

ORIGINAL ARTICLES

Synthesis and Characterization of New Modified Anti-corrosive Polyesteramide Resins Incorporated N-phthaloylglutamic Acid (NPGA) for Surface Coating

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ABSTRACT

Polymeric systems based on polyesteramides (PEA) are high performance material, which combine the useful properties of polyester and polyamide resins, so new modified polyesteramide compositions were prepared and evaluated as vehicles for surface coating. The polyesteramide resins were obtained by means of a condensation polymerization reaction between phthalic anhydride (PA) and N, N- bis - (2-hydroxyethyl) linseed oil fatty acid amide (HELA) as the ingredient source of the polyol used. and phthalic anhydride (PA) which was partially replaced with N-phthaloylglutamic acid (NPGA) as the ingredient source of the dibasic acid. The structure of the resin was confirmed by FT-IR spectral studies. The coatings of 50 +/- 5 µm thickness were applied to the surface of glass panels and mild steel strips by means of a brush. The coating performance of the resins was evaluated using international standard test methods and involved the measurement of physico- mechanical properties and chemical resistance. The results show that the modification enhances both physico- mechanical and chemical properties. Also the resins were incorporated within primer formulations and evaluated as anti-corrosive single coatings. The results illustrate that the introduction of N-phthaloylglutamic acid, within the resin structure, improved the film performance and enhances the corrosion resistance performance of polyesteramide resins.

Key words: Polyesteramide; N-phthaloylglutamic acid; corrosion inhibition; anti corrosive coating; surface coating.

Introduction

Polyesteramide resins (PEA) combine the advantageous properties of both polyester and polyamide resins, such as high melting temperature, fast crystallization, good mechanical properties, good solvent resistance and low water absorption (C. Bori, E. Sorta, L. Zotteri ., 1975; P. F. van Hutten, R. M. Magnus, R. J. Gaymans., 1993). The presence of repeating units of ester (-COOR) and amide (-NCOR) in the polymeric chain of polyesteramide improves the ease of application, thermal stability, chemical and water resistance, and also contributes to faster drying and enhanced hardness compared to normal alkyds (L.E.Gast *et al* 1966-1969; J. Economy, 1984). Several polyesteramides have been synthesized from conventional and non-conventional seed oils to improve their drying ability and mechanical and corrosion protective efficiency (S. S. Mahapatra, N. Karak ,2004; P.G. Shende, 2002-2003; S. Mistry, D. Agarwal 2009). The modification results in the formation of N, N-bis (2-hydroxyethyl) fatty acid amide (HEFA) monomer, which plays a vital role in the synthesis of organic polymers and in addition also finds application as a polymer cross- linker. Sharif Ahmad *et al.* (1999-2009) have successfully used it as a starting material for the development of polyetheramides, polyesteramides, polyesteramide urethanes and polyamide urethanes, which find application as protective coating materials and can be used as an effective antibacterial and biologically safe corrosion protective material. New modified polyesteramide compositions were prepared and evaluated as anticorrosive varnish. The resin prepared by partial replacement of hydroxy ethyl fatty acid amide (HEFA) by polyethylene glycol (PEG) without affecting the resin constants. Primer formulations based on this resin showed good corrosion inhibiting properties.(Salem Aqeel, *et al* 2010), Also new modified polyesteramide compositions were prepared and evaluated as vehicles

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for surface coating. This modification, as expected, improved the film performance and durability and lead to substantial antimicrobial growth control (H. Abd El-Wahabb 2008, M. Mostafa., A.M. Naser., F. Abdel-Hai and, H. Abd El-Wahab 2009; Ibrahim Sabbah 2010). Blends of poly (vinyl chloride) (PVC) and polyesteramides were prepared to improve the notch impact strength of PVC (R. Kalouskova, *et al* 2009). The aim of the present work was to characterize and evaluate new modified anti-corrosive polyesteramide resins for use in protective coating formulations. These modified resins were prepared by partial replacement of N, N- bis - (2-hydroxyethyl) linseed oil fatty acid amide (HELA) with 7- (2,3-dihydroxypropyl) NPGA as a new source of the polyol source.

