

Ambient-Cured Polyesteramide-Based Anticorrosive Coatings from Linseed Oil—A Sustainable Resource

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ABSTRACT: Ambient-cured polyesteramide (APEA) coating resin synthesized from dihydroxy fatty amide obtained from linseed oil, a sustainable resource, and poly(styrene-*co*-maleic anhydride), a bifunctional acid component, was found to exhibit improved physicochemical and anticorrosive properties. The structural elucidation of APEA resin has been carried out by FTIR, ¹H-NMR, and ¹³C-NMR spectroscopies. The physicochemical and chemical resistance properties were investigated by standard methods. The corrosion resistance performance was evaluated in acid,

alkali, and organic solvent. The thermal behavior was studied by TGA technique. A comparative study of these properties of APEA with reported baked polyesteramide (PEA) coatings was carried out. A remarkable improvement in the drying property of APEA was observed. The APEA coatings also showed improved physicochemical and anticorrosive properties as compared to the baked PEA coatings. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 97: 1818–1824, 2005

Key words: resin; coatings; adhesion; gloss; polyesteramide

INTRODUCTION

Fatty acids from linseed and other vegetable oils have long been used as a sustainable resource in the production of industrially important decorative and anticorrosive coating materials.^{1,2} The petrochemical-based monomers used in the manufacturing of polymers, due to their spiraling prices, high rate of depletion of petroleum stocks, as well as carcinogenicity, need to be replaced by polymers derived from sustainable and eco-friendly resources like vegetable seed oils.^{3–6}

N,N-bis-(2 hydroxyethyl) oil fatty amides have been used as monomer in the preparation of many oil-based polyesteramide (PEA) resins by reaction with dibasic acids or anhydrides.^{1,7–9} Oil-based PEA resins containing repeating ester (—COOR) and amide (—CONR) units in the polymer chain possess improved characteristics over normal alkyds in terms of hardness, ease of drying, water vapor resistance, chemical and thermal resistance, as well as durability and other physicochemical properties.^{7,10–11}

The high melting point, slight intractability, high drying temperature, and time cause difficulties in the application of PEA as coating materials. Generally, PEA coatings are obtained by baking at and above 175°C.^{12–14} Several attempts have been made to reduce

the curing temperature and time as well as to improve the other physicochemical properties of the oil-based PEA resin by the incorporation of vinyl monomers such as styrene and some drying activators such as metal octoate and naphthenate.^{1,15} The room temperature cured PEA coatings were also developed by modification through urethane linkages.¹²

Poly (styrene-*co*-maleic anhydride) (SMA) copolymer, a bifunctional acrylic alternate copolymer, is hard and brittle in nature as well as insoluble in most of the organic solvents.¹⁶ It is capable of reacting with alcohols, amines, and water to produce ester, amide, and acid, respectively.¹⁷ SMA copolymer possesses a unique combination of chemical and mechanical properties that may help in the development of fairly good anticorrosive coating materials. It has also been used as a modifier of latex paints to increase the adhesion and gloss of the coatings.¹¹ The replacement of phthalic anhydride by maleic anhydride in the case of alkyds was reported to result in the formation of alkyds of shorter stoving time with improved color and drying properties.¹⁸

Literature survey reveals that no work has been done on the synthesis of SMA copolymer-based ambient-cured polyesteramide from vegetable seed oils.^{1,7,8,12,19} We have tried in our earlier work to partially replace phthalic anhydride with SMA copolymer by the incorporation of 5 to 25 wt % of the latter into *N,N*-bis-(2 hydroxyethyl) linseed oil fatty amide (HELA) and found an appreciable decrease in the baking temperature, from 220°C (plain PEA) to 150°C poly(styrene-*co*-maleic anhydride), along with good physicochemical properties of polyesteramide coat-

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TABLE I
Physicomechanical and Anticorrosive Properties of Ambient-Cured Polyesteramide (APEA)

