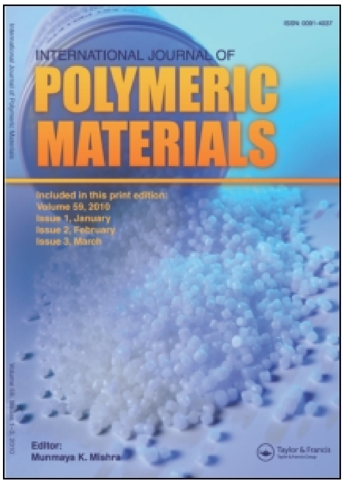

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Coloured Glass-fibre Reinforced Composites

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Coloured glass-fibre reinforced composites of conventional *di*-glycidylether of *bis*-phenol-A and novel heterocyclic pigments, 1,4-*di*-keto-pyrrolo[3,4-c]pyrrole (DPP) were prepared, using *di*-ethylenetriamine as curing agent and evaluated for their physical properties (such as density, % resin content and % void content) and mechanical properties (such as flexural strength, interlaminar shear strength and shore-D hardness). The laminates were also evaluated for their chemical resistance and electric properties. It is observed that the incorporation of DPP pigments imparts colour, but does not deteriorate the above mentioned properties of the DGEBA based glass-fibre reinforced composite.

Keywords: Coloured glass-fibre reinforced composites; heterocyclic pigments; properties

INTRODUCTION

In the recent history of polymers, epoxy resins have gained increasing importance due to their wide range of applications in many fields. The formation of a three-dimensional, infusible, hard thermoset network via a curing process makes these resins very useful in castings, coatings, mouldings, electric components, high-strength composites and hardware applications for aircraft missiles and space structures [1–4]. For this purpose, commercial epoxy resin, *di*-glycidylether of *bis*-phenol-A (DGEBA), is widely used and has been studied by several

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researchers [4–6]. However, not a single reference is reported in the literature giving information about the coloured glass-fibre reinforced composite prepared using DGEBA resin and DPP pigments.

The DPP pigments [7–10] do not solublize at elevated temperature and are known to have very good thermal stability. New DPP pigments are being tested in a number of applications including aerospace, automotive and industrial maintainence [11].

All these encouraging results prompted us to prepare coloured glass-fibre reinforced composites using DPP pigments.

The present paper discusses the preparation and properties of coloured glass-fibre reinforced composites of the above mentioned epoxy resin using four different *di*-aryl-*di*-keto-pyrrolo[3,4-c]pyrrole pigments. The fabricated laminates were evaluated for their physical properties, e.g., density of composites, % resin content, % void content, their mechanical properties, e.g., flexural strength, ILSS, shore-D hardness and their electric properties, e.g., dielectric constant, loss tangent, dielectric loss, resistivity and resistance to chemical reagents.

EXPERIMENTAL

Materials

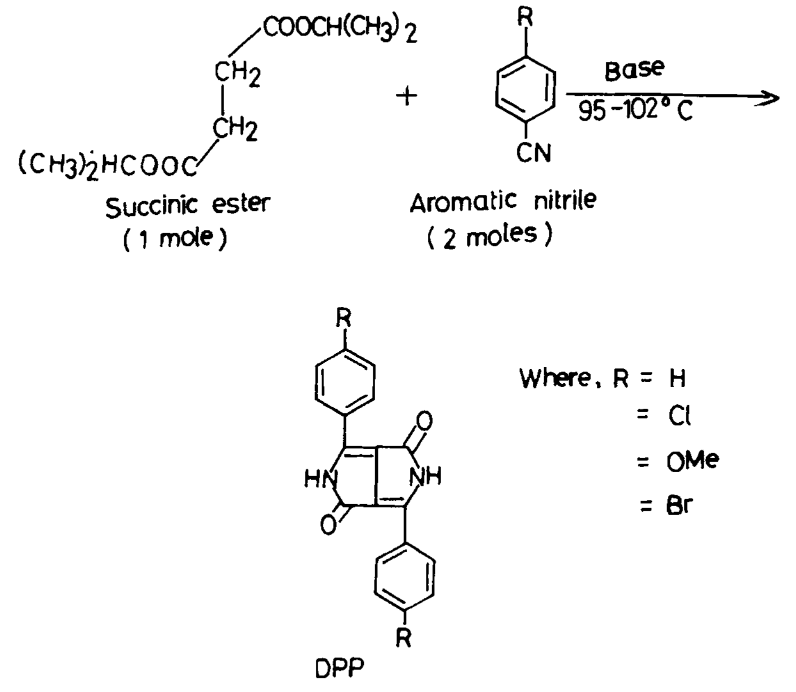
The reinforcement material used for the composite fabrication was 0.25 mm thick, plain weave, epoxy compatible, *E*-type glass cloth purchased from Unnati Corporation, Ahmedabad, India. *Di*-glycidyl-ether of *bis*-phenol-A was obtained from Synpol Products, Ahmedabad (epoxy equivalent weight – 190 $gm \cdot eq^{-1}$). The curing agent *di*-ethylene-triamine (DETA) was laboratory grade reagent.

Synthesis of DPP Pigments

Di-keto-pyrrolo[3,4-c]pyrrole pigments were synthesized as described in literature [12–13]. The synthesis scheme is shown in Scheme 1.