2. Experimental:

2.1. Materials:

The linseed oil fatty acid (LOFA) used was supplied by Echantillon, Belgium, the phthalic anhydride (PA) by Avondate Laboratories, England and the diethanolamine (DEA) and N-phthaloylglutamic acid were obtained from Sdfine Indian,. sulphuric acid, anhydrous sodium carbonate, benzene, glacial acetic acid and ethyl acetate were obtained from El Nasr Pharmaceutical Company, Egypt. The xylene and mineral turpentine products were obtained from the Misr petroleum Company, Egypt.

2.2. Methods and Techniques:

2.2.1. Synthesis of N, N-bis (2-hydroxyethyl) Linseed Oil Fatty Acid Amide (HELA):

A mixture of freshly distilled diethanolamine (DEA) (11.55g, 0.11mol) and linseed oil fatty acid (28.0 g, 0.1mol) was added to a 250 ml round-bottom flask fitted with a Dean and Stark trap. The mixture was allowed to reflux in the presence of 15 % xylene until the theoretical amount of water (1.8ml, 0.1mol) was collected, indicating the complete formation of N,N-bis (2-hydroxyethyl) linseed oil fatty acid amide (HELA).

2.2.2 Synthesis of N- phthaloyl glutamic acid.

A mixture of L-glutamic acid and phthalic anhydride was heating at 180c-185c is the optically active phthalyl-L-glutamic acid obtained

2.2.3. Synthesis of Polyesteramide and New Modified Anti-corrosive Polyesteramide Resins:

Polyesteramide resins were prepared through a condensation polymerization reaction via a solvent process, in a one step reaction. A mixture of a calculated amount of hydroxy ethyl linseed oil fatty acid amide (HELA), and phthalic anhydride (PA) replaced partially with *n-phthaloylglutamic acid* as the ingredient source of the dibasic acid ingredient , was added to a 250 ml round-bottom flask fitted with a Dean and Stark trap and refluxed in the presence of 10 % xylene. The course of esterification was followed by observing the theoretical amount of water liberated. The resins were prepared, covering a wide range of oil lengths and hydroxyl content (0, 10, 20 and 30 % excess-OH). It should be noted that within each set of formulations the total number of acid and hydroxyl equivalent for the various runs were kept constant (T.C. Patton 1962). Resin characteristic constant and weight changes of the various formulations are illustrated in Tables 1 and 2. Alkyd calculations can be used to predict the completed formulation, properties of the resin, the amount of reaction water liberated and the probable risk of gelation. The calculation of water evolved is also useful both as a tool for following the course of esterification reaction and to find the theoretical yield.

Take in table 1,2.

2. 3. Measurements:

The structure of the prepared *N-phthaloyl glutamic acid* was confirmed by the melting point,. The structure of the prepared N, N-bis (2-hydroxyethyl) linseed oil fatty acid amide (HELA) was confirmed by acid value (ASTM D 1639-96) and hydroxyl value (ASTM D 1957- 01). Infrared spectra of the prepared polyesteramide resin was recorded in polymer/ KBr pellets using Shimadzu FT-IR 400 spectrometer was also undertaken for confirmation of the polyesteramide resin structure as showed in figure 1 and tabulated in table 3. The color (ASTM D 1544-04) of the prepared polyesteramide and the new modified anti-corrosive polyesteramides resins was undertaken using the 'Gardner 1953 standard colour', whereas the viscosity (ASTM D4287-00(2010)) was established by the use of Brookfield cone plate Viscometer.

Table 1: Resin constants for NPGA -modified polyesteramides.

Resin No.	Excess -OH %	Ingredients	E	F	e_0	e_A	e_B	R	K	H ₂ O off ml
I _{a-d}	0	HELA	184	2	0.260	--	0.26	1.00	1.00	2.3
		NPGA	127	2	0.078	0.078	--			
		PA	74.1	2	0.181	0.181	--			
II _{a-d}	10	HELA	184	2	0.300	--	0.3	1.10	1.05	2.5
		NPGA	127	2	0.082	0.082	--			
		PA	74.1	2	0.191	0.191	--			
III _{a-d}	20	HELA	184	2	0.358	--	0.358	1.20	1.10	2.7
		NPGA	127	2	0.089	0.089	--			
		PA	74.1	2	0.208	0.208	--			
IV _{a-d}	30	HELA	184	2	0.44	--	0.44	1.30	1.15	3.1
		NPGA	127	2	0.10	0.10	--			
		PA	74.1	2	0.239	0.239	--			