Resin code	Drying time (min)	Scratch hardness (kg)	Impact resistance (passes)	Gloss at 45°C	Bend test 1/8 inch	Chemical resistance				
						H ₂ O 7 days	NaOH (5%) 2h	HCl (2%) 7 days	NaCl (3.5%) 7 days	Xylene 7 days
APEA-I	25	1.5	150	65	Pass	d	d	c	c	e
APEA-II	20	2.5	150	65	Pass	e	e	d	e	e
APEA-III	18	2.0	150	61	Pass	c	b	e	e	e
APEA-IV	15	1.8	100	58	Fail	c	a	c	b	e
PEA*	—	2.0	100	50	Fail	e	a**	b	b	d

* Plain PEA baked at 220°C.²⁰

** Film completely removed in 1 h.

^a Film completely removed.

^b Loss in gloss.

^c Slight loss in gloss and adhesion.

^d Slight loss in gloss.

^e Unaffected.

ings.¹¹ Curing of the PEA at elevated temperatures is a multistep process and is also energy consuming.¹² In the present article, we have, therefore, attempted to synthesize the ambient-cured PEA by the complete replacement of phthalic anhydride by SMA copolymer. The structure of the newly developed ambient-cured polyesteramide (APEA) was confirmed by FTIR, ¹H-NMR, and ¹³C-NMR spectral analysis. The physicochemical, physicomechanical, and thermal characterization as well as anticorrosive performance of its coatings under different corrosive environment was also carried out. The APEA coatings show better performance than the reported baked plain PEA coatings.

EXPERIMENTAL

Materials

Oil was extracted from linseed (procured from local market) by using a Soxhlet apparatus. Petroleum ether (boiling range 60–80°C) was used as a solvent. The fatty acid composition of the oil was determined by gas chromatography²⁰ (GC; 111/8 S. S column, FID detector). Sodium methoxide, xylene (Merck, New Delhi, India), diethanolamine, and sulfuric acid (SD Fine Chemicals, New Delhi, India) were of analytical grade and poly(styrene-*co*-maleic anhydride) was an Aldrich (New Delhi, India) product with an average molecular weight of 1600 Da.

Synthesis of HELA

HELA was synthesized by a reported method.²⁰ According to this method, aminolysis of linseed oil (0.1 mol) with diethanol amine (0.32 mol) in the presence of sodium methoxide (0.007 mol) was carried out at 100–110°C. The completion of aminolysis reaction was confirmed by thin layer chromatography (TLC). After

the completion of the reaction, the reaction product was purified by washing successively with 15 wt % NaCl solution and diethyl ether. After evaporation of diethyl ether, pure HELA was obtained.

Synthesis of APEA

HELA and SMA copolymer were reacted in molar ratios, 1 : 0.06, 1 : 0.084, 1 : 0.11, and 1 : 0.135 assigned as APEA-I, APEA-II, APEA-III, and APEA-IV, respectively. The solution of HELA in xylene : acetone mixture (3 : 1) was taken in a four-necked conical flask fitted with a Dean–Stark trap, nitrogen inlet tube, thermometer, and mechanical stirrer. The contents were heated at 100 ± 5°C under continuous stirring. The solution of SMA copolymer in xylene : acetone mixture (3 : 1) was added dropwise in the presence of acid catalyst over a period of 30 min. After the complete addition of SMA copolymer solution, the temperature was raised to 145 ± 5°C. The progress of the reaction was monitored by acid value and TLC. The reaction was carried out until the theoretical amount of water was collected in the Dean–Stark trap and the reaction product attained the desired acid value. The reaction was stopped at this point; the contents were cooled, and xylene was removed from the resin in a rotatory vacuum evaporator under reduced pressure to obtain a highly viscous APEA resin.

