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Glass Reinforced Composites Based on (Epoxy-Furfural-Acetone) Resin Blends

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A novel Epoxy-furfural-acetone matrix resin (FA resin) has been developed to prepare glass reinforced composites. The polycondensation reaction of FA resin was carried out in a single step under different experimental conditions. This FA resin is used for blending with epoxy resin in presence of various catalysts. The system has been employed to fabricate glass reinforced composites and the laminates have been characterised by mechanical and chemical properties.

Keywords: Composites; epoxy-furfural-acetone resins; glass fiber

INTRODUCTION

Among the thermoset resins the furan resin *e.g.*, furfural-ketone resin gained importance because they are produced from the agricultural waste material. The liquid furfural and furfuryl alcohol both are obtained from the agricultural waste. Since they have remarkable resinification properties, they have reached a prominent position in industry. In their fully cured state, both have remarkable resistance to solvents, acids, bases and heat [1, 2]. These properties and the low cost of furan resin have prompted their uses in various important applications.

Hitherto the blending of such furfural-ketone resin with other curable resin like epoxy resin has not received little attention except a few patents [3]. To investigate the properties of both furan resin and epoxy

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resin such as fast curing, ease of processing, good resistivity of end product *etc.*, it was therefore, interesting to study the blending of furfural-acetone resin (FA resin) with epoxy resin. The present work deals with the synthesis of furfural-acetone resin and its blending with epoxy resin in presence of various catalysts.

The system has been employed to fabricate glass reinforced composites and the laminates have been characterized by mechanical and chemical properties.

EXPERIMENTAL

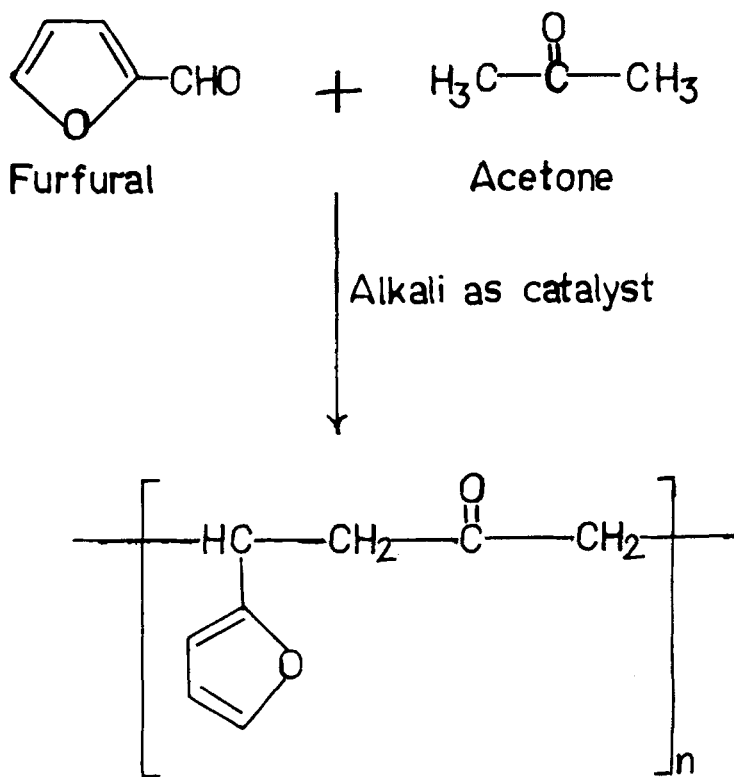
Materials

- (1) Furfural It was obtained from SDS fine chemicals Limited, Boisar, India and purified by vacuum distillation under reduced pressure. Pure furfural is a colourless liquid (bp 162°C) which becomes reddish-brown on exposure to air and temperature due to its self resinification to dark black solid. So furfural was immediately used after vacuum distillation.
- (2) Epoxy resin Commercial epoxy resin was obtained from Synpoil product Pvt. Limited Ahemdabad, India. The specification of epoxy resin are as follows: epoxy equivalent weight 190–210, viscosity 4–10 P at 25°C, density 1.16–1.17 gm/cm³ at 25°C temperature.
- (3) Acetone Acetone was of LR grade and was purified by the method reported in the literature [4]. All other chemicals were of LR grade and were used without purification.