HELA: Hydroxy Ethyl Linseed Amide PA: Phthalic Anhydride NPGA: *N-phthaloyl glutamic acid* E: Equivalent Weight
 e_A : Number of acid equivalent e_B : Number of hydroxyl equivalent
 e_0 : total equivalent present at the start of the reaction F : Functionality K: Alkyl constant (m_0 / e_A) R: Ratio of total-OH groups to total-COOH groups (e_B / e_A)

Table 2: List of hydroxyl equivalent of different runs.

Resin	HELA	NPGA
a	1.00	0.00
b	0.90	0.10
c	0.80	0.20
d	0.70	0.30

2.4. Preparation of Polyesteramide Coatings:

The solid content (ASTM D1644-01) of the modified and unmodified polyesteramide resins were adjusted by means of thinning with mineral turpentine to 50 % solids for the 0, 10, 20 and 30 % excess – OH samples. The resin samples were then filtered and the driers combination added (Co, Zr and Ca Octoates, 0.04, 1.0 and 0.05 % based on metal / solid resin, respectively). The resin material was then applied onto both glass panels (ASTM D3891-02) and steel strips (ASTM D609 - 00) at room temperature by means of a brush.

2.5. Film Casting and Testing:

Glass plates (100 x 150 mm) coated with the individual resins were utilized to measure the drying schedule, in terms of time of set-to-touch and dry - through times (ASTM D1640-03). In addition, the same coated glass plates were also used to measure the degree of gloss for individual resin coatings (ASTM 523-08). The measurements were undertaken using a gloss meter set at a 20° angle. The chemical resistance (ASTM D 870-02, D 2792-04) of the dried resin coatings to different solvent media was undertaken on glass panels (25 x 75 mm). The coated steel strips were utilized to measure the coating film thickness (ASTM D 1005- 07), with this being undertaken using a Model 2100 Minitest microprocessor coating thickness gauge, manufactured by Electro – Physic, West Germany. The thickness of the coatings was found to be 50 +/- 5µm. The coated steel strips were also used also to measure the mechanical resistance of the coated resin films, with testing undertaken on adhesion strength, by means of the cross-cut method (ASTM D 3359-02), flexibility (ASTM D 522-93), scratch hardness (ASTM D5178-98(2008)) and resistance to mechanical damage (impact resistance) (ASTM D 2794-04).

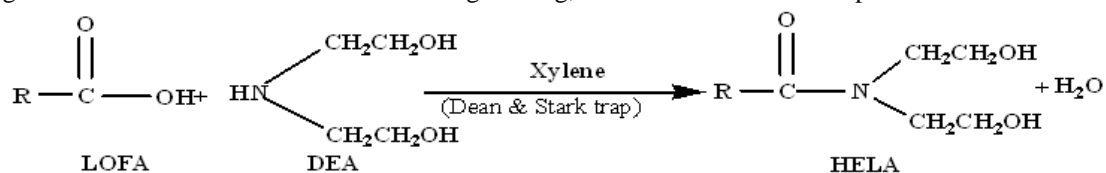
2.6. Anti-corrosive Performance Evaluation of Primer Formulations Through Accelerated Testing:

A salt fog chamber was utilized for each of the resin formulations; with a set of three coated panels being placed in the salt spray chamber according to ASTM B117-03. The corrosion resistance was evaluated in terms of blistering, scribe failure and degree of rusting, in relation to ASTM standards, i.e. ASTM D714-02, D1654-00 and D610-01 respectively.