Characterization

APEA was characterized by FTIR, ¹H-NMR, ¹³C-NMR, and TGA techniques. FTIR spectra of these resins were taken on a Perkin–Elmer 1750 FTIR spectrophotometer (Perkin–Elmer Instruments, Norwalk, CT) by using a NaCl cell. ¹H-NMR and ¹³C-NMR spectra were recorded on a JEOL GSX 300MHZ FX-

TABLE II
FTIR Peaks of HELA and APEA

Functional group	IR (cm ⁻¹)	
	HELA	APEA
OH	3450–3410	3407
>C=O (ester)	—	1733
C—C(=O)—O str	—	1250, 1160.07, 1080
>C=O (amide)	1635	1630.6
C—N str	1464	1450
CH ₂ _{sym}	2855.6	2855
CH ₂ _{asym}	2926.8	2926
C=C—H str	3007	3007
Ar C=C str	—	1585, 1500, 1450, 750, 702
Ar C=C—H str	—	3050

Ar, Aromatic ring.

1000 spectrometer by using deuterated chloroform and dimethyl sulfoxide (DMSO) as solvents, respectively, and tetramethylsilane (TMS) as an internal standard. The thermogravimetric analysis of APEA and plain PEA was done on thermogravimetric analyzer, TA-57 (TA Instruments, U.S.A.) at 20°C/min in nitrogen atmosphere to study the thermal stability of the resin. The inherent viscosity of APEA in *N*-methyl pyrrolidone (0.5 g/100 mL) was determined by Ubbelohde viscometer at 25°C. Iodine value (ASTM D555-6) and hydroxyl value, saponification value, specific gravity, and refractive index were determined by AOCS methods (Table III).

Preparation and testing of coatings

Coatings of APEA resin were prepared by brush technique by using 40 wt % APEA resin in xylene on commercially available mild steel strips 30 × 10 × 1 mm size for chemical resistance and 70 × 25 × 1 mm size strips to determine the specular gloss at 45° by glossmeter (model RSPT 20; Digital Instruments, Santa Barbara, CA), scratch hardness (BS3900), bending (ASTM D3281-84), and impact resistance (IS:101part5/s.3,1988). The coating thickness was measured by Elcometer (model 345; Elcometer Instrument, Manchester, UK). The thickness of these coatings were found to be 70 ± 5 μm. Corrosion tests were performed in water, acid (5 wt % HCl), alkali (5 wt % NaOH), and

xylene by placing them in 3-in.-diameter porcelain dishes and dipping the coated samples in the aforementioned media. Periodic examination was conducted until the coatings showed evidence of softening or deterioration (Table I). Salt spray test (ASTM B177-94) was also carried out for a period of 7 days in a salt mist chamber.

RESULTS AND DISCUSSION

Figure 1(a) shows the reaction scheme for synthesis of HELA from linseed oil and diethanolamine in the presence of sodium methoxide (NaOCH₃) by aminolysis method. Figure 1(b) shows the reaction scheme for the synthesis of APEA, from HELA and SMA copolymer in the presence of acid catalyst, containing both ester and amide linkages in the backbone of polymer. In our earlier studies we found that the coatings of plain PEA and SCPEA were obtained by baking at 220 and 150°C, respectively.^{11,20} The coatings of APEA resin prepared above, however, dried at room temperature. The coatings of these resins were prepared by applying on mild steel strips. The physicochemical, physicomechanical, and anticorrosive properties are provided in Tables I and II. The structure of APEA was established by FTIR, ¹H-NMR, and ¹³C-NMR spectral analysis. Thermal stability behavior of APEA was studied by TGA technique. The aforementioned properties of APEA were also compared with those of plain PEA baked coatings.

Spectral analysis

HELA

The structure of HELA was confirmed by FTIR and ¹H-NMR spectral analysis.²⁰

APEA

The analysis of IR spectra of HELA and APEA reveals the following characteristic absorption bands as given in Table II. IR spectra of HELA show bands at 2926.8 and 2855.6 cm⁻¹ because of —CH₂ asymmetrical and symmetrical stretching, respectively, for methylene groups of fatty acid chains. The same in APEA spectra

TABLE III
Physicochemical Characterization of APEA

Resin code	Hydroxyl value	Saponification value	Iodine value	Specific gravity	Refractive index	Inherent viscosity (dl/g)
APEA-I	3.28	78.0	38.09	0.9865	1.579	0.780
APEA-II	2.58	76.6	36.52	0.9872	1.581	0.785
APEA-III	2.00	75.1	34.02	0.9880	1.586	0.790
APEA-IV	1.36	74.0	32.35	0.9889	1.590	0.798
PEA	7.87	128.0	48.00	0.9380	1.507	0.652

