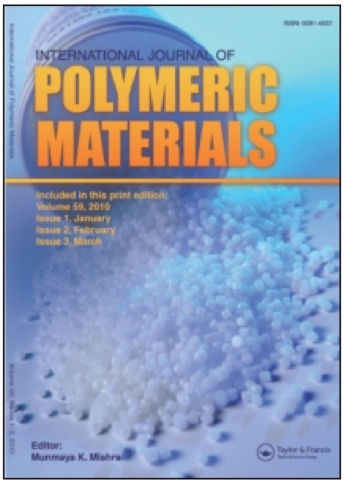

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Novel Vinylester Resin Based on Hydroquinone as Coating Materials

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Novel Vinylester resin of *p*-dihydroxy benzene (VER-HQ) was prepared by the reaction between diglycidyl ether of hydroquinone and acrylic acid. The VER-HQ resin was characterized by its viscosity, acid value, number averge molecular weight and IR spectrophotometry. Curing conditions for the resin were established by differential scanning calorimetry. Thermal stability of the resin was studied by thermogravimetric analysis.

The VER-HQ resin was coated on mild steel panels and the coated panels were tested for their flexibility, impact strength, scratch hardness and chemical resistancy.

Vinyl monomer such as styrene lowers the curing temperature of all resin systems when incorporated prior to curing and significantly improves the properties of coating.

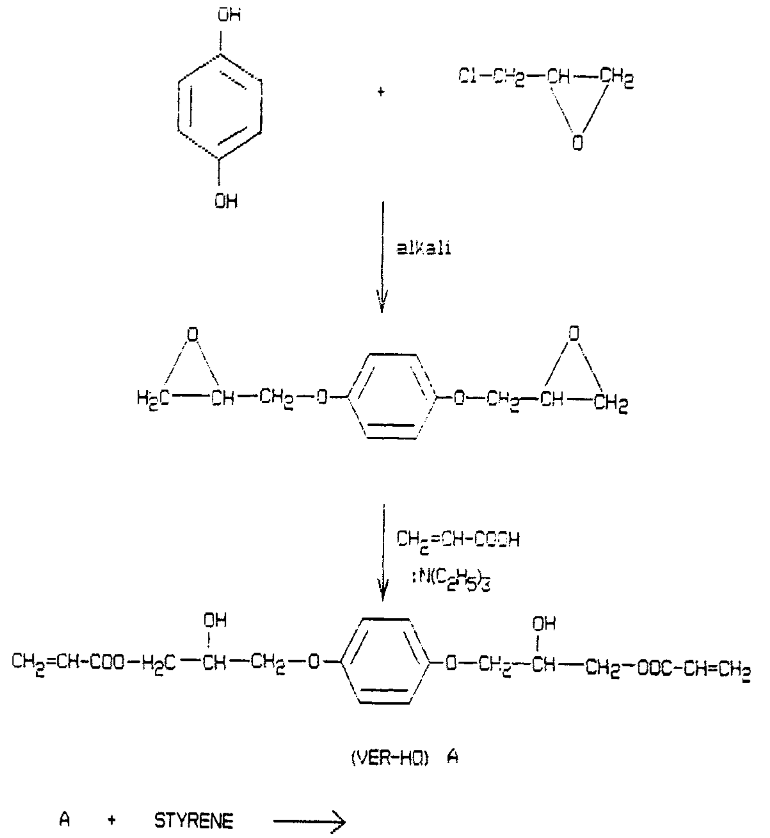
Keywords: Vinylester resins; hydroquinone; coatings; curing; properties

INTRODUCTION

Vinylesters are used for the manufacturing of automotive parts, laminates and coatings [1–7]. Hence it was appropriate to extend the study of properties to vinylester resin based on hydroquinone VER-HQ. The present work deals with the novel vinyl ester coating material (VER-HQ) coated on mild steel panels. The synthesis is presented in Scheme 1.

RESULTS AND DISCUSSION

The viscous resin VER-HQ is soluble in common organic solvents such as acetone, benzene, dioxane, tetrahydrofuran, N,N-dimethyl formamide etc.



Scheme 1

The number average molecular weight of the resin determined by vapour pressure osmometry was about 382, while intrinsic viscosity in chloroform at 30°C measured by Ubellholde viscometer was observed to 0.042 dlg^{-1}. The acid value and iodine value of VER-HQ resin were about 0.23 and 26.0 respectively.

Some IR spectral characteristic frequencies of VER-HQ at 3555 cm^{-1}, 1720 cm^{-1}, 1630 cm^{-1} due to hydroxyl group, ester group and double bond of the vinyl group respectively were noted.

