Formulation of Paints Using Azo Pyridone Pigments and Alkyd (Soya Beans Oil), Characterisation and Their Application on Leather

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***Abstract*-The use of locally available raw materials in the formulation of paint is of paramount importance as it provides job opportunity for poor local people, produce ecofriendly and biodegradable products as well as substantiates the aspect of modern biotechnology in developing countries. In this research,Paints were formulated using azopyridone complexes and soya alkyd derived fromesterification of soya oil with maleic anhydride and glycerol. The formulations were applied on leather using hand brush, and the samples produced were subsequently, subjected to physical characterisation such as density, viscosity, solubility, surface drying times, pH and light fastness and also compared with the commercial paint. The pH and the fastness property of the coatings were found to be 8 and 6-7, respectively, which is very good.**

Keywords **– Pyridone pigment, soya bean, alkyd, leather, viscosity.**

I. INTRODUCTION

Paint is a term used to describe a number of substances that consist of a pigment suspended in a liquid or paste vehicle such as oil or water [1]. Azo pyridone pigments were found to be very brilliant with different shades and also high melting points [2, 11]. Someazopyridone metal complexes were synthesised and paints formulated using polyvinyl alcohol as binder, characterized and subsequently, applied on wood substrate. The coated samples were subjected to physical characterisation and their overall performance was found to be very good as sighted "in press" [3].Based on the aforementioned coupled withexcellent performance of the complexes in emulsions paints, hence, formulation of oil paints wasstrongly recommended.

Therefore, thispaperdescribed the formulation of paints using alkyd (soya beans oil) and some selected azopyridone pigments, their characteristics and application on leather.

II. MATERIALS AND METHODS

A. Materials:

- The pigments used in the formulation of paints were synthesised and sourced from the Department of Textile Science and Technology, Ahmadu Bello, University, Zaria – Nigeria [2]. See appendix 1.
- Alkyd resins were synthesized by esterification reaction of refined soya oilwith maleic anhydride and glycerol.
- Leather was obtained from Nigerian Institute of Leather and Science Technology (NILEST), Zaria.

B. Preparation of Soya beans Alkyd Resin

The reaction was carried out in two stages:

- Alcoholysis: the first step is alcoholysis or monoglyceride reaction of oil with glycerol in the presence of calcium oxide as catalyst. 40g of oil, 8g of glycerol and 0.04g of calcium oxide was charged into 1 liter three necked flask with dean stack apparatus connected to a reflux condenser with nitrogen gas.
    - The mixture was then heated to 180°C for one hour under constant mechanical stirring.
    - The completion of the reaction was verified by mixing one part of the reaction mixture in three part of methanol at room temperature. The mixture was completely dissolved in methanol and gave a clear liquid indicating that monoglycerolysis was completed [4].
- Esterification: the reaction was done, when the mixture was cooled down to 150°C, and then 20g of maleic anhydride, 8g of glycerol were added,4ml of xylene was introduced to aid distilling of water molecules by azeotropical method and heated to 180-

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200°C again. The reaction was maintained at this condition for 5 hours [5].

C. Formulation of Alkyd Paint

Table 1: The Recipe Used For the Formulation of Paints

Raw Materials	Quantity (g)
Azo-pyridone pigments	0.5
Alkyd resin	14
Solvent(petroleum spirit)	7.54
Cobalt	1.08
Anti–skin	0.54
Cobalt naphthanate	0.54
Titanium dioxide	5.38
Total weight of recipe	29.58

Azo pyridone pigment and titanium dioxide were grinded in a ceramic mortar and pestle until it becomes fine powder, then alkyl resin (soya bean oil) was added to form a paste. Solvent (petroleum spirit), cobalt, anti-skin and cobalt naphthanate were added and the paste was thoroughly mixed until homogenous paste was formed. Same procedure was carried out using the remaining four azopyridone pigments as shown in Appendix 1.