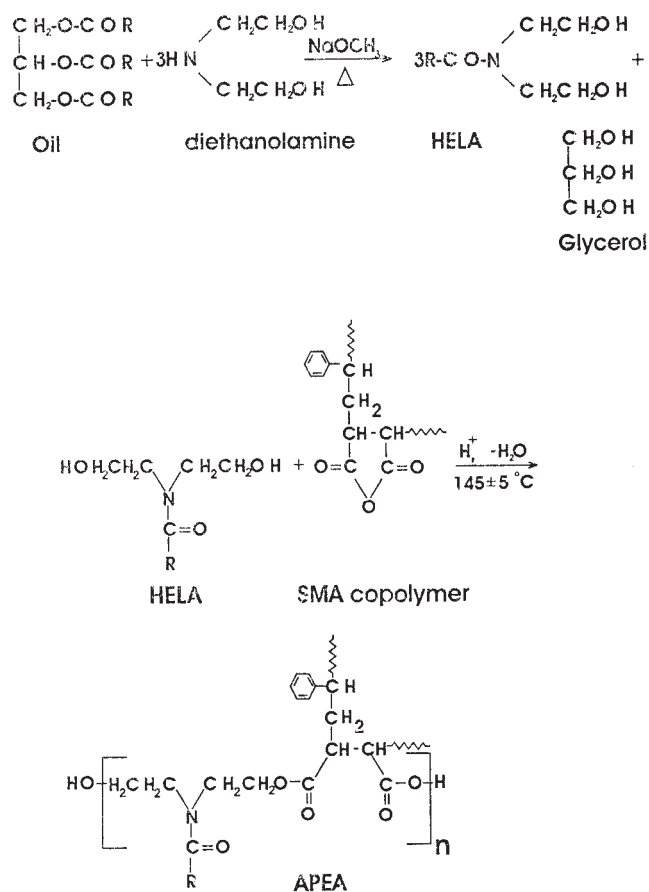


Figure 1 (a) Synthesis of HELA; (b) synthesis of APEA; R = alkyl chain of fatty acid of oil.

is observed at 2926 and 2855 cm^{-1} . The broad bands for hydroxyl groups occur at 3450–3410 and 3407 cm^{-1} , respectively, for HELA and APEA. The characteristic bands for C—N stretching and —C=O stretching of amide appear at 1464 and 1635 cm^{-1} in the spectra of HELA and at 1450 and 1630 cm^{-1} in the spectra of APEA. These absorption bands occur at lower frequencies due to the incorporation of SMA copolymer in the resin. The appearance of characteristic bands for ester moieties (Table II) confirm the esterification reaction between HELA and SMA copolymer forming APEA. The distinct bands in the aromatic region also confirm the incorporation of styrene through SMA copolymer in APEA backbone. The structure of APEA is further confirmed by $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectral analysis.

In the $^1\text{H-NMR}$ spectra of APEA (Fig. 2) peaks appearing at $\delta = 3.491$ ppm, $\delta = 3.855$ ppm, and $\delta = 2.30$ – 2.26 ppm are characteristic bands of —CH₂ attached to free hydroxyl, amide nitrogen, and amide carbonyl, respectively. The peaks in the range $\delta = 7.33$ – 7.15 ppm are characteristic of aromatic ring protons present in benzene ring of styrene; we have also observed these peaks in our earlier studies on polyesteramide due to the presence of aromatic ring protons of phthalic acid.²⁰ The $^1\text{H-NMR}$ spectra also show a band at $\delta = 4.21$ ppm for CH₂ attached to —OC=O, band at $\delta = 2.26$ – 2.24 ppm, $\delta = 1.12$ ppm for —CH, —CH₂ of styrene, and at $\delta = 2.7$ – 2.6 ppm attributed to —CH— of maleic anhydride.

