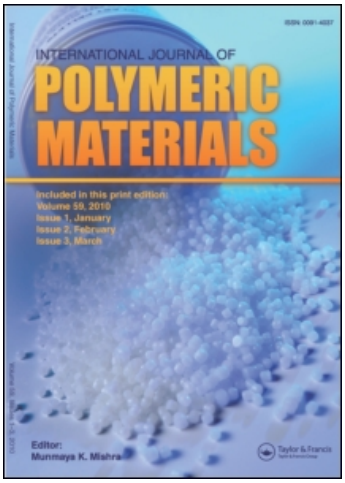

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Coating Properties of Di- and Tri-functional Epoxy Resin Using Novel Epoxy Based Polyamide as Curing Agent

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Coating Properties of Di- and Tri-functional Epoxy Resin Using Novel Epoxy Based Polyamide as Curing Agent

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Bis ester namely 1, 1'-(1-methylethylidene) bis [4-{1-(1-imino-4-ethyl benzoate)-2-propanolyloxy}]benzene was synthesized by the reaction of epoxy resin, diglycidyl ether of bisphenol-A-(DGEBA) and 4-amino ethyl benzoate (4-AEB) using triethyl amine as catalyst. The synthesized bisester was reacted with two different aliphatic diamines viz., 1, 4-butylene diamine (BDA) and 1, 6-hexamethylene diamine (HMDA) to obtain respective polyamide resins (PAs) abbreviated as DGEBA-4-AEB:BDA and DGEBA-4-AEB:HMDA respectively.

The PAs synthesized were used as a curing agent for the difunctional epoxy resin, (DGEBA) and trifunctional epoxy resin, (TGPAP) in three different ratios. Using triethylamine as a catalyst and PAs as a curing agent, DGEBA and TGPAP were polymerized on mild steel panels at 120°C for 1 hr. The coated panels thus obtained were tested for scratch hardness, flexibility, impact strength and chemical resistancy.

It appears from the results that epoxy resins, DGEBA based polyamides can successfully be used as a curing agent for the coating application.

Keywords: Polyamides; bisester; epoxy resin; scratch hardness; flexibility; impact strength; chemical resistancy

INTRODUCTION

Epoxy resin have attained a prominent position in industry. In their fully cured state, they have remarkable properties to solvents, acids, base and heat [1]. In addition, the low cost of these resin have

prompted its use as substitutes for phenolics in various important applications [2, 3]. Epoxy resin are no longer a novelty to the coating industry. Coatings based on epoxy resins are generally characterized by their excellent adhesion and overall chemical resistancy. They also exhibit a high degree of resistance to impact, abrasion and other types of physical abuse. It is therefore not surprising that epoxies have found use in applications such as plant maintenance, auto-motive primers, can and drum coatings, pipe coatings, appliance coatings, adhesives for home and industry and military and aero-space usage etc [4].

The reaction of epoxy ring with amine and alcohol is well established [5] and hence the reaction of epoxy resin with an organic compound having amino and ester group like 4-amino ethyl benzoate may yield bisester derivative, which can lead to polyamides on further condensation with diamine.

Hitherto, most of the commercial polyamides were prepared from dibasic acids and diamines. Polyamide formation through epoxy resin based bisester has not received attention inspite of well defined applications of both epoxy resin and polyamides.

Polyamide resin adducts have recently appeared on the market. These curing agents provide epoxy coatings with improved gloss and flow, better water and chemical resistance, and offer better resistance to exudation problems if the coating is applied under adverse weather conditions [6].

It has been reported that the polyamide curing agent displaces the water from the surface of the wet substrate, allowing the coating to adhere and cure to a dense, impermeable barrier [4].

The great deal of work on epoxy resin based polyamide has been carried out in our laboratory [7]. The work was mainly based on curing study of epoxy resin (DGEBA) using epoxy resin based polyamides as curing agent. The present communication discusses the polymerisation of difunctional and trifunctional epoxy resin (i. e. DGEBA and TGPAP) on the mild steel panels and presents the comparative study of coating properties.

RESULTS AND DISCUSSION

The synthesised epoxy resins DGEBA, TGPAP and DGEBA based polyamides (PAs) are soluble in common organic solvents.

Coating compositions for DGEBA and TGPAP are listed in Table I. The listed composition are coated on mild steel panels and cured thermally using triethyl amine (1 % by weight of resin) as a catalyst.

The novel epoxy based polyamides synthesized are DGEBA-4-AEB:BDA (a condensation product of diglycidyl ether of bisphenol-A-4-amino ethyl benzoate:1, 4-butylene diamine) and DGEBA-4-AEB:HMDA (a condensation product of diglycidyl ether of bis phenol-A-4-amino ethyl benzoate:1, 6-hexamethylene diamine). The curing temperatures were decided from the evaluation of dynamic scans obtained from differential scanning calorimetry (DSC) [7].

