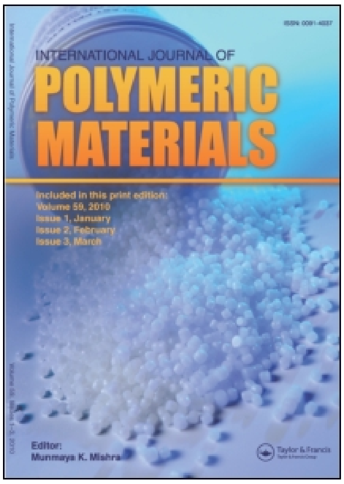

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Surface Coating Studies of Alkyd-Castor Oil-Epoxy Resin Condensate-Ketone Resin Blends

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Castor oil (CO) was reacted with commercial epoxy resin (ER) (diglycidylether of bisphenol-A, DGEBA) at various mole ratios. The resultant products were designated as COERs and characterized by physical, chemical and IR spectral studies. A commercial alkyd resin was blended with various proportions of COERs and ketone resin (i.e., cyclohexanone-formaldehyde resin) (CHF). All the blends were applied on mild steel panels and characterized for drying time, adhesion, flexibility, hardness, impact resistance and chemical resistance properties.

Keywords alkyd resin, blends, castor oil-epoxy resin condensate (COERS), coating, cyclohexanone-formaldehyde resin (CHF), epoxy resin (DGEBA)

INTRODUCTION

The alkyd resin is a prime candidate for surface coating materials. It is generally manufactured from phthalic anhydride, polyol and drying oils. In order to improve properties of alkyd resins, modification with various other materials is the best method. These other materials may be present in physical or chemical combination. The modification of alkyd resin with

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cellulose nitrate gives fast-drying, and modification of alkyd resin with chlorinated rubber gives good fire-resistance. Alkyd resin is also used in coating applications [1].

Castor oil, known primarily for its medicinal use as a cathartic, is now also used as an industrial raw material for the manufacturing of products used in coatings, surfactants, dispersants, cosmetics, and lubricants, etc. Castor oil serves as raw material for the manufacture of a number of industrial products like polyurethane coatings, adhesives and casting compounds, to increase hydrolytic stability and electrical insulating properties. Castor oil is also used in coating applications, in surfactants, in lubricants and in the textile industries [2–6].

Epoxy resins are versatile resins having a wide range of properties such as adhesion to substrate, corrosion resistance and high tensile, flexural and compressive strengths. Because of the versatile properties exhibited by epoxy resin it has found a number of applications [7,8].

Ketonic resins (CHF) play a prime role in the field of coating as an excellent additive due to their high compatibility and solubility. Ketonic resin (CHF) in a blend with other polymers may serve as a modifier for other polymers, or vice versa, to improve properties. Preparation of alkyd-ketonic (CHF) blends have been reported [9,10] and the effect of the blend ratio on various coating properties was studied.

A literature survey about ketonic resin (CHF) reveals that when ketonic resin is used in a blend with other polymers, it can improve some properties of the blend. Hence, it was thought interesting to blend ketonic resin (CHF) with alkyd-COERs blends and study the film properties of the resulting alkyd-COERs-CHF resin blends.

EXPERIMENTAL

Materials

Castor oil was purchased from the local market. Specifications of castor oil: (1) Viscosity at 28°C, 130 s (2) Density at 28°C, 0.95 g/cc

Epoxy resin (i.e., DGEBA) was obtained from Atul Industries Limited (Valsad, India). Specifications of epoxy resin: (1) Epoxy equivalent weight, 190 (2) Viscosity 40–100 poise at 25°C (3) Density at 28°C, 1.16–1.17 g/cc.

Alkyd resin was procured from Pacific Paints (V.U. Nagar, India). Specifications of Alkyd resin: (1) Viscosity at 28°C, 130 ± 10 s.

Cyclohexanone-formaldehyde resin and other chemicals were obtained from Chiti-Chem. Corp. (Baroda, India). Specifications of CHF resin: (1) Physical form, pearl-shaped granular solid (2) Melting point, 90–95°C.

All other chemicals used were of pure grade.

Table 1: Mole ratios of CO: ER and physical properties of COERs.

Mole of reactants taken					
Castor oil	**Epoxy resin**	**Designation**	**Appearance**	***Viscosity in sec.**	**Sp. gravity in g/cc**
0.268	0.134	COER-1	Pale yellow and clear	135	0.98
0.268	0.268	COER-2	Dark yellow and clear	240	1.01
0.268	0.402	COER-3	Brownish yellow and clear	290	1.05

*Viscosity time was measured using ford cup type ``B'' IV (at 30°C).