The $^{13}\text{C-NMR}$ spectra (Fig. 3) shows the peaks at $\delta = 59.376$ ppm, $\delta = 47$ – 40 ppm, and $\delta = 33.69$ ppm for

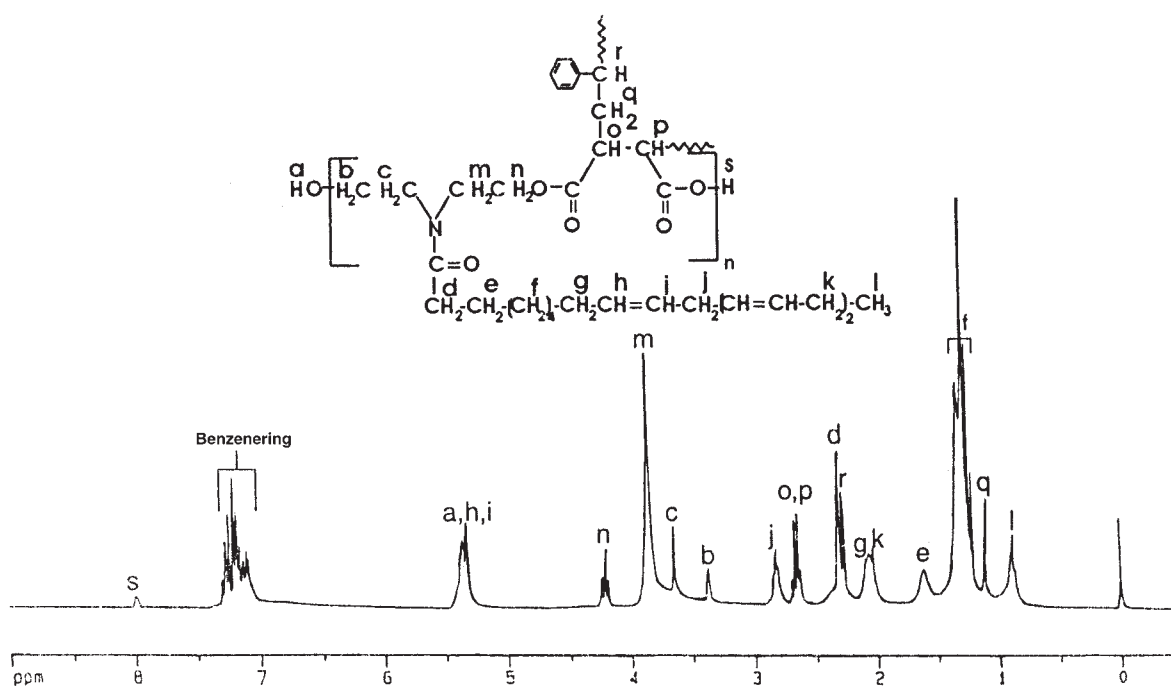


Figure 2 $^1\text{H-NMR}$ spectra of APEA.