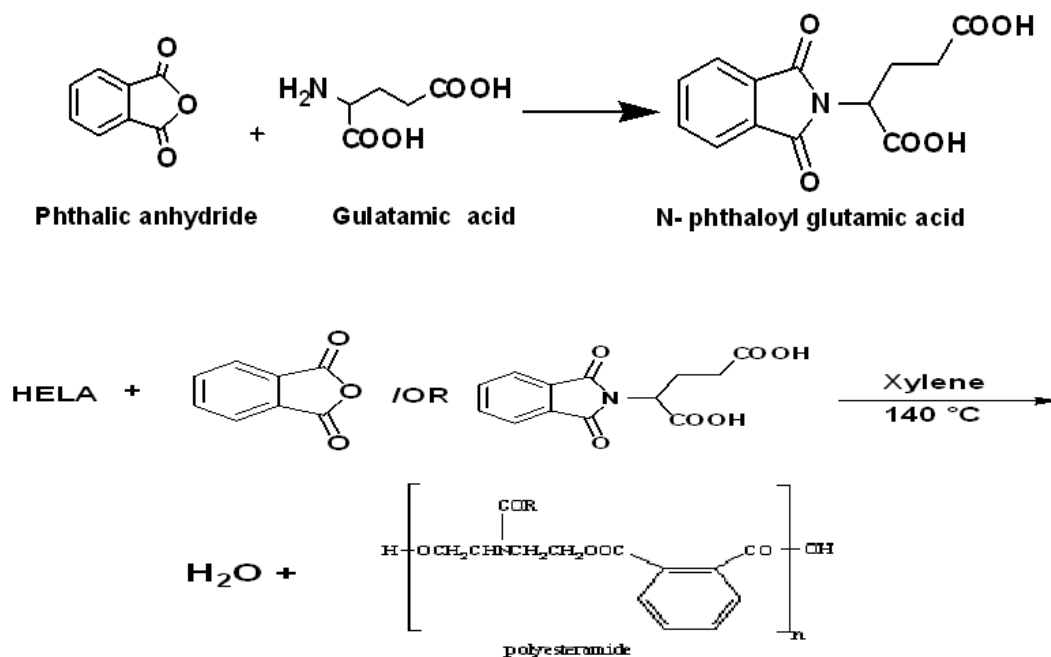
Results and Discussion

In recent years there has been considerable interest in improving polyesteramide resins. A number of polyesteramide have been developed by the reaction of linseed oil fatty amide with dicarboxylic acids/anhydrides (H. Abd El-Wahabb 2011). In the present work, the newly modified anti-corrosive polyesteramides resins were obtained by partial replacement of phthalic anhydride with *n-phthaloylglutamic acid* as the ingredient source of the polybasic acid. The reaction of LOFA with DEA to give HELA is

demonstrated in Scheme 1, which was confirmed by acid and hydroxyl value. The measured acid value was 6 mg KOH/g, whilst the theoretical value is 0 mg KOH/g, and the measured hydroxyl value was 308 mg KOH/g whilst the theoretical value is 305.72 mg KOH/g, which indicates the complete formation of HELA.



Scheme 1: Synthesis of N, N-bis (2-hydroxyethyl) linseed oil fatty acid amide (HELA).



3.1. Spectral Analysis of Polyesteramide Resin:

Polyesteramide resins were prepared by the reaction of HELA or *N-phthaloyl glutamic acid* with phthalic anhydride (PA) in the presence of xylene, as illustrated in Scheme 1. It contains both ester and amide linkages in the polymer backbone. This has been confirmed by both FT-IR spectral analyses, as shown in Figure 1. The key peaks of the IR spectra of polyesteramide are shown in Table 3, which indicates the presence of both ester and amide groups, confirming the formation of polyesteramide.

Table 3: FTIR spectra of polyesteramide resin.

Functional group	IR peak (cm ⁻¹)
OH	3398
CON amide carbonyl	1648
COO ester carbonyl	1725
C-N	1424
CH ₂ symmetric	2930
CH ₂ a symmetric	2852
Ring stretching vibration of aromatic nuclei	713

