_a (activation energy) and n (order of reaction) were calculated by assuming that the curing reaction obey Arhenius type kinetics and that for peak maximum represents a point of constant conversion for each heating rate. To obtain information about the properties of unreinforced crosslinked materials, larger cured specimens were prepared using the same proportion and temperature as stated in Table I. The unreinforced cured samples (neat products) are dark brown in colour and slightly hard materials that form powder under normal pressure. They are insoluble in all common solvents including formic acid. They are swelled little by 25% w/V alkali. Thermogravimetric analysis of all unreinforced crosslinked materials reveals that all the materials degrade in a single step and start their decomposition at around 100°C degradation become faster between 350°C to 550°C and decomposes almost completely at 700°C (Table II).

IR spectra (not shown) of these unreinforced cure product show clearly disappearance of oxirane ring (~910 cm^{-1}). The inflexions at 1170 cm^{-1} in the spectra of unreinforced product obtained by using high proportion of epoxy may be due



SCHEME I

TABLE I

Curing of DGE-RAP epoxy resin by various diamines

DGE-RAP * -Diamine system	Epoxy diamine ratio	Kick off Temp. T_i(°C)	Peak Temp. T_p(°C)	Final Temp. T_f(°C)	Activation Energy E_a Kcal/mole	Order of reaction
MPD	1 : 1	73	106	152	31.36	0.87
	1.2:1	72	110	150	32.2	1.30
B_2	1 : 1	71	110	154	40.0	1.15
	1.2:1	70	108	159	39.0	0.95
DDM	1 : 1	72	109	149	30.0	0.82
	1.2:1	69	106	143	27.0	1.29

*MPD: 1,3-phenylene diamine

B_2: Benzidine

DDM : 4,4'-Diamino-diphenyl methane

TABLE II

TGA of unreinforced DGE-RAP Diamine Cured material (i.e. Neat products)

Curing system	Epoxy-Diamine Ratio	% wt. loss at °C from TGA				
		250	300	400	500	600
DGE-RAP MPD	1 : 1	3.0	7.9	26	45	88
	1.2: 1	4.0	8.9	28	52	92
DGE-RAP Benzidine	1 : 1	3.1	7.6	29	55	80
	1.2:1	3.8	9.9	31	54	86
DGE-RAP DDM	1 : 1	3.9	8.0	42	60	74
	1.2: 1	4.1	10.2	49	63	78.6

TABLE III

Mechanical and electrical properties of glass-reinforced composite based on DGE-RAP Diamine system

Curing system	Diamine epoxy ratio	Percentage change on exposure to 25 % (w/v) NaOH		Specific gravity	Flexural strength (mPa)	Compressive strength (mPa)	Impact strength (mPa)	Rock-well hardness	Dielectric strength (in air) (KV/mm)
		Thickness	Weight						
DGE-RAP MPD	1 : 1	1.1	1.2	1.81	260	234	280	122	12.3
	1.2:1	1.0	0.9	1.91	120	209	240	126	12.7
DGE-RAP B_2	1 : 1	1.1	1.0	1.60	159	150	153	122	14.3
	1.2: 1	1.1	0.9	1.80	166	179	209	131	13.9
DGE-RAP DDM	1 : 1	0.9	1.1	1.70	160	162	199	130	17.1
	1.2: 1	1.0	1.2	1.76	171	172	209	132	17.0

to ether linkage exhibited due to species at homopolymerization of epoxy resin by tertiary amine formed 'in situ.' However, the homopolymerization of epoxy resin only be possible in the presence of catalyst tertiary amine at elevated temperature.[3]

The glass reinforced composites of DGE-RAP epoxy-diamine system were in the form of dark brown sheets. The specific gravity of these composites is in the range 1.53–1.98 (Table III). Results indicate that there is no appreciable change in the specific gravity with respect to the nature of the diamine and the processing temperature. Chemical resistance studies indicated that the glass fibre composites were not affected by immersion in organic solvents (DMF, ketones, alcohols, DMSO, 1,4-dioxane, THF), no change in weight or thickness was observed. It was also noted that conc. HCl (25% v/v) did not affect the composites. However, exposure to conc. alkali (25% w/v NaOH) resulted in changes in thickness and weight (Table III). The high chemical resistance of all the composites indicates that the diamine aromatic moiety might contribute to high level of crosslinking of epoxy resin with diamine during composite fabrication. Comparison of the mechanical properties of produced glass fibre reinforced composites and those reported about the composites based on commercial epoxy resins (bisphenol based) individually reveals that the produced laminates have comparative mechanical properties.

The dielectric strength of all the composites is in the range 12.3 to 17.1 kv/mm. These values are low. This could result in a charred path over which subsequent discharge could take place more and more rapidly.

EXPERIMENTAL

Materials

2,4-Dihydroxyacetophenone (Trivial name Resacetophenone (RAP)) mp 144°C, was prepared by method reported in literature.[4] All other chemicals used were of Analar grade.

Synthesis of Epoxy Resin Diglycidyl Ether of 2,4 DHAP(DGE-RAP)

This was prepared by method reported[5] for well known epoxy resin namely diglycidyl ether of bisphenol-A (DGE-BA). It was highly viscous material. The epoxy equivalent weight determined by the method reported[6] and was found to be 140 g/eq. Analysis: $C_{14}H_{16}O_5$ (264) C (calcd. %) 63.63, H (calcd. %) 6.06. Found 64.6, Found 6.14.

Glass Reinforced Composites

A suspension of DGE-RAP(I) and diamine (Scheme I) in tetrahydrofuran (THF) was prepared by gentle heating and stirring for 2 hrs. The glass preprages were prepared by applying the mixture on glass matting (6″ × 6″) with brush and the solvent was then allowed to dry up. Ten preprages were stacked together on compression moulded at the temperature and pressure of 160°C and 70 psi, respectively. The composites so obtained was cooled before the pressure was released.

Test specimen were made by cutting the composites on machining to the final dimensions.

Measurements

The C, H contents of all the samples were obtained from Carlo Erba C, H, N, S Elemental Analyser, Italy. IR spectra of all the samples were scanned in KBr pellets on a Perkin Elmer 983 Spectrophotometer. Curing of epoxy resin by diamines was carried out by differential scanning calorimetry. A Du Pont 900 DSC was used for this study. The weight of the sample for this investigation was in the 4 to 5 mg and an empty cell was used as a reference.

Thermogravimetric analysis (TGA) of all the cured products was carried out on Du Pont thermobalance in air at a heating rate of 10°C/min.

All the chemical, mechanical and electrical test of laminates were conducted according to ASTM or IS methods.

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