PREPARATION OF FURFURAL-ACETONE RESIN (FA-RESIN)

Fufural-acetone resin (FA-resin) was prepared by the method reported in the literature [5]. The well stirred 2-furfuraldehyde (redistilled, 0.2 mole), acetone (0.4 mole) and alkali (10%, 100 ml) mixture was kept at room temperature for 2 hrs. are refluxed at 60–65°C temperature for 30 min. and at last 75°C for 3 hrs. The reaction mixture was then cooled and poured into water (200 ml). The solid which separated out was filtered and was washed with water and then with acetic acid

(1% 100 ml). It was finally washed with water and was dried in air. The unreacted material was removed by repeatedly treating the product with warm 50% ethanol (100 ml). The resin was yellow coloured powder and the yield was 80%. The prepared FA resin (Scheme 1) softened at 90–95°C temperature.



FA resin

SCHEME 1

COMPOSITE FABRICATION

A typical method of fabrication for composites is given here. A suspension of FA and epoxy resin was prepared on weight basis in

triethylamine (TEA) and was stirred well for 2 minutes. To a 9" × 9" fibre glass cloth, this suspension was applied with a brush and the solvent was allowed to evaporate. The eight dried so prepared samples were stacked one over the other and were pressed between iron plates using the teflon films as mould releasing sheets, and then compressed in a platen press of about 60–70 P.s.i pressure. The preforms were cured by heating them to about 140–160°C temperature for 12 hrs in an air circulated oven. The composites so obtained were cooled to 30°C temperature before releasing the pressure. Test specimens were made by cutting the composites and machining them to final dimensions. All the chemical, mechanical and electrical tests were conducted according to ASTM or IS method.

MEASUREMENTS

Curing of FA-epoxy resin was carried out by Differential Scanning Calorimetry (DSC). A Dupont 900 DSC instrument was used for this study. The instrument was calibrated by using materials of known heat of fusion. Curing was carried out by using a single heating rate (10°C/min) in air. The weight of the sample for this investigation was in the range of 4 to 5 mg. And an empty cell was used as a reference. The data obtained from the DSC measurements are presented in Table I. The chemical resistance of the composites was measured according to ASTM method. The sample size was approximately 20 mm × 20 mm.

TABLE I Curing characteristics of FA-epoxy resin system

<i>Resin system</i>	<i>FA : epoxy resin ratio</i>	<i>Cured onset temp. Ti (C)</i>	<i>Peak temp. Tp (C)</i>	<i>Final temp. Tf (C)</i>	<i>Activation energy Ea(Kcal/mole)</i>	<i>Order of reaction</i>
FA-Epoxy (TEA)	1.1	170	225	235	30.00	0.91
FA-Epoxy (DDM)	1.1	176	217	226	33.51	1.00
FA-Epoxy (PTS)	1.1	202	254	266	40.00	1.11
FA-Epoxy (PTS + DDM)	1.1	206	231	254	40.50	0.92

TABLE II Mechanical and electrical properties of glass reinforced composites based on FA-epoxy resin system

Resin system	FA : epoxy resin ratio	Specific gravity	Flexural strength (mpa)	Impact strength (mpa)	Compressive strength (mpa)	Rockwell hardness	Breakdown potential (in air) (KV/mm)	Percentage change on exposure to 25%(w/v) NaOH Thickness	Weight
FA-Epoxy (TEA)	1.1	1.70	175	200	205	110	12.5	1.0	1.3
FA-Epoxy (DDM)	1.1	1.50	290	285	250	120	13.5	1.1	1.2
FA-Epoxy (PTS)	1.1	1.72	125	150	160	130	17.0	0.9	0.9
FA-Epoxy (PTS + DDM)	1.1	1.68	170	200	190	120	13.5	1.0	0.8

All the mechanical properties were measured on three individual specimen and average results have been presented in Table II.

- (1) **Flexural Strength Test** The measurement of flexural strength of composites were carried out on a Instron testing machine model number A-74.37 at room temperature according to the testing method of ASTM D770. The cross head speed was 100 mm/min.
- (2) **Compressive Strength Test** The compressive strength was measured according to an IS method. The sample size was 12.5 mm × 12.5 mm.
- (3) **Impact Strength** According to the testing method of ASTM D256, the measurements were made through an 120d-type impact tester at room temperature.
- (4) **Hardness Strength** The rockwell hardness strength was measured according to ASTM D785, The sample size was 25 mm × 25 mm.
- (5) **Electrical Testing:** Dielectrical strength measurements were carried out on a high-voltage tester machine oil test set.