3.2. Physical-mechanical Characterization of the New Modified Anti-corrosive Polyesteramide Resin:

During the course of the preparation of the resin, the amount of collected water present in the trap was observed against the reaction time these represented in figure 2. The effect of the *N-phthaloyl glutamic acid* on the physical properties of the resin i.e. colour, viscosity, air drying time, are shown in Table 4. The effect on the mechanical properties such as gloss, flexibility (bend), adhesion, scratch hardness and resistance to mechanical damage (impact resistance) are represented in Table 5 and figure 4. The results tabulated in Table 5 show that the polyesteramide, and the new modified anti-corrosive polyesteramide compositions, give a very clear transparent and homogenous appearance. The figure also shows that the reaction time is marginally

increased with an increase in the amount of *N-phthaloylglutamic acid*, but excess hydroxyl content leads to a decrease in the reaction time. The colour of resins are observed to be dark brown due to the presence of nitrogenous base and the viscosity of resins decrease with an increase in the percentage of both *n-phthaloylglutamic acid* NPGA and excess hydroxyl content. The drying time is observed to be proportional with both *n-phthaloylglutamic acid* and excess hydroxyl content. The mechanical characteristics of the various modified resins tabulated in Table 5. The results indicate that the modified films show outstanding performance, compared to unmodified films, The gloss results for the resins increases through modification, which may be attributed to the incorporation of dihydroxyl polyol containing a NPGA ring within the resin structure. The presence of the repeating units of ester (-COOR) in the polymeric chain of polyesteramide improves the ease of application, as well as enhancing the hardness and impact resistance properties as showed in figure 4. The flexibility test for the coated panel, carried out at the mandrel diameter of 6mm, show no evidence of cracks or de-laminating of the coating film. This high flexibility of the coating is attributed to the resin being composed of a polyester backbone. The cross-hatch test, carried out to determine the adhesion of the coating, clearly indicates that the modified coating has very good adhesion properties. No de-laminating or detachment, of coatings at the edges, and within the square lattice, was observed. It is clear that the ability of the coating on the pre- treated mild steel substrate, in terms of preventing water ingress, is good, which is one of the contributing factors for the improved adhesion of the coating to the substrate. Improvement of the mechanical properties appears to be due to the combination of amide and imide groups, which results in poly (amide), containing a NPGA ring within the resin structure.