Preparation of Castor Oil-Epoxy Resin Condensate Products (COERs)

Castor oil (0.268 mole) was taken in a three-necked flask equipped with a mechanical stirrer and placed in a water bath where the temperature was maintained at 70–80°C. Under continuous stirring, the desired amount of epoxy resin (as shown in Table 1) was added with 0.05% of triethylamine (added as a base catalyst). At a regular interval of times, a sample was withdrawn from the reaction mixture using a siphoning device and a test was performed for the negative epoxy group [11]. When the sample showed the negative test for the epoxy group, reaction was stopped and the product was allowed to cool to room temperature. The resultant products were designated as castor oil-epoxy resin (COERs) products. The varying type of mole ratios of castor oil (CO): epoxy resin (ER) used for the preparation of COERs and physical properties of the resulting products (COERs) are given in Table 1. Chemical properties of the products (COERs) are given in Table 2.

Preparation of Alkyd-COERs-CHF Blends

The CHF resin was obtained in solid granular form and was dissolved in a solvent mixture [9] having following composition: Toluene: 60 (wt%), Cyclohexanone: 20 (wt%), n-Butanol: 20 (wt%).

Table 2: Chemical properties of COERs.

Type of COERs	Hydroxyl number	Hydroxyl value in mg of KOH/g	% hydroxyl	Number of unsaturation per molecule
COER-1	3	145	4.56	3
COER-2	3	130	3.89	3
COER-3	3	113	3.37	3

Table 3: Proportions of alkyd-COERs-CHF blends.

Type of COERs	Weight of alkyd resin taken in g	Weight of COERs taken in g	Weight of CHF resin taken in g	Designation
COER-1	100	10	10	ACCH-101
	100	10	20	ACCH-102
	100	10	30	ACCH-103
COER-2	100	10	10	ACCH-201
	100	10	20	ACCH-202
	100	10	30	ACCH-203
COER-3	100	10	10	ACCH-301
	100	10	20	ACCH-302
	100	10	30	ACCH-303

The solid CHF resin was dissolved in the above-described solvent mixture to prepare 50% solutions of resin which was then utilized to prepare alkyd-COERs-CHF blends.

In a three-necked round-bottom flask equipped with a mechanical stirrer, alkyd resin was charged and stirred for 5 min. Under continuous stirring, the desired amount of specific COERs was added, and mixing allowed for 15 min. Solution of CHF resin thus prepared, was then slowly added to COER-alkyd mixture with continuous stirring. Upon completion of the addition of CHF resin solution, the final alkyd-COER-CHF mixture was stirred for 30 min to have a homogeneous mixture. After stirring, the mixture was kept in a cylindrical glass container overnight to check for any tendency of separation of layers. In neither case separation of distinct layers was observed. The proportions of alkyd-COERs-CHF blends along with designations are given in Table 3.

Preparation of Coating Composition Based on Alkyd-COERs-CHF Blends

To study the film properties of alkyd-COERs-CHF blends, the coating compositions of various blends were prepared in the following manner:

50.0 g of the desired blend was taken in a 250 ml glass beaker. The driers lead octoate (18% Pb), cobalt octoate (6% Co) and manganese octoate (6% Mn) were added to it in the proportions of 0.5, 0.05, and 0.05%, respectively. The resultant blends of alkyd-COERs-CHF resins were diluted with the above solvent mixture [9] to obtain a viscosity appropriate for application with a brush on clean mild steel panels.

Panel Preparation

The mild steel panels were first degreased in alkali solution and subsequently swabbed with xylene to remove any type of oily material or contaminants. After the xylene has evaporated, the panels were coated by the above-prepared coating composition.

FILM CHARACTERIZATION

The coated panels were examined for drying time, adhesion test, flexibility test, scratch hardness, pencil hardness, impact resistance and chemical resistance by standard methods. The results are given in Tables 4–6, respectively.

Determination of Drying Time

Mild steel panels were used to determine the air-drying time of films of various coats. The panels were prepared in the above manner and coating compositions were applied. The films were checked for "surface dry" and "tack-free dry" stages at regular intervals of time. The results are given in Table 4.

Determination of Adhesion Time

Adhesion of films to substrate was determined by performing cross-hatch adhesion test the panels for the test were prepared in the manner described above. Cross-hatch adhesion test was carried out after 168 hours of coating application. The results are given in Table 4.

Determination of Flexibility

For the determination of flexibility, tinned mild steel panels were used. The coating compositions were applied and cured in the manner mentioned above. Flexibility test were carried out using mandrels having specific rod diameter. Generally 1/8 inch rod diameter mandrel was used and if film

Table 4: Properties of films prepared from alkyd-COERs-CHF blends.