Fabrication of Composites

The samples described in Table I were dissolved in acetone and applied to 12″ × 12″ square piece of woven glass fabric by a hand lay-up



SCHEME 1

TABLE I Composition, codes and colour of glass-fibre reinforced composites

Epoxy resincuring agent system	*Pigments*	*% Pigment*	*Code*	*Colour*
DGEBA + DETA	–	–	A	Colourless
DGEBA + DETA	DPP	2%	B	Reddish orange
DGEBA + DETA	Cl-DPP	2%	C	Red
DGEBA + DETA	MeO-DPP	2%	D	Orange
DGEBA + DETA	Br-DPP	2%	E	Dark red

DPP – 3,6-Diphenyl-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione.
Cl-DPP – 3,6-Bis(4-chlorophenyl)-2,5-dihydro pyrrole[3,4-c]pyrrole-1,4-dione.
MeO-DPP – 3,6-Bis(4-methoxyphenyl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione.
Br-DPP – 3,6-Bis(4-bromophenyl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione.

technique. The impregnated layers were kept in an oven at 60°C for the solvent to evaporate. Ten dried layers prepared in this way were staked one over another between Teflon coated glass cloth and then placed between the platens of a press. The temperature was held at 100°C for 20 min. A pressure of 250 psi was applied and the temperature was maintained at 150°C for 1.5 hour and at 180°C for 2 hours. The mould was cooled to room temperature before the pressure was released; the composite was then removed and cut into specimens for testing as per ASTM standards.

Measurements

Densities of the composite samples were determined by the liquid displacement technique in water. Resin contents and void contents of the composite samples were determined following the method reported in literature [14–15].

All the mechanical properties were tested on a Universal Instron testing machine model 1193 at room temperature (30°C). Flexural strength and interlaminar shear strength (ILSS) were measured using ASTM standards D790-71 and D2344-76 respectively. Shore-D hardness of the composites were measured using a shore-D hardness tester TSE testing machine.

A Hewlett Packard 4329-A high-resistance meter at an applied voltage of 250 V was used to measure an electrical resistivity. The capacitance and dissipation factor were measured on a digital LCR meter.

Resistance to chemical reagents was estimated by ASTM D543-67.

RESULTS AND DISCUSSION

For each of the compositions A to E (Tab. I), the curing agent DETA was used in stoichiometric amount which was calculated by considering epoxy equivalent weight of DGEBA. An attempt has been made to compare the physical, mechanical, electrical and chemical resistant properties of the coloured glass-fibre reinforced composites with that of simple DGEBA composite.

Mechanical Properties

The results for some of the physical and mechanical properties of GFRC prepared by using DGEBA resin and DPP pigments are shown in Table II. The Table II shows that the density and resin content of

TABLE II Physical and mechanical properties of glass-fibre reinfoced composites

System	*Density* ($g\ cm^{-3}$)	*Resin content* (%) *V/V*	*Void content* (%) *V/V*	*Flexural strength* ($Kg\ cm^{-2}$)	*Interlaminar shear strength* ($Kg\ cm^{-2}$)	*Shore-D hardness*
A	1.150	27	4.8	2250	115	86
B	1.180	35	5.0	2260	118	89
C	1.183	30	3.3	2262	120	90
D	1.175	28	4.0	2275	122	89
E	1.173	32	4.8	2269	119	85

GFRC are in the range of $1.150-1.183\,g\cdot cm^{-3}$ and 27.0–35.0% respectively.

Data listed in Table II reveals that the mechanical properties like flexural strength and interlaminar shear strength of the laminates are not much influenced by the addition of various DPP pigments. However, the data regarding shore-D hardness ranges within 85–90 indicate no appreciable penetration due to the load on the surface of laminates.

Dielectric Properties

The values of the electrical properties furnished in Table III show that the coloured glass-fibre reinforced composite can be ranked as a very good insulating material. No significant change in the values of dielectric constant ε' and $\tan\delta$ of the coloured composites were observed as when compared with that of simple DGEBA composite.

Chemical Resistance

The examination of the specimens for chemical resistance shows neither a loss in gloss nor a change in dimensions. However, a weight

TABLE III Electric properties of composites

System	*Dielectric constant* (ε')	*Dielectric loss* (ε'')	*Dissipation factor* *Tan* δ	*Resistance* ($\Omega \times 10^{-15}$)
A	34.79	17.30	0.497	1.0
B	30.59	16.36	0.535	1.0
C	38.34	19.63	0.512	1.0
D	38.70	18.81	0.486	0.95
E	35.53	15.32	0.457	0.95

TABLE IV Percentage weight change of the glass-fibre reinforced composites after exposure to various chemicals for 7 days

System	*Weight change after 7 days (%)*			
	Water	*Acetone*	*20%HCl*	*20%NaOH*
A	2.18	2.40	2.97	3.22
B	2.10	2.32	2.80	3.11
C	2.15	2.38	2.90	3.16
D	2.11	2.38	2.87	3.07
E	2.08	2.40	2.85	3.17

change of the specimens was observed. The results are summarized in Table IV. The changes in weight data show that water absorption increased in all composites.

CONCLUSION

Incorporation of the DPP pigments, imparts colour without any deterioration in the physical, mechanical, electrical and chemical resistance properties of the DGEBA based glass-fibre reinforced composite. It can be concluded from the results of dielectric properties, that the coloured composites can be ranked as very good insulating materials!.

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