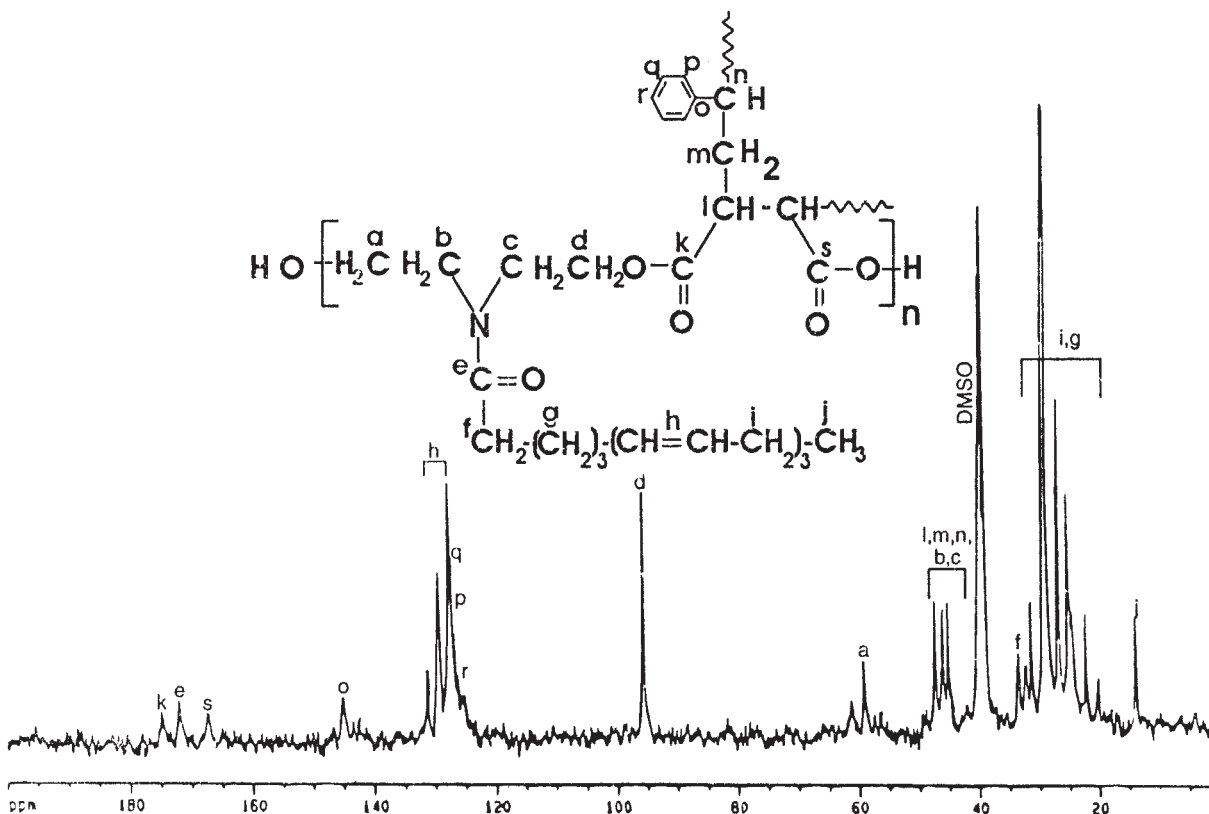


Figure 3 ^{13}C -NMR spectra of APEA.

$-\text{CH}_2$ attached to free hydroxyl, amide nitrogen, and amide carbonyl, respectively. The peaks at $\delta = 32\text{--}20$ ppm are attributed to the $-\text{CH}_2$ of fatty acid chains, whereas peaks for olefinic carbons ($-\text{C}=\text{C}-$) appear at $\delta = 130\text{--}127.7$ ppm. The characteristic peak for quaternary carbon of styrene appears at $\delta = 144.5$ ppm, while other peaks for styrene ring appear at $\delta = 127.6$ ppm, $\delta = 126.66$ ppm, and $\delta = 125.8$ ppm. The peaks for $-\text{CH}_2$, $-\text{CH}-$ of the copolymer appear at $\delta = 47\text{--}40$ ppm; the values of these peaks overlap with those of the peaks of $-\text{CH}_2$ attached to amide nitrogen. A sharp peak of higher intensity appears at $\delta = 96$ ppm, characteristic of $-\text{CH}_2$ attached to $-\text{OC}=\text{O}$ of the maleic anhydride group of the copolymer. These correlations derived through spectral studies confirm the structure of APEA.^{11,21-22}

Physicochemical characterization

The physicochemical characterization data of the APEA resins are given in Table III. It is observed that as the proportion of SMA copolymer increases in the APEA, the hydroxyl value, saponification value, and iodine value decrease. This can be attributed to the decrease in the relative proportion of unsaturation and progressive increase in the molar mass of APEA. The increase in molar mass of the resin is also ob-

served through the progressive increase in the value of specific gravity, refractive index, and inherent viscosity of the resin as more and more copolymer is incorporated in the resin. Incorporation of SMA copolymer in the resin at APEA-IV results in a resin of very high viscosity, which finally undergoes gelation within 6-7 days.