	Drying time in minutes			
Designation	**Surface dry**	**Tack-free dry**	**Adhesion**	**Flexibility**
ACCH-101	80	240	F	F
ACCH-102	65	215	F	P
ACCH-103	55	195	P	P
ACCH-201	55	210	P	F
ACCH-202	50	195	F	P
ACCH-203	40	180	P	P
ACCH-301	55	210	P	P
ACCH-302	40	190	P	P
ACCH-303	30	170	P	P

P = Pass, F = Fail.

Table 5: Mechanical properties of films prepared from alkyd-COERs-CHF blends.

Designation	Scratch hardness in g	Pencil hardness	Impact resistance in lb
ACCH-101	2200	2H	125
ACCH-102	2400	3H	125
ACCH-103	2400	4H	150
ACCH-201	2400	3H	125
ACCH-202	2600	4H	150
ACCH-203	2600	4H	175
ACCH-301	2600	4H	150
ACCH-302	2800	5H	175
ACCH-303	2800	5H	200

passed through 1/8 inch mandrel then it was said to pass the flexibility test. The results are given in Table 4.

Determination of Scratch Hardness

In this method, a hand-operated instrument was used in which a coated panel was scratched under specific load with a needle in contact with the film on the test panel. The load was increased until the film was scratched, which was indicated by a light bulb that glows when film is scratched. The results are given in Table 5.

Determination of Pencil Hardness

In this method, pencils having different hardness were used. Sharp tipped pencils having hardness 4B (soft) and 6H (hard) were used to scratch the film.

Table 6: Chemical resistances properties of films prepared from alkyd-COERs-CHF blends*.

Designation	Acid resistance 5% HCl 24 h	Alkali resistance 3% NaOH 2h	Water resistance (Dist. Water) 168 h	Solvent resistance Xylene 168 h
ACCH-101	2	3	3	2
ACCH-102	3	4	2	2
ACCH-103	4	4	1	3
ACCH-201	3	4	4	3
ACCH-202	4	4	3	4
ACCH-203	4	5	3	4
ACCH-301	4	4	3	4
ACCH-302	5	5	4	5
ACCH-303	5	5	4	5

*0 = film completely removed.
*1 = film removed and particularly cracked.
*2 = film partially cracked.
*3 = loss in gloss.
*4 = slight loss in gloss.
*5 = film largely unaffected.

The pencil was held approximately at an angle of 45° to the film and with uniform pressure pulled down over the length of the film. The test was repeated till a pencil with specific hardness was able to scratch the film. The hardness of that pencil was reported as the pencil hardness test. The results are given in Table 5.

Determination of Impact Resistance

The coated test panels for the impact resistance test were prepared in the manner described above. The test was carried out after 168 hours of coating application. The coated panel was kept on a platform (coated side upward). The panel was then indented with an object of specific weight from varying heights. The test was repeated by increasing the height from which the object falls till the film was cracked or detached. The results are given in Table 5.

Determination of Chemical Resistance Properties

For the assessment of chemical resistance of the films to various chemicals, tinned mild steel panels were used which were prepared, coated and cured as mentioned above. The panels were immersed vertically in the baths containing solutions of different chemicals in specific concentration at room temperature for the specific time period. Upon completion of the specified time period the panels were removed from the baths and allowed to dry before visual examination. The results of chemical resistance are given in Table 6.

RESULTS AND DISCUSSION

From Table 1 we can say that the viscosity of COERs increases as the mole ratios of epoxy resin increases. Specific gravity of COERs increases as the amount of epoxy resin increases in COERs. From Table 2, it is evident that the number of hydroxyl groups in all three COERs is 3, but hydroxyl value and % hydroxyl decreases as the mole of epoxy resin increases. From the unsaturation test it is clear that the number of unsaturation per molecule in each COER is 3, as in castor oil. It means that the reaction of epoxy resin has not occurred at the double bonds of castor oil.

The IR spectra of castor oil and all three COERs were scanned neat on KBr pellets and were found to be consistent with their predicted structures. From the results given in Table 4, it suggests that these films give good surface dry and good tack-free dry properties as well as good adhesion and flexibility. Excellent scratch hardness was obtained from the films prepared from these blends; pencil hardness and impact resistance were also good. Chemical resistance tests of the films gave satisfactory results.

CONCLUSION

Room temperature curing composition can be prepared easily and give satisfactory results. Castor oil and epoxy resin reaction products upon blending with resins like alkyd and CHF resins gives good mechanical and chemical properties. These blends give good mechanical and chemical resistance properties. In conclusion, the alkyd-COERs-CHF blends give good properties.

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