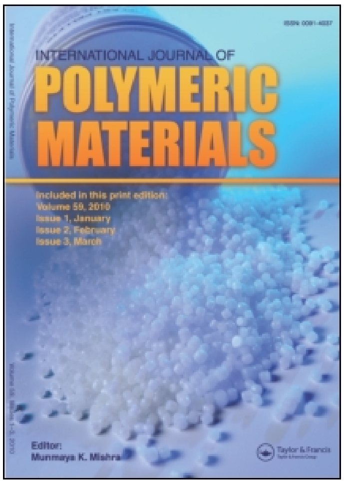

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SYNTHESIS, CHARACTERIZATION, AND PROPERTIES OF ACRYLATED EPOXY RESINS

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Acrylated resins of diglycidyl ether of ethylene glycol and diglycidyl ether of bisphenol-A resins were prepared and characterized by various methods. The curing behavior of the neat resins and their blend were studied with the help of differential scanning calorimetric technique using styrene. Thick films of these resins were prepared, their physical, mechanical and chemical resistance properties were evaluated.

Keywords: acrylated epoxy resins, curing, physical and mechanical properties, chemical resistancy

INTRODUCTION

Acrylated epoxy resins, more commonly known as vinyl ester resins are generally prepared by reacting low molecular weight epoxides with either acrylic or methacrylic acids [1]. Such polymers have the characteristics of epoxy resins but can be cured using vinyl monomers. As the unsaturated groups are present at the end of the structure, the crosslinking is terminal. As a result these resins cure more rapidly than polyester under the same conditions.

The cured resins are tough and their chemical resistance is also good. They have high resistance to cracking during service. In particular, the hydrolysis resistance is much improved by virtue of the fact that there are fewer ester linkages and those that are present are shielded by the crosslink. These resins have better wetting characteristics due to their epoxy nature. Because of these wide range of properties, they are becoming increasingly important in coatings, automotive parts, printed circuit boards, building materials and fiber-reinforced composites [2–5]. Looking to these applications, vinyl ester resins were prepared, characterized and the effect of styrene content on the properties of the cured resins were investigated by preparing thick films.

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EXPERIMENTAL

Materials

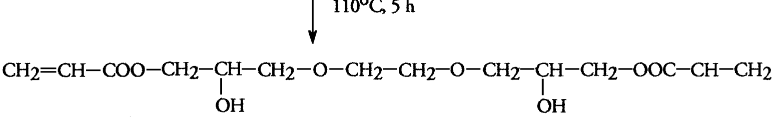
All the chemicals used were laboratory grade. Benzoyl peroxide was crystallized in chloroform prior to use. Diglycidyl ether of bisphenol-A (DGEBA) and diglycidyl ether of ethylene glycol (DGEEG) were prepared by the methods described in literatures [6, 7]. Vinyl ester resin of diglycidyl ether of bisphenol-A (ADGEBA) was prepared using the reported method [8].

Synthesis of Vinyl Ester Resins

Diglycidyl ether of ethylene glycol (100 gm) was reacted with acrylic acid (38 gm) at 110°C for 5 h using 0.1%(w/w) triethylamine as a catalyst and 0.1%(w/w) hydroquinone as stabilizer for unsaturated groups. After the completion of reaction the mixture was washed with distilled water and the unreacted monomers and water were removed under vacuum to get an amber-colored liquid resin (acrylated diglycidyl ether of ethylene glycol, ADGEEG) having the structure shown in Scheme 1.

Characterization

The number average molecular weight of ADGEEG was determined by vapor pressure osmometry and was found to be 312, the intrinsic viscosity in chloroform as measured by Ubbelohde viscometer at 30°C was 0.028 dl · g^{-1}. Acid value [9] was determined to be 0.31. This resin was also characterized by infrared spectroscopy. The IR spectrum of ADGEEG resin was scanned as neat thin film on a sodium chloride disk on a Perkin Elmer 983 spectrophotometer. A strong absorption band at 1730 cm^{-1} indicates the presence of ester group. The absence of characteristic absorption bands of epoxy at 920 cm^{-1}, 860 cm^{-1} and 1120 cm^{-1} proves that almost all epoxy groups were reacted with acrylic acid. The absorption band at 1640 cm^{-1} and 975 cm^{-1} are due to stretching and bending vibrations of vinylic group. The



ADGEEG

SCHEME 1

ADGEBA resin has the number average molecular weight 480 and intrinsic viscosity 0.04 dl $\cdot$ g^{-1} as measured by the above methods.

