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Preparation and Characterization of Polyester Amide Resin derived from Nigerian Soya Bean Oil and its Performance Evaluation as Binder for Surface Coatings.

[1]Musa, H., [2]Hamada, A. and [2]Jamila, M. K.
[1]Department of Pure and Industrial Chemistry, Bayero University, P. M. B. 3011, Kano, Nigeria.
[2]Department of Pure and Industrial Chemistry, Umaru Musa Yar'adua University, P.M.B. 2018, Katsina, Nigeria.
Email: hmusa.chm@buk.edu.ng, jamilakankara779@gmail.com

ABSTRACT

Polymeric systems based on polyesteramides (PEA) are high performance materials, which combine the useful properties of polyester and polyamide resins. In this work, a new modified polyesteramide resin was prepared and evaluated as binder for surface coating.The resin was synthesised by the reaction of N,N-bis-(2-hydroxyethyl) soya bean oil fatty acid amide (HESFAA) as a source of the polyol and as preservative against microbiological attack, with phthalic anhydride as the ingredient source of the polybasic acid. The measured acid value of the resin was found to be 7 mg KOH/g, which indicates the complete formation of the resin. The structure of the resin was confirmed by FT-IR spectral studies. The results show that the modification enhances both phyisco-mechanical and chemical properties of the PEA. From the analyses carried out, the synthesized resin could be suitable as binder for different types of paint.

Keywords: Binder, Phthalic anhydride, Polyester amide, Soya bean oil fatty acid

INTRODUCTION

The current utilization of renewable raw material resources by chemical industries is 10-20% and it is expected to reach up 25% by 2020 (Lucas, 2010). Polymer industries compel from its dependence on non-renewable sources and accept industrially applicable renewable sources (Meier and Lucas, 2010). In Nigeria, the demand of alkyd resin has increased over the years even though technical information on local production is scanty and large quantities of oils are always needed for the production of alkyd resin (Onukwli and Igbokwe, 2008). Alkyd resins are widely used in the paint and coating industries and are formed by condensation reaction between polyalcohol and carboxylic acids (Blaise *et al.*, 2012).

Polyester amide 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. The presence of repeating units of ester (-COOR) and amide (-NCOR) in the polymeric chain of polyester amide improves the ease of application, thermal stability, chemical and water resistance, and also contributes to faster drying and enhanced hardness compared to normal alkyds (Bori *et al.*, 1975).

Gast *et al.*, (1969) have prepared several polyester amides from conventional and non-conventional seed oils to improve their drying ability and mechanical and corrosion protective efficiency. 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.

New modified polyester amide compositions were prepared and evaluated as anticorrosive varnish. The resin prepared by partial replacement of hydroxyethyl fatty acid amide (HEFA) by polyethylene glycol (PEG) without affecting the resin constants. Primer formulations based on this resin showed good corrosion inhibiting properties (Aqeel *et al.*, 2010).

Also new modified antimicrobial polyester amide compositions were prepared and evaluated as vehicles for surface coating (AbdEl-Wahab, 2008).

Similarly, polyester amides were prepared by palladium-catalysed polycondensation reaction of aromatic iodides and amino alcohols in the presence of carbon monoxide. The polymer produced exhibited characteristics infrared frequencies corresponding to amide and ester carbonyl groups. The rate of of carbonylation and intrinsic viscosity of the resulting polyester amide increase with an increase in the carbon monoxide pressure (Shrikant, 2004).

Polymeric systems based on polyester amides, prepared from diethanolamine derivatives of fatty acids are high performance materials and finds many applications, most importantly as protective surface coatings (Moustafa, 1992).

MATERIALS AND METHOD

Materials

Materials used for this study were soya bean oil fatty acid, diethanolamine, phthalic anhydride, xylene and ethanol. All glassware used were washed thoroughly with distilled water and dried in an oven, weighing was carried out on an electric balance model AR2140.

Methods

Preparation of N, N-bis (2-hydroxyethyl) Soya Beans Oil Fatty Acid Amide

A mixture of freshly distilled diethanolamine DEA (0.1 mol) and soya bean oil fatty acid (0.1 mol) was placed in 250 ml round-bottomed flask fitted with dean stark apparatus and condenser. The mixture was allowed to reflux until approximately the theoretical amount of water (0.1 mole, 1.8ml) was collected, indicating the complete formation of N, N-bis (2-hydroxyethyl) soya bean oil fatty acids amide (HESFAA).

Synthesis of Long Oil-Length Polyester Amide Resins.