The solubility of the resin was tested in different polar and nonpolar solvents such as DMSO, dimethylformamide (DMF), ethanol, methanol, chloroform, ether, acetone, toluene, xylene, hexene, petroleum ether, and benzene. The resin shows good solubility, 80-100 wt %, in DMSO, DMF, chloroform, ether, acetone, toluene, and xylene and, in the rest of the solvents, the solubility varies from 20 to 40 wt %. Thus, the resin is soluble in either polar aprotic solvents or aromatic hydrocarbons due to the presence of polar groups and benzene rings in the backbone of the resin.

Coating properties

The coatings of APEA of different compositions were applied on mild steel strips of standard size to evaluate their physicochemical and anticorrosive performance as summarized in Table I. The aforementioned properties of APEA were compared with those of the plain PEA. The effect of copolymer loading on the

properties of APEA among themselves was also investigated. Table I reveals a considerable improvement in the drying properties of APEA among themselves and remarkably over plain PEA. The conventional PEA resin, developed by the reaction of fatty amide with phthalic acid and other dicarboxylic acids or anhydrides, was reported to take shorter times in curing at elevated temperatures and fairly longer times for curing at room temperature.^{1,7-9} The new SMA copolymer-based polyesteramide resins exhibit remarkable improvement in the drying property of the resin as well as the physicochemical and anticorrosive properties. The coatings of APEA become dry within 15–25 min. With the incorporation of styrene, drying times of the order of a few minutes have been obtained, the phenomenon being purely physical.²³⁻²⁴ Maleic anhydride also facilitates drying ability.²⁵⁻²⁶ Thus, the air-drying property of APEA is attributed to the maleic anhydride and styrene content of the copolymer. The decrease in drying time with the increase in the molar ratio of SMA copolymer in the resin is attributed to the simultaneous increase in styrene and maleic anhydride moieties in APEA polymeric resins, the styrene content, the latter two conferring rapid drying.²³⁻²⁶ APEA-II exhibits a higher degree of gloss, scratch hardness, and impact resistance as compared to APEA-I, APEA-III, APEA-IV, and baked plain PEA coatings. This effect can be explained considering that with a particular amount of loading of copolymer (APEA-II), the polymer attains a reasonably larger chain length, along with desired crosslink density, resulting in a fine and dense structure that would result in the enhancement of these properties. However, among themselves, the APEA resins show a decrease in gloss with the gradual increase in the copolymer content. It is reported that the gloss of air-dried styrenated alkyds is rather poor.²³ Here, the higher gloss value was observed predominantly on account of the high-gloss characteristic of bifunctional acrylic maleic anhydride content and fatty acid chains.²⁷ The decrease in gloss with higher copolymer loading can be attributed only to the polystyrene content. The coatings up to APEA-III passed the 1/8-in. conical Mandrel bend test, which indicates high flexibility of the films. Beyond this loading, coatings failed the 1/8-in. bend test, but their bending ability is comparable to that of baked PEA coatings (1/4-in. bend test). The decrease in bend test properties can be attributed to a disproportionate increase in aromatic moieties and a higher crosslink density that hampers the free rotation of polymer chains, causing a decrease in the values of the bend test. It is observed that with the increased loading of copolymer beyond its optimum loading in APEA resin, the physicochemical properties deteriorate, which may be attributed to an increase in the molar mass of APEA resin to such an extent that it develops an internal strain within the