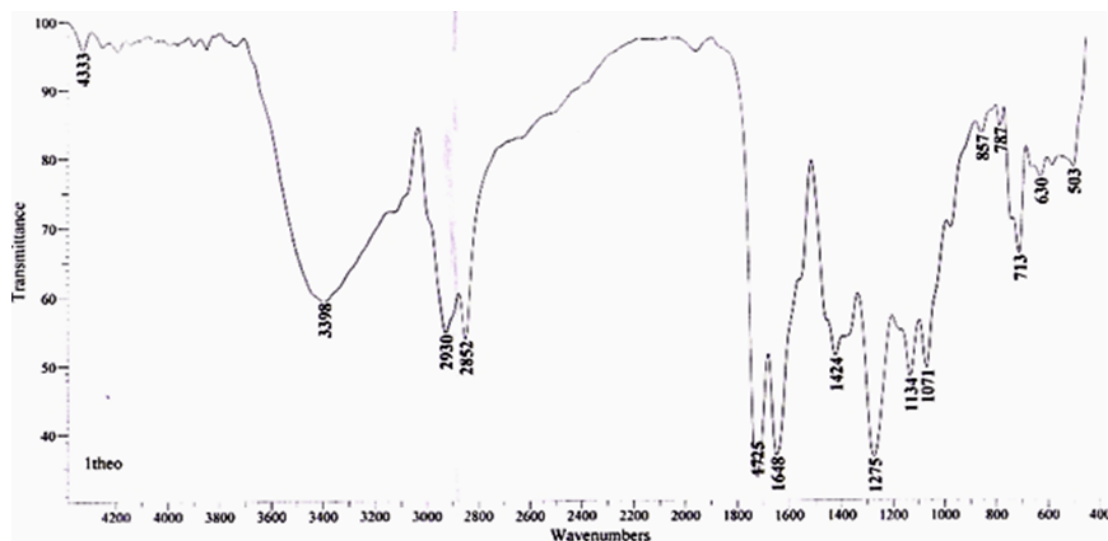


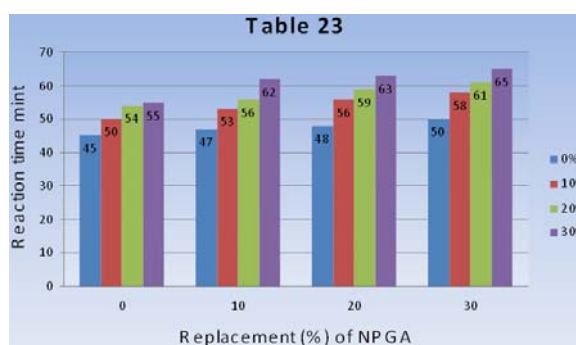
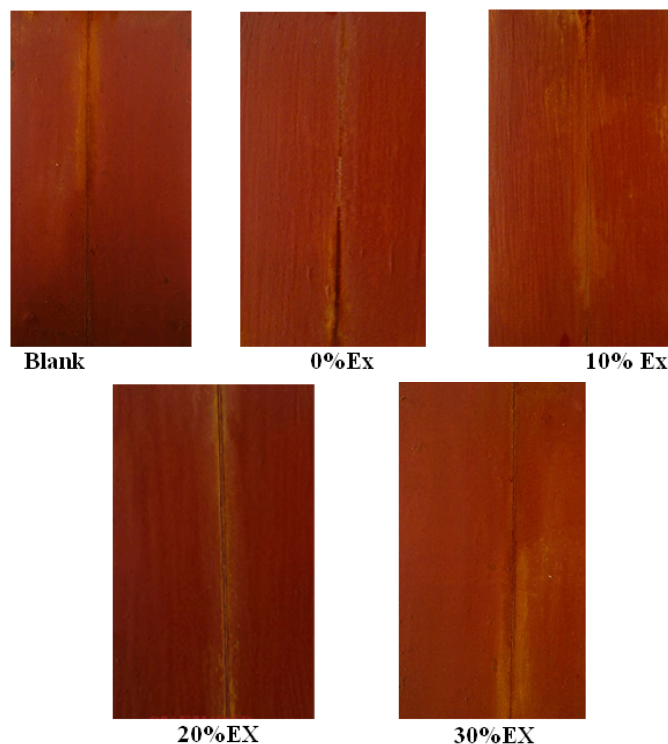
Fig. 1: I.R Spectrum of [NPGA] Modified Polyesteramide Resin.

Table 4: Varnishes characteristics data.

Resin No.	Excess-OH (%)	Replacement (%) of NPGA	Viscosity (centipoises)	Air Drying (HD) Time (hr)	stove				Color (Gardner)
					110°C		120°C		
					1hr	2hr	1hr	2hr	
Ia	0	0	23	6	T	ST	VST	HD	8
Ib		10	13.4	4.45	T	HD	--	--	15
Ic		20	13.6	4.30	ST	VST	--	--	16
Id		30	13.7	4.30	T	HD	--	--	17
IIa	10	0	23.5	5	T	ST	VST	HD	8
IIb		10	14	4.30	ST.	VST	HD	--	16
IIc		20	14.5	4.15	ST	HD	--	--	16
IId		30	14.7	4	ST	HD	--	--	17
IIIa	20	0	24	5	T	VST	HD	--	9
IIIb		10	14.5	4	ST.	VST	HD	--	17
IIIc		20	14.8	4.20	ST	HD	--	--	17
IIId		30	15	4.15	ST	HD	--	--	18
IVa	30	0	24.5	4.30	T	ST	HD	--	10
IVb		10	15.3	3.45	ST	HD	--	--	18
IVc		20	15.6	3.30	ST	HD	--	--	>18
IVd		30	15.8	3.30	ST	HD	--	--	>18

Table 5: Effect of NPGA on mechanical and chemical properties of cured modified polyesteramide.