Polyester amide resin was prepared through a condensation polymerization reaction via a solvent process, in a one step reaction. A mixture of the calculated amount of hydroxyethyl soya beans oil fatty acid amide (HESFAA) as the ingredient source of the polyol (0.1 mol), and phthalic anhydride (PA) as the ingredient source of the dibasic acid (0.1 mol) were mixed in a 250 ml round-bottom flask fitted with a dean stark trap. The temperature of the reaction ranged from 140 to150^{o}C. The ingredients were refluxed in the presence of 10% w/w xylene as solvent. The solvent was removed on a rotary evaporator and the resin was obtained as dark brown solid, characterised by FTIR and ^{1}H NMR spectroscopy.

Water Resistance Test

The varnish was poured on a glass panel (3 x 5 inches) and allowed to dry for 48 hours at 25ºC and 10 % relative humidity in nearly vertical position. The edges of the panels were sealed by paraffin wax. The coated panels were placed in a beaker containing about 2.5 liters of distilled water at room temperature and allowed to remain in the water for 24 hours. The panel was then removed from the water, wiped carefully and allowed to dry at room temperature. The panel was tested for any change. To accelerate the test, the same procedure was applied with the exception that water was gently boiled throughout the immersion period, which may be from 15 minutes to several hours. The panels were removed and examined. The films were considered not affected when no film defects were observed after 24 hours when immersed for 15 minutes in boiling water.

Alkali resistance Test

The test panels were coated with the material to be tested and allowed to dry for 24 hours. The edges were coated by dipping in molten paraffin wax. The coated panels were then immersed in an aqueous solution containing 5 grams of anhydrous sodium carbonate per 100 ml of water, for four hours. The panels were removed, washed and the films examined for any defects immediately after drying for one hour.

Acid Resistance

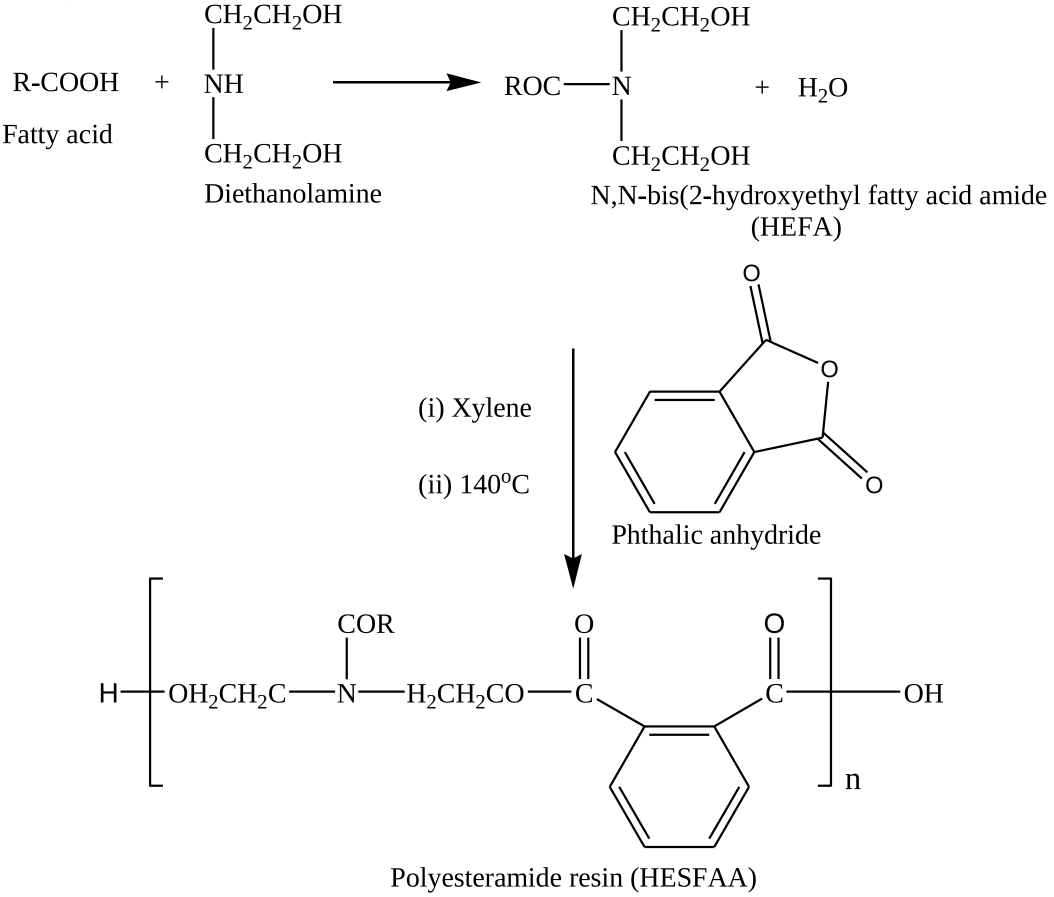
The panels were prepared in the same manner as in the previous test and immersed to half its length in solution containing 20 grams of sulphuric acid per 100 ml of water (20% w/v) and allowed to stand for 24 hours at room temperature. The panels were then removed and washed with cold water and allowed to dry in vertical position for two hours at room temperature and the immersed portion of the films was examined for any defects.