polymer. The latter produces deterioration in physicochemical properties of the coatings. In general, APEA coatings have superior corrosion resistance properties than PEA coatings. The anticorrosive properties like physicochemical properties of APEA-II are far superior to the other APEA and baked PEA coatings (Table I). The APEA-II coatings are unaffected in water, xylene, and NaCl (3.5 wt %) for 7 days, in NaOH (5 wt %) for 2 h, and in HCl (2 wt %) it loses its gloss slightly after 7 days. The baked PEA coatings get completely removed in 5 wt % NaOH within 1 h.²⁰ The high corrosion resistance of APEA-II coatings can be attributed to the presence of an optimum amount of styrene moiety in the resin and the required degree of crosslink density, which helps in producing a most uniform and well-adhered coating on the surface of the substrate. Literature survey reveals that the higher the styrene content, the worse the resistance to solvents, in particular, the aromatic solvents.²³ However, we found that in the case of APEA-I to APEA-IV the higher styrene content through the increased copolymer loading has not affected the resistance to xylene, an aromatic solvent. Presumably, the high copolymer content leads to a higher crosslink density; the coating material is converted to an insoluble, infusible thermoset,²⁵ which does not allow the aromatic solvents or other corrosives to penetrate.

As discussed above in APEA resins, the drying is fast and occurs at room temperature because of the presence of copolymer. The coatings obtained from APEA-IV initially show a smooth uniform structure but after a period of 6–7 days cracks appear on the surface. It appears that initially the simple esterification reaction between HELA and SMA copolymer predominates followed by auto-oxidation through unsaturation present in fatty acid chains. It gradually results in the high degree of crosslinking across the neighboring chains. This type of molecular structure is liable to be distorted by too much crosslinking through polyesterification and auto-oxidation causing gelation of the resin as mentioned earlier. These two factors along with the rigidity introduced due to high SMA copolymer content, beyond the optimum loading, produce highly strained film, responsible for the development of cracks on the surface.

Thermal analysis

The TGA thermogram (Fig. 4) shows an initial sluggish degradation rate in the temperature range of 200–290°C. Beyond this, a faster rate of degradation occurs, which extends up to 490°C, apparently as a single event. A 10 wt % loss is observed at 280°C and a 50 wt % loss is observed at 382°C. The TGA thermogram of the plain PEA shows 10 wt % loss at 275°C and 50 wt % loss at 370°C. The TGA thermogram also shows a decomposition event in APEA between 300

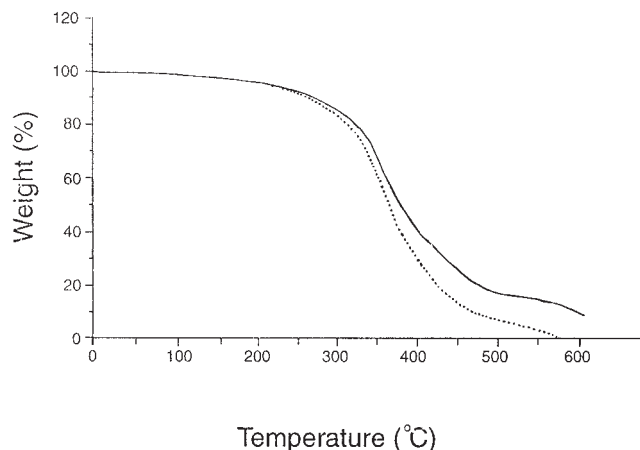


Figure 4 TGA of PEA (· · ·) and APEA (—).

and 500°C and 250 and 450°C in plain PEA. The comparative study highlights the higher thermal stability of APEA than plain PEA because of the incorporation of SMA copolymer; the presence of polystyrene moiety is imparting higher thermal stability^{17,28–29} to the APEA resin.

CONCLUSIONS

The newly developed SMA copolymer-based polyesteramide resin exhibits remarkable improvement in the drying property of the resin, whereas their physico-mechanical and anticorrosive properties are better than the plain PEA resin. The earlier workers have reported the introduction of air-drying properties through the modification of the resin; the present work reports the air-drying ability as an inherent tendency of the polyesteramide resin itself. The APEA-II shows excellent acid, NaCl, and xylene resistance, and good alkali resistance. The thermal studies confirm improved thermal stability of APEA-II that can be safely used up to 280°C. Thus, the incorporation of SMA copolymer has a cumulative effect on the physico-mechanical and anticorrosive properties of the resin up to APEA-II, which also facilitates the air drying through a simple and shorter route. Thus, the new APEA-II resin may prove to be a candidate of commercial importance in the world of anticorrosive surface coatings.

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