RESULTS AND DISCUSSION

The data obtained from the DSC thermogram of FA-epoxy system are presented in Table I. The results obtained from the DSC show that all the system cured independently in presence of catalyst. All the FA-epoxy resin system in presence of catalyst give single exothermic peak with slight inflection in the range of 150–270°C temperature. The DSC thermogram at 10°C/min is shown in Figure 1. From the thermograms the cure onset temperature (T_i), peak exotherm temperature (T_p) and temperature of completion of curing temperature were obtained.

The glass reinforced composites of FA-epoxy resin system were prepared in the form of dark brown sheets. The specific gravity of these composites was in the range of 1.52–1.70 (Tab. II). Chemical resistance studies indicated that the composites were not affected by immersion in organic solvents (DMF, alcohol, DMSO, 1,4-Dioxane, THF), no change in weight or thickness was observed. It was also noted that connected hydrochloric acid (25% v/v) did not affect the composites. However, exposure to alkali (25% w/v NaOH) resulted in change in thickness and weight (Tab. II). The high chemical resistance of all the

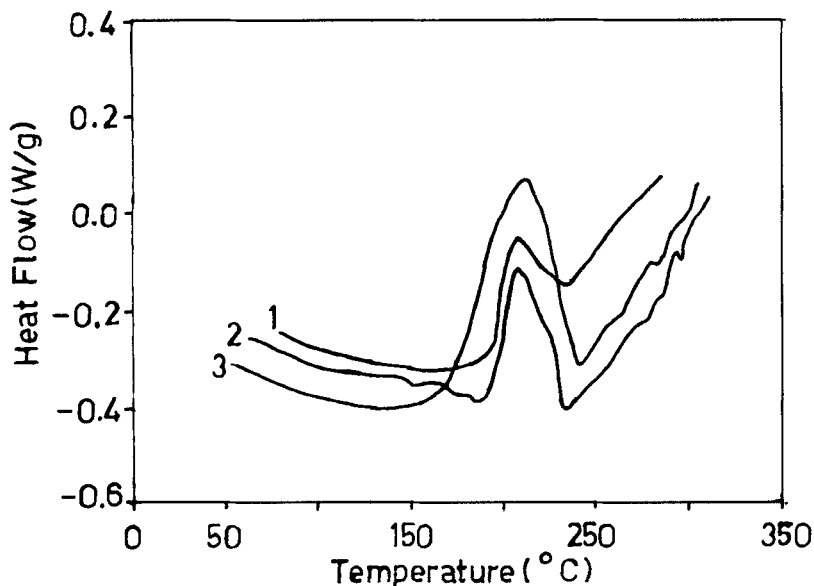


FIGURE 1 DSC Thermograms of: 1. FA-epoxy (TEA); 2. FA-epoxy (DDM); 3. FA-epoxy (PTS).

composites indicates that the furan epoxy resin system might contribute to high level of cross-linking during composite fabrication.

The unreinforced cured product obtained are yellow amorphous powders. They did not melt upto 250°C and were insoluble in mineral acids and organic solvents. The film or cast of unreinforced system is too brittle and therefore hardness and other studies are not presented. Since furan-epoxy resin produce a highly cross-linked and brittle polymeric product, several modifications to FA-epoxy resin system by addition of vinyl monomers, diamines and elastomers have been examined to improve toughness and mechanical properties. Addition of epoxy resin to FA resin may increase the toughness of final product. Such resin system and other polymers have been presented recently [3] and there is no information regarding the properties of these glass fibre reinforced composites. Hence comparison of the mechanical properties of produced glass-reinforced composites and those reported about the composites based on furan resins and epoxy resins individually reveals that the produced laminates have better mechanical properties.

The dielectric strength of all the composites is in the range 13.5–18.2 KV/min. These values are low. This could result in a charred path, over which subsequent discharge could take place more readily. Additionally, minute leakage of current may have arisen from surface contamination.

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