Table 5: Effect of ACR on mechanical and chemical properties of cured epoxy resin-polyesteramine																	
Resin No	Gloss at 20 o		Flexibility		Adhesion		Scratch hardness 1Kg		Impact (J)	Water Resistance Per (day)	Alkali ** Resistance Per (day)	Acid * Resistance Per (day)	Solvent Resistance Per (day)				
	a	s	a	s	a	s	a	s	P	a	s	a	s	a	s		
Ia	90	91	Pass	Pass	5B	3B	Pass	Pass	1.2	18	20	2	3	14	17	24	26
Ib	92	92	Pass	Pass	4B	4B	Pass	Pass	1.2	18	22	4	4	19	20	27	28
Ic	92	92	Pass	Pass	4B	4B	Pass	Pass	1.3	22	25	4	5	21	22	27	28
Id	93	94	Pass	Pass	5B	5B	Pass	Pass	1.4	22	26	5	6	20	22	30	30
IIa	93	94	Pass	Pass	4B	5B	Pass	Pass	1.2	21	22	3	4	17	18	25	18
IIb	95	95	Pass	Pass	4B	5B	Pass	Pass	1.3	23	24	4	5	21	19	26	19
IIc	94	96	Pass	Pass	5B	5B	Pass	Pass	1.3	23	24	5	6	20	21	27	21
IId	96	97	Pass	Pass	5B	5B	Pass	Pass	1.4	24	26	6	6	24	23	29	30
IIIa	94	95	Pass	Pass	4B	5B	Pass	Pass	1.2	21	22	3	4	18	20	28	28
IIIf	95	95	Pass	Pass	4B	5B	Pass	Pass	1.4	25	28	5	6	21	22	27	28
IIIfc	95	96	Pass	Pass	5B	5B	Pass	Pass	1.4	25	27	6	6	22	23	28	30
IIId	95	96	Pass	Pass	5B	5B	Pass	Pass	1.4	26	27	5	6	23	20	30	30
Iva	96	95	Pass	Pass	4B	5B	Pass	Pass	1.2	23	24	4	5	20	23	27	28
IVb	95	96	Pass	Pass	5B	5B	Pass	Pass	1.3	26	29	6	7	21	22	28	30
IVc	96	97	Pass	Pass	5B	5B	Pass	Pass	1.4	28	29	7	8	24	23	30	30
IVd	96	97	Pass	Pass	5B	5B	Pass	Pass	1.4	28	29	8	9	24	25	30	30

*Acid concentration (H_2SO_4) 10% by weight**Alkali concentration (Na_2CO_3) 5% by weight**Fig. 2:** Effect on the course of esterification of NPGA -Modified Polyesteramide Resins.**Fig. 3:** Corrosion resistance of painted films.

3.3. Chemical Resistance of the New Modified Anti-corrosive Polyesteramide Resin:

The chemical resistances testing of the new modified anti-corrosive polyesteramide resins were undertaken on glass panels (25 x75 mm). The resin coated glass panels were sealed using paraffin wax on the edges of the panels. The samples were immersed to half their length in the various test solutions (water, 5% by weight anhydrous sodium carbonate, 10% by weight sulphuric acid, and benzene / mineral turpentine solvent mixture (1:3 by volume). The panels were then removed from the solution, wiped carefully and allowed to dry at room temperature, prior to testing for any change. The obtained data is tabulated in Table 5. The data indicates that an increase the levels of *N-phthaloyl glutamic acid* lead to a substantial improvement in the films resistance towards water, alkali and acid. This may well be attributable to the introduction of the NPGA ring within the resin structure. However, the *N-phthaloyl glutamic acid* does not appear to affect the solvent resistance properties, since all the examined films showed excellent performance within the experimental time of 30 days. In addition, it was observed that the stove dried films showed enhanced characteristics compared to the air dried films.