Curing Procedure

The curing behavior of the resin systems were studied by differential scanning calorimetry (DSC) technique on a Du Pont 900 differential scanning calorimeter [10]. Various amounts of styrene (10 and 20%) and 1% benzoyl peroxide (curing catalyst) were added to vinyl ester resins. These were mixed well, placed in a vacuum oven to remove gas bubbles and were used for DSC studies at a heating rate of 10°C $\cdot$ min^{-1} using empty cell as reference.

Preparation of Films

A teflon sheet of 2.5 mm thick was punched to the required size (0.2 × 5 × 9 cm). This sheet was placed on a triangular leveling plate and leveled by moving the rotating screw of the leveling plate. Required amount of acrylated resins was mixed thoroughly with styrene and benzoyl peroxide by placing the beaker on a magnetic stirrer for 5 min, kept in a vacuum oven to remove bubbles and finally poured on the prepared side of the teflon sheet. The plate was kept inside an oven at 70°C for 3 h. Post curing of the films were done at 140°C for 1.5 h.

Testing of the Films

Dimensions of the films were measured with the help of micrometer screw. Shore D hardness of the films were measured using shore-D hardness tester TSE testing machine. Flexural and tensile testing of the films were measured by ASTM D790-71 & D 636-76 using Instron Model 1121 at room temperature. The chemical resistance properties in various chemicals were also determined.

RESULTS AND DISCUSSION

The curing behavior of vinyl ester resins can be understood from DSC technique. Table 1 shows the curing characterization of vinyl ester resins in presence of 10% and 20% styrene contents. Curing parameters such as the temperature at which curing reaction is initiated (T_i), peaked (T_p) and completed (T_f) can be obtained from the DSC thermograms while activation energies (E) of the curing reactions were calculated by Freeman Carroll relation [11]. The exotherms obtained for various systems range in between 100 – 185°C. Increase in the styrene content from 10% to 20% does not make any significant change in the curing characteristics.

TABLE 1 Curing characteristics of vinyl ester resins with different styrene contents

Resin systems	*Styrene content* (%)	T_i^{a} (°*C*)	T_p^{b} (°*C*)	T_f^{c} (°*C*)	$E \pm 2^{d}$ $kJ \cdot mol^{-1}$
ADGEBA		105	145	180	84.5
ADGEEG	10	109	155	185	92.3
ADGEBA + ADGEEG (80 : 20)		105	150	180	90.4
ADGEBA		105	145	170	82.1
ADGEEG	20	100	150	170	90.4
ADGEBA + ADGEEG (80 : 20)		105	148	180	85.4

ADGEBA, Acrylated diglycidyl ether of bisphenol-A.
ADGEEG, Acrylated diglycidyl ether of ethylene glycol.
[a] T_i Temperature of the onset of curing.
[b] T_p Temperature of the peak of the exotherm.
[c] T_f Temperature of the completion of curing.
[d] E Activation energy of the curing reaction.

TABLE 2 Physical and mechanical properties of thick films

Resin systems	*Styrene content* (%)	*Shore D hardness*	*Flexural strength* $Kg \cdot cm^{-2}$	*Tensile strength* $Kg \cdot cm^{-2}$	*Elongation* (%)
ADGEBA		75	638	223	7.8
ADGEEG	10	62	588	192	8.6
ADGEBA + ADGEEG (80 : 20)		70	612	208	8.2
ADGEBA		79	602	247	7.9
ADGEEG	20	60	548	201	8.2
ADGEBA + ADGEEG (80 : 20)		75	589	227	8.4

Data listed in Table 2 shows the physical and mechanical properties of the films. Shore D hardness of the films were measured. As the styrene content increases Shore D hardness increases. In mechanical properties, as styrene content increases the tensile strength increases while flexural strength decreases. The decrease in flexural strength with styrene content may be due to the increase in crosslinking density which results in rigidity [8].

Data of Table 2 also reveal that the properties are quite inferior for ADGEEG resin compared to the ADGEBA resin. This may be due to the presence of simple long hydrocarbon backbone in ADGEEG resin. The presence of aromatic rings in the ADGEBA resin make them rigid and strong, resulting in better mechanical properties. The above results enabled us to make blends of these resins to obtain improved properties. The mechanical properties of the mixture of these two resins ADGEBA (80 parts) ADGEEG (20 parts) lie in between those of the neat resins.

Chemical resistance properties of these resins were measured by dipping the samples in methanol, 10% sodium hydroxide and 10% hydrochloric acid solutions for 10 days. No change in color was observed. In each case not more than 0.05% weight change was observed. The hydrolytic resistance of all the films were also evaluated and found to be very good. Neither a change in weight nor a change in dimensions were observed.

CONCLUSION

The data obtained in the curing reaction were utilized in the preparation of the films. Present study reveals that ADGEEG resin can be used in high build up formulations in presence of ADGEBA resin.

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