The recipe used for the formulation of paints is shown in 1.

$$\text{Pigment Mass Concentration (PMC)} = \frac{\textit{Mass of pigment}}{\textit{Total mass of recipe}} \times 100 \quad (1)$$

$$= \frac{0.5}{29.58} \times 100 = 1.7\%$$

The formulated paints were subjected to series of test which include,

D. Determination of Density

In the coating industry the density of the paint is important because of its influence on other factors such as pigment dispersion, brushability of paint, leveling and sagging [6]. The density was determined by taking the weight of known volume of paint inside a density bottle as shown in Table 2.

E.Viscosity Measurements

Viscosity measurements of the formulated paints were carried out using viscometer (Brookfield synchro electric viscometer) with spindle number 4 at a speed of 100r.p.m.This was achieved by inserting the paddle into the paints in a container, and hence viscosity values were taken. The results are shown in Table 2.

F.Solubility Test

This is done to check the solubility of the paints in different solvents. The solubility of the paints formulated were tested in the following solvents; methanol, kerosene, sulfuric acid and water. The results are shown in Table 3.

G.pH Test

The pHmetre was used to check the alkalinity or acidity of the paints.

H. Application of Paints to Substrate (leather)

The leather was cleaned and ensured to be free from dirt and other imperfections. The paints were applied on leatherusing hand brush. Series of test were carried out on the coatings such assurface drying time, hard drying time, water resistance and light fastness.

I. Surface Drying Time

This measure relative degree of cure (dry to touch), It was measure by the quantitative finger marking test [7].

The sample was painted using hand brush and the time taken for the coated sample to dry was measured by touching the coated sample with white paper until when no stain was noticed on the paper. The timing was achieved using stop watch. The experiment was repeated for all the five paints formulated including the commercial paint which was used as control. The results are shown in Table 4.

J. Hard Drying Time

This was the time when the sample was completely dried and could withstand scratch. The results are shown in 4.

K. Water Resistance

This test was carried out to show the stability of the paint in the presence of water swelling, blistering and absorption. Moisture absorption taking place in paints can lead to swelling of the film and if this severe blisters form. However, highly pigmented film has less swelling but greater permeability to both air and moisture can lead to severe blistering.

The coated samples were immersed in a beaker of water for 24 hrs.

L. Light Fastness

An area of the leather 3x2 cm was used; the side to be tested was exposed to light from a Xenon arc lamp (Microscal light fastness tester) along with the eight blue wool standard for 96 hours. The fastness was assessed by comparing the fading of the leather to that of the standard by the procedure outlined in ISO 105 (SLTC, 1996). The results obtained are shown in Table 5.

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III. RESULTS AND DISCUSSIONS

A. Density

Table 2: Density and Viscosity Values of the Formulated Paints and Control

Paint	Density (g/cm^3)	Viscosity(Ns/M×10^{-2})
Paint A	1.1150	10.5
Paint B	1.1450	10.5
Paint C	1.1002	9.5
Paint D	1.1002	9.7
Paint E	1.0720	9.5
Control	0.9785	12

Paints formulated from the metalised pigments were found to have higher density than the commercial paint; this could be as a result of difference in composition ratio and also the pigment used. The densities of the paints and control are shown in Table 1.

B. Viscosity

The viscosity of paint must be low enough to allow flow and high enough to inhibit sag (runny paint). Decorative, brush applied paints and paints designed to be highly protective tend to have higher viscosities [3]. The viscosities of paints formulated were found to slightly higher than the control sample acquired from the market. This could be due to the different molecular mass and the quantity of pigments adopted in the formulation of the paints. The viscosities of formulated paints and the control are show in Table 2.