Solvent Resistance

The coated panels were immersed in benzene/mineral turpentine solvent mixture (1:3) for 15 minutes at room temperature. The panels were removed from the test solvent followed by drying in vertical position for one hour. The panels were then examined for any defects.

RESULTS AND DISSCUSION

Polyesteramide antimicrobial resin was successfully synthesised prepared by the reaction of N,N-bis-(2-hydroxyethyl) soya bean oil fatty acid amide (HESFAA) as a source of the polyol and phthalic anhydride as the ingredient source of the polybasic acid according to the reaction Scheme 1.



Scheme 1: Synthesis of Polyester amide Resin.

The successful formation of the resin was confirmed by Fourier Transform Infrared (FTIR) and proton Nuclear Magnetic Resonance (NMR) spectroscopy.

The FT-IR Spectrum of the Polyester Amide Resin

The FT-IR spectrum of the polyester amide resin (Figure 1) shows key absorption peaks which indicates the formation of the resin.

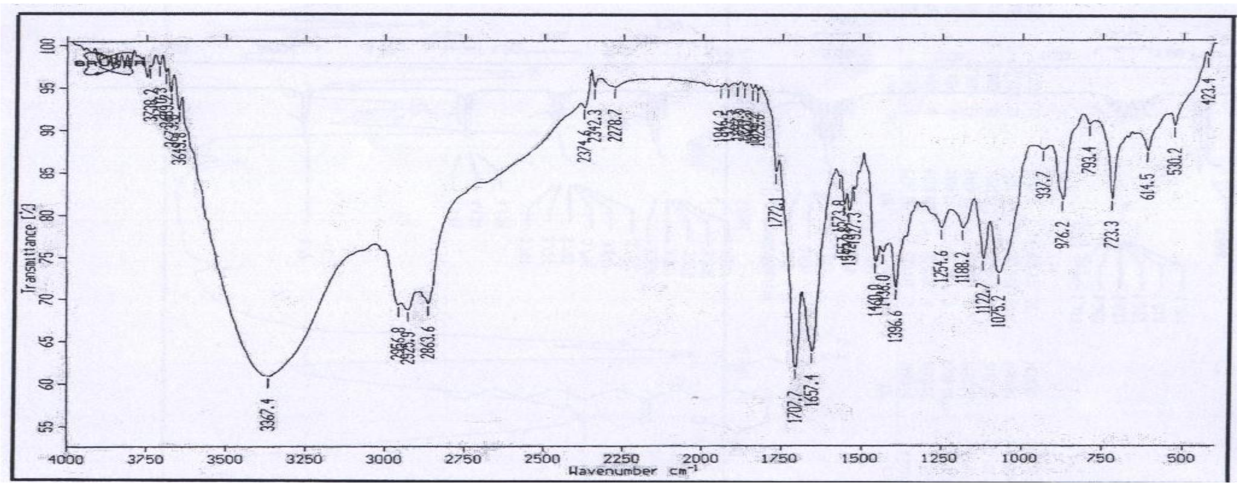


Figure 1: FTIR Spectrum of the Synthesised Polyester Amide Resin

Functional group IR peaks (cm^{-1}): OH (3394), CON amide carbonyl (1657), COO ester carbonyl (1707), C-N (1460), CH_2 symmetric stretch (2930) CH_2 asymmetric stretch (2852) and ring stretching vibration of aromatic nuclei was observed at 723 cm^{-1}.

^{1}H **NMR Spectrum of the Synthesised Polyester Amide Resin**

Similarly, ^{1}H NMR spectral analysis of the resin as presented in Figure 2 has further confirmed the successfull preparation of the polyester resin.