The DSC scans obtained for the resin compositions described in Table I were analyzed to obtain the various important characteristic temperatures [9]. Such as initiation temperature T_i, peak exotherm

TABLE I Curing characteristics and kinetic parameters for curing of VER-HQ using peroxide (1% by weight) as catalyst

Resin System	*Styrene Content (% by wt)*	T_i (°C)	T_p (°C)	T_f (°C)	E_a (±2 KJ mole^{-1})	*Order of Reaction n*
VER-HQ	–	106	145	180	83.8	1.12
VER-HQ	10	99	138	172	78.5	1.11
VER-HQ	20	92	130	160	73.3	1.09
VER-HQ	30	90	125	156	70.7	1.04
VER-HQ	40	87	118	150	68.1	1.01
VER-HQ*	–	104	141	180	80.6	1.10
VER-HQ*	10	97	134	174	75.3	1.07
VER-HQ*	20	90	126	167	70.2	1.05
VER-HQ*	30	87	120	155	68.2	1.02
VER-HQ*	40	84	113	144	66.7	0.99

VER-HQ* = Addition of N,N'-dimethyl aniline as promoter to the system.

temperature T_p and curing completion temperature T_f for the curing reactions. The data suggested that incorporation of vinyl monomer such as styrene in this resin system prior to curing lowers the curing temperature.

The values of activation energy and order of reaction from the data analysis were carried out by the methods reported in literature [9, 10], All the reaction followed simple n^{th} order type Arhenius kinetics, having order of reaction about one.

From the TGA curves, the value of T_{max}, (The temperature at which maximum decomposition occured) was 390 for VER-HQ. The glass transition temperature T_g decreases on the incorporation of styrene into the resin system. The observation of a single glass transition temperature for each system, indicates the very good miscibility of styrene with a system.

The resin coated on the mild steel panel was cured thermally using benzoyl peroxide (1% by wt of resin) as catalyst.

Data given in Table II present the scretch hardness, impact strength and flexibility test of the coating on the mild steel panel using vinyl ester resin (VER-HQ) system. The coating applied using brush produced flexible and adherent coating which were backed at 120°C for 1 hour.

All resin formulations from VER-HQ passed flexibility test on 1/4″ and 1/8″ conical mandrel, which is expected for vinylester based coatings. All the coated panel passed stripping test for adhesion. This

TABLE II Flexibility, impact strength and stretch hardness of the vinyl ester resin (VER-HQ)

Resin	*Styrene Content (% by wt)*	*Flexibility*	*Impact Strength*	*Scratch hardness (gm)*
VER-HQ	–	Pass	118	820
VER-HQ	10	Pass	132	860
VER-HQ	20	Pass	139	935
VER-HQ	30	Pass	146	990
VER-HQ	40	Pass	157	1075

might be due to free hydroxyl group present in vinylester resin which contributes to the strong metallic bond formation with the surface of mild steel panels.

The values of scratch hardness and impact strength observed in VER-HQ resin systems may be responsible for the unsaturation present in the resin system.

The incorporation of styrene in VER-HQ should improve its properties as it may increase the thoughness of the resin system. On incorporation of N,N-dimethyl aniline (0.1% w/w) as a promoter for curing, the curing reaction becomes more rapid.

The panels immersed for 72 hours in water and solvent, showed no cracking, blistering or change in colour which indicates that the resin system have excellent solvent resistancy.

On exposing the coated panels to 2% acid and 2% alkali solution, no substantial loss in gloss or change in appearance were observed indicating excellent adhesion and also good resistance to acid and alkali.

EXPERIMENTAL

Materials

All the chemicals used were of laboratory grade.

Synthesis of VER-HQ Resin

Vinyl ester resin based on hydroquinone (VER-HQ) was prepared by the method which are described in our earlier communication [8].

Characterization of VER-HQ Resin

IR spectra were recored on a Perkin-Elmer IR 983 spectrophotometer using KBr cell. DSC scan were recorded on Du pont 9900 thermal analyzer connected to a Du pont 910 differential scanning calorimeter module which was used to measure the heat flow as a function of temperature. TGA analysis of the resin was carried out by Du pont model 951 thermal analyzer at heating rate 10°C/min.

Panel Coatings

Various resin compositions were formulated using ethyl cellosolve as thinner. These compositions were made free from coarse skin by passing them through a sieve, 150 μm [IS:460–1960] and applied on mild steel panel using flat brush (IS:384–1964). The coated panels were kept vertically for flat drying. After specific times, the coated panels were examined for tack free test and then cured thermally.

Measurements

All the coated panels were tested for flexibility, scretch hardness, Impact strength and chemical resistance by following ASTM standard method. Flexibility of the coated film was tested on 1/4″ and 1/8″ conical mandrel. The scratch hardness of the film was mearued using mechanically operated scheen Scratch Hardness Tester. The chemical resistance of the film was studied by dipping separately in 2% sulphuric acid, 2% NaOH solution and acetone for specific time at room temperature.

Acknowledgement

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