B. Solubility Test

Table 3: Solubility of Paints in Various Solvents

PAINTS	SOLVENTS				
	Water	*Methanol*	*Kerosene*	*Petroleum spirit*	*Sulfuric acid*
Paint A	I	I	S	S	I
Paint B	I	I	S	S	I
Paint C	I	I	S	S	I
Paint D	I	I	S	S	I
Paint E	I	I	S	S	I
Control	I	I	S	S	I

S = *Soluble* and I = *Insoluble*

This test shows the solubility of the formulated paints in various solvents, as shown in Table 3.

D. pH Test

The test indicates the alkalinity of the paint. The paints formulated are found to have pH values of 6.4 to 6.6, which indicate that the paints are alkaline. The result conforms to standard [8].

E. Surface Drying Time

Table 4: Surface and Hard Drying Times of the Paints

Paints	Surface Drying Time (hour)	Hard Drying Time (hour)
Paint A	1:30	24
Paint B	1:40	24
Paint C	1:45	24
Paint D	1:50	24
Paint E	1:55	24
Control	1:00	6

The drying time must be considered in the manufacture of paint because very fast drying paints are prone to brittleness and if too slow the paint may be susceptible to pick up dirt and other impurities [9].

The surface drying time of the formulated paints are shown in Table 4, this is almost the same with the commercial one which was used as control. This shows that the paint formulated has good surface drying time.

F. Hard Drying Time

The hard drying time of the formulated paints were found to be 24 hours to completely dry and able to withstand scratches, compared with the control sample which takes 6 hours to completely dry. This difference in drying time could be due to different driers used in the formulation.

G. Water Resistance Test

The water resistance test carried out on the coated samples shows that the paint formulated can resist water penetration into the film.

H. Light Fastness

Table 5: Light Fastnesses of Painted Samples

S/N	Paints	Light Fastness Rating
1	Paint A	7
2	Paint B	6
3	Paint C	7
4	Paint D	7
5	Paint E	7
6	Control Paint	7

As seen in Table 5, the paints, generally, have a very good light fastness. Although, paint formulated using a pigment derived from nitro aniline have shown dulling effect (slightly darkened) on exposure to the light. The overall good fastness experienced could be as a result of the heavy metals that protect the azochromophore of the pigment against ultraviolet (UV) light degradation. The results conform to Hooker, J [10].

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IV. CONCLUSION

Oil based paints using alkyd resin from soya beans oil was successfully formulated using some azopyridone pigments. Some properties such as density, viscosity, and pH were eventually determined and compared with the commercial paint acquired from the market. The selection of soya beans oil was as a result of it been bio-degradable and non toxic.

The paints formulated were applied to leather using hand brush, subsequently; the coated samples were subjected to surface drying time, hard drying time, hardness test as well as light fastness. During these test the oil based paints formulated were found to have very good performance.

V. RECOMMENDATION

The formulation of oil based paint (soya beans oil) with azopyridone pigment has been a success, and its overall performance found to be good. Therefore, we strongly recommend the formulation of oil based paint with the same pigments using different alkyd resins derived from other oils such as neem oil, castor oil etc and their application on metals. In addition, refractive index and emission test should be carried out.

Appendix 1: Pigments Used In the Formulation of Paints

Paints	Pigments	Complexes
Paint A	ECu	Cu(II) complex of 3-(p-Aminobenzoic)azo-1-methyl-2-hydroxyl-4-methyl-5-cyano-6-pyridone
Paint B	BCu	Cu(II) complex of 3-(p-Bromophenyl) azo-1-methyl-2-hydroxyl-4-methyl -5-cyano-6-pyridone
Paint C	BCr	Cr(II) complex of 3-(p-Bromophenyl) azo-1-mehyl-2-hydroxyl-4-methyl-5-cyano-6- Pyridone
Paint D	ENi	Ni(II) complex of 3-(p-Bromophenyl)azo-1-methyl-2-hydroxyl-4- methyl-5-cyano-6- Pyridone
Paint E	BNi	Ni(II) complex of 3-(p- Amino benzoic) azo-1-methyl -2- hydroxyl -4-methyl-5-cyano-6-pyridone

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