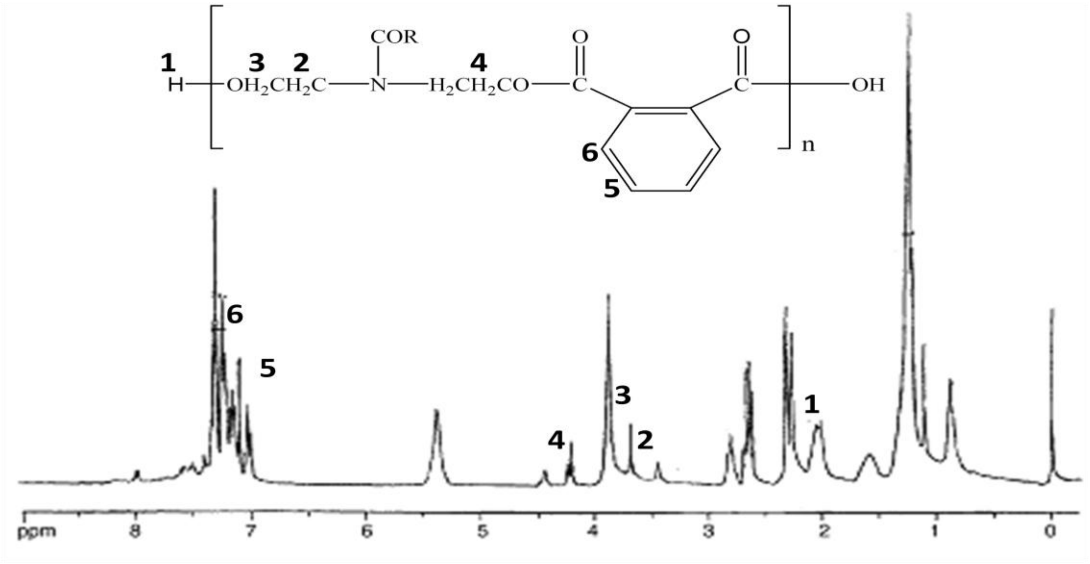


Figure 2: ^{1}H NMR Spectrum of the Synthesised Polyester Amide Resin.

The ^{1}H NMR spectrum (Fig.2) shows characteristic peaks for aromatic ring protons of phthalic anhydride at 7.80-6.90 ppm. The peaks due to CH_2 attached to ester, free hydroxyl and amide nitrogen at 4.35 ppm, 3.75 ppm and 3.56 ppm respectively while the peak due to hydroxyl group occur around 2.20 ppm.

Physical Characteristics of the Polyester Amide Resin Coating Blend

Table 1 shows the results of physical characteristics of coating blends for the resin. Resin was coated into three different glass plates labeled A, B and C. The degree of gloss was 55, 60 and 65 at 60^0 angle and scratch hardness of 1.5, 2.0 and 2.0 kg respectively, the colour obtained was >18 and adhesion 5B for the coated resins A, B and C respectively.

Table 1: Physical Characteristics of Coating Blends

Resin No.	Color (Gardner)	Air drying time (h)	Gloss at 60^{o}	Adhesion	Scratch hardness (kg)
A	>18	48	55	5B	>1.5
B	>18	72	60	5B	>2
C	>18	72	65	5B	>2

Chemical resistance test of the polyester amide coating blends

This test shows that the modified resin shows better film performance in terms of chemical resistance to water, acid, solvent and alkali (**Table 2**).

Table 2: Chemical Resistance of Coating Blends (days)

Resin No.	Water resistance	Acid resistance	Alkali resistance	Solvent resistance
A	> 90	84	40	2
B	> 90	>90	40	2
C	> 90	> 90	45	4

The resin shows a perfect solvent resistance because it can take more than 90 days in solution (2, 2 and 4 days for coated resins A, B and C), water resistance is good here because the polyester amide resin is a water-resistant polymer (>90 days for all the coated resins), the acid resistance is fairly good because it is easily affected by H_2SO_4 (80, 90 and 90 days respectively) but alkali resistance is poor (40, 40 and 45 days respectively) which indicates that the polyester amide is easily hydrolyzed by Na_2CO_3.

CONCLUSION

Polyester amide resin was prepared by the reaction of phthalic anhydride as dibasic acid source with hydroxy ethyl soya beans oil fatty acid amide (HESFAA) as source of the polyol. The yield obtained is very promising, suggests the suitability of soya bean oil as raw material for resin preparation. FTIR and ^1H NMR analyses of the resin have confirmed the successful synthesis of the polyester-amide resin (PEA) as evident from the key absorption frequency (cm^{-1}) and chemical shift (ppm) observed in the spectra.

Based on the results of the performance evaluation of the PEA, it is evident that Nigerian Soya bean oil fatty acid can be utilised as feedstock for the manufacture of polyester amide resin. The modified resin shows enhanced physico-mechanical properties such as gloss, adhesion, scratch hardness and resistance to mechanical damage. It was also observed that the modified resin shows better film performance in terms of chemical resistance to water, acid and alkali hence can serve as a suitable binder for different type of paints.

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