3.4. Corrosion Resistance of the New Modified Anti-corrosive Polyesteramide Resin:

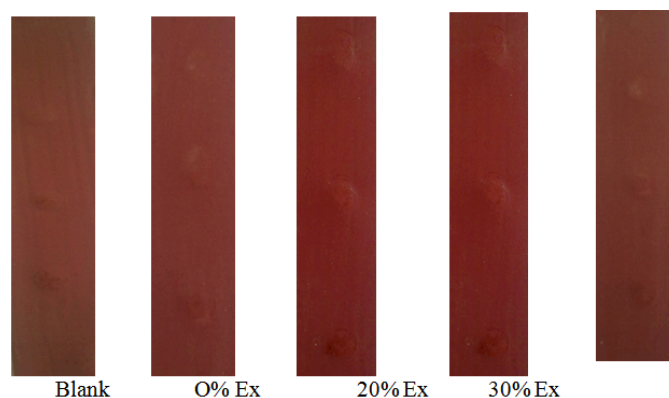
The research work was extended to study the utilization of the new modified anti-corrosive polyesteramides as a binder in primer formulations. Primer formulations based on unmodified and modified polyesteramide were prepared, with the various formulations represented in Table 6. The primer formulations were evaluated on coated steel panels. To ensure that the steel panels were free of all surface contamination before the coating application, pre-treatment involved wire brushing and sand paper cleaning of the surface. The coated steel panels were exposed to salt spray (fog) corrosion cabinet manufactured by Sheen Instruments Limited, England, for 500 hours. Photographic reference standards were used to evaluate the degree of blistering. The reference standards highlight the various degrees of blistering that can potentially develop when paint systems are subjected to these specific test conditions. The blistering size is graded from 10 to 0, where 10 represents of no blistering and 0 representative of the largest blister. Blistering frequency is denoted by F, M, MD and D (few, medium, medium dense and dense). Painted, or coated, specimens subjected to a corrosive environment are also evaluated by recording the average (mean) maximum and minimum creepage from the scribe mark. Scribe failure is also rated on a scale from 10 to 0, with 10 being zero (mm) from the scribe mark and 0 is 16 (mm) from the scribe mark. Finally visual comparison of the surface with photographic reference standards, to determine the percentage of the area that has been rusted, is also used. These visual standards were developed in cooperation with the Steel Structure Painting Council (SSPC) to further standardization of test methods. The amount of rusting beneath, or through, a paint film is a significant factor in determining whether a coating system should be repaired, or replaced. The rust grade is rated on scale from 10 to 0, where 10 is no rusting and 0 is severe rusting. The results of salt spray test are represented in Table 7. Figure 3 shows that there are significant differences between the tested formulations, which depend upon the amount of modifier present *N-phthaloyl glutamic acid*. The anti-corrosive performance of coatings containing the modifier, improves with increased levels of the modifier. The improvement in corrosion resistance may be attributed to the introduction of a compound that contains amide and imide linkages, which results in poly (amide), containing a NPGA ring within the resin structure. Also, the high molecular weight of the modifier decreases the permeability of coating to water, oxygen and the aggressive ions such as chloride. The corrosion resistance increase is also due to the absorption of resin onto the metal through its hydroxyl function.

Table 6: Primer formulations of based on [NPGA] modified polyesteramide resin.

[illegible]

Table 7: Evaluation the corrosion resistance of primer formulations.

Formulation No.	Blistering		Scribe Failure (mm)	Rust grade
	Size	Frequency		
Ia	6	D	9	5
Ib	7	D	8	6
Ic	7	MD	6	6
Id	8	M	5	7
IIa	6	D	9	5
IIb	7	MD	6	7
IIc	7	M	6	7
IId	9	F	5	8
IIIa	7	MD	8	5
IIIb	8	M	7	7
IIIc	8	M	6	8
IIId	9	F	4	8
Iva	7	MD	8	6
IVb	8	M	6	7
IVc	8	M	5	8
IVd	9	F	4	8

**Fig. 4:** Impact resistances of painted films.

4. Conclusion:

We have successfully modified polyesteramide resin by the partial replacement of phthalicanhydride [PA] as dibasic acid source by *N-phthaloyl glutamic acid* with hydroxy ethyl linseed oil fatty acid amide (HELA) as the ingredient source of the polyol, where the time of reaction and curing time are the only minor drawbacks. The modified resin shows enhanced physico- mechanical properties such as gloss, adhesion, scratch hardness and resistance to mechanical damage. It is also observed that the modified resin shows better film performance in terms of chemical resistance to water, acid and alkali. The corrosion resistance of the modified NPGA resins, incorporated within primer formulations, also improves with increased levels of the modifier. This improvement may be attributed to (i) the combination of amide and imide groups, which results in poly (amide), which containing a NPGA ring within the resin structure; (ii) the presence of repeating units of ester and amide in the polymeric chain of the modified polyesteramide; and (iii) the high molecular weight of the modifier decreases the permeability of the coating to water, oxygen and the aggressive ions such as chloride.

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