In Table I are presented the data on scratch hardness, impact strength and flexibility test of the coatings on the mild steel panels using various resin systems. As mentioned in the Table I, the four types of resin systems viz., DGEBA:A, DGEBA:B, TGPAP:A and TGPAP:B [where, A: DGEBA-4-AEB:BDA polyamide and B:DGEBA-4AEB:HMDA polyamide] in three different ratios have been prepared for the study. An attempt has been made to evaluate structure-property relations between di- and trifunctional epoxy resin to discuss various applications of the coatings. These coatings on application using brush produced adherent and flexible coatings which were baked at 120°C.

TABLE I Flexibility, impact strength and scratch hardness of the coated panels

System Resin:Curing agent	*Ratio (in wgt %)*	*Flexibility*	*Impact Strength*	*Scratch hardness (gm)*
DGEBA:A	60:40	Pass	250	1400
DGEBA:A	70:30	Pass	275	1500
DGEBA:A	80:20	Pass	300	1600
DGEBA:B	60:40	Pass	260	1400
DGEBA:B	70:30	Pass	275	1500
DGEBA:B	80:20	Pass	300	1800
TGPAP:A	60:40	Pass	300	1500
TGPAP:A	70:30	Pass	300 >	1800
TGPAP:A	80:20	Pass	300 >	1900
TGPAP:B	60:40	Pass	275	1500
TGPAP:B	70:30	Pass	300	1700
TGPAP:B	80:20	Pass	300 >	1900

DGEBA : Diglycidyl ether of bisphenol-A.
TGPAP : Triglycidyl ether of para amino phenol.
A : DGEBA-4-AEB:BDA Polyamide.
B : DGEBA-4-AEB:HMDA Polyamide.
4-AEB : 4-amino ethyl benzoate.
> : Greater than.

All samples passed flexibility test on 1/4″ and 1/8″ conical mandrel, which confirm the good flexibility of the coated film. All the panels passed stripping test for adhesion, which is expected of free hydroxyl group present in the resin system which contributed in the strong metallic bond formation with the surface of the mild steel panels. The good adhesion of the film may also be due to the possibility that polyamide characteristics of the curing agents displaces the water from the surface of the substract, allowing the film to adhere and cure to a dense impermeable barrier [4].

The results of testings furnished in Table I reflects that the trifunctional epoxy resin systems shows better scratch hardness and impact strength compared to difunctional epoxy resin systems. It is also observed that both the strength increases, with a decrease in the proportion of curing agent. This may be ascribed to the increased proportion of epoxy content which is also responsible for the improvement in the flexibility of the coated film.

The coated films are also tested for the water and solvent resistancy. In case of water resistance, coatings based on all resin systems shows no color change or blistering. The coated panels were immersed for 48 hours in water and solvent, but no cracking or loss in gloss were observed which indicates that all the system have excellent solvent and water resistancy. 2% alkali solution test for 48 hours shows that all the panels were remained unaffected. The observation reveal that on exposing the coated panels to 2% H_2So_4 for 48 hours, no substantial loss in gloss or change in appearance were observed indicating excellent adhesion as well as good resistance to acid, alkali, water and organic solvents.

EXPERIMENTAL

Materials

DGEBA was obtained from Synpol Chemicals Ltd., Ahmedabad, India having epoxy equivalent weight 190, viscosity 4–10 poise and density 1.16–1.17 gm/cm^3 at 25°C. The other chemical used 1, 4-butylene diamine, 1, 6-hexamethylene diamine, triethyl amine, tetrahydrofuran, methanol and methyl cellosolve were of laboratory reagent grade.

Synthesis of Resins

TGPAP was synthesized according to the method given in literature [8,9]. 4-amino ethylbenzoate was synthesized according to method given in literature (m.p. 88 to 90°C) [10]. The bisester derivative and two polyamides were prepared by the methods described in our earlier communication [7].

Coating on Mild Steel Panels

Coating compositions were prepared by combination of epoxy resin (DGEBA and TGPAP) and two PAs in three different ratios viz., 60:40, 70:30 and 80:20. The compositions were then thinned with methanol : methyl cellosolve mixture (1 : 3) to the required viscosity for application and were made free from coarse skin by passing through 150 μm sieve (IS:460–1960). All resin systems were then coated on the mild steel panel [confirming to deep drawing quality as per IS : 513–1960, size 150 × 1.25 mm) using flat brush confirming to IS : 384–1964. The coated mild steel panels were placed in vertical position for drying immediately, then examined after specific time intervals for the tack free test and cured thermally.

Measurements

The films were applied on mild steel panels (6″ × 4″) and mechanical properties were studied as per Indian Standard Specification [11]. The flexibility was measured using 1/4″ and 1/8″ conical mandrel. The scratch resistance and adhesion were determined as per IS methods [11]. Resistance towards water, acid, alkali and organic solvents were determined aas per standard methods described in the literature [12].

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