

Synthesis and effect of 7-{4-[2- Chloro- 4-N,N-di (2-hydroxyethyl) amino] phenylazo} phenyl- 6-oxo-2-methyl-4-(4-methoxy- 3-methylphenyl)- oxazolo [5,4-b] pyridine On Polyester Fiber

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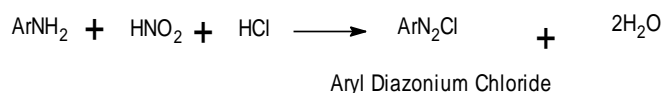
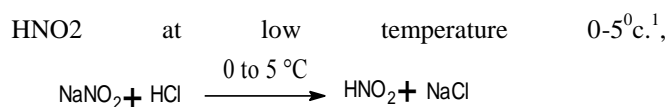
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Abstract— The demand for various dyes of high wet fast ness on polyester isincreasing Polyestes tends to have better fastness to wet treatments such as washing and perspiration fastness . all disperse dyes gave very good to excellent results. Azo dyes cover a whole gamut of colours, from yellow to brown hues, by varying theintermediates especially when heterocyclic diazo components are coupled to aminobenzene couplers substituted with powerful electron donating groups.

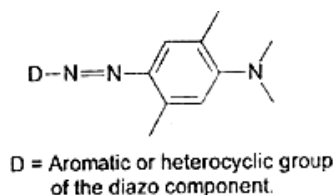
Keywords- Disperse azo dyes, Diazoniumsalt , XENO test, Thermo Test, Tinctorial Power

I. INTRODUCTION

Dyes are coloured, ionising, aromatic organic compounds. These are synthesized by two-stage process : Diazotisation & Coupling. Diazotisation: - The process in which primary aromatic amino compound convert into the diazonium salt is known as Diazotisation, this reaction is carried out in presence of



Coupling: - When diazonium ring gets attached to another aromatic ring, then it is called coupling. It can be represented by general formula (1) given below, where D is Aromatic or Heterocyclic group of the diazo component.²



II. MATERIALS AND METHODS

For synthesis of 7-{4-[2- Chloro- 4-N,N-di (2-hydroxyethyl) amino] phenylazo} phenyl- 6-oxo-2-methyl-4-(4-methoxy-3-methylphenyl)- oxazolo [5,4-b]pyridine dye following steps ;

Preparation of 3-(4-methoxy-3-methylphenyl)-2-pentene-1,5-dioic acid (x):

To the acetone dicarboxylic acid prepared from citric acid (400 gms, 2.25 moles) and concentrated sulphuric acid (98 %) (640 ml, d=1.83) 2-methyl anisole (140 ml, 2.10 moles) was added slowly with stirring at -2 to 0°C over a period of 1 hour. Stirring was continued further, for a period of 3 hours and the temperature of the reaction was maintained at 0-5°C. The contents were then poured in about 150ml cold water under stirring. The precipitated solid thus obtained was crystallized from boiling water to get the colorless needles of the product.³

Melting point : 161-162°C

Yield : 180

gms (32 %)

Elemental Analysis :

Found : C, 62.46 ; H, 5.62

Calc. For C₁₃H₁₄O₅ : C, 62.40 ; H, 5.60

Preparation of 1-(4-Acetamidophenyl)-4-(4-methoxy-3-methylphenyl)-2,6-(1H,5H)-pyridinedione (y) :

Compound x (25 gms, 0.1 moles) was thoroughly mixed with finely powdered 4-aminoacetanilide (15 gms, 0.1 moles) and was heated in an oil bath at 170°C for 30 minutes. The fused mass was cooled to room temperature

and treated with aqueous sodium carbonate, followed by water and then with dilute hydrochloric acid to give compound **y**. The solid obtained was crystallized from glacial acetic acid to yield buff colored compound.⁴

Melting point : 239 – 241°C

Yield : 32.7 gms (90%)

Elemental Analysis :

Found : C, 69.19 ; H, 5.52 ; N, 7.65

Calc. For C₂₁H₂₀N₂O₄ : C, 69.23 ; H, 5.49 ; N, 7.69

Preparation of 1-(4-Acetamidophenyl)-5-amino-6-hydroxy-4-(4-methoxy-3-methylphenyl)-pyridine-2(H)-one (z):

To a solution of **y** (18.2 gms, 0.05 moles) in 25 ml of acetic acid, 10 ml of N,N-dimethylformamide and 2 drop of conc. HCl acid maintained at 0-5°C, solution of NaNO₂ (3.45 gms, 0.05 moles) in 10 ml water was added in small portion with stirring. The rate of addition was controlled so that the temperature did not rise above 12°C. After the NaNO₂ was added, the mixture was stirred further for a period of 2-3 Hrs. The temperature of the reaction mixture was then allowed to reach to room temperature and was stirred for another 4 Hrs. The reaction mixture was then poured in to ice cold water when yellow coloured oxime separated out. It was filtered, washed with water and dried.⁵ Oxime thus obtained was dissolved in about 30 ml acetic acid and cooled to 15°C. To this solution zinc dust (3.40 gms, 0.052 moles) was added in portion with vigorous stirring. The rate of addition was regulated so that temperature never rises above 60°C. After the addition was complete, the reaction mixture was refluxed for 2 – 3 Hrs., until the unreacted zinc dust collects in balls. The hot solution was then filtered and poured in ice cold water. Yellow coloured solid was separated out. It was filtered, washed with water several times and dried.⁵

Melting Point : 282- 285°C Yield : 9.85 gms (52 %)

Elemental Analysis

Found : C, 66.55 ; H, 5.49 ; N, 11.05.

Calc. For C₂₁H₂₁N₃O₄ : C, 66.49 ; H, 5.54 ; N, 11.08.

Preparation of 7-(4-Aminophenyl)-6-oxo-2-methyl-4-(4-methoxy-3-methylphenyl)-oxazolo [5,4-b] pyridine (W):

A mixture of compound **Z** (5.685 gms, 0.015 moles) and acetic anhydride (15 ml) was refluxed for 3 Hrs, cooled to room temperature and then slowly added to a cold aqueous methanol solution (1:1:25 ml) with stirring. The mixture was neutralized with sodium carbonate, the product obtained was filtered, washed with water and dried. Product thus obtained was refluxed in hydrochloric acid : acetic acid mixture (15-10 ml) for 2 Hrs. The reaction mixture was cooled and poured in to ice cold water, it was neutralized with dilute sodium hydroxide solution when faint brown

colour solid separated out. Solid was filtered, washed several times with water and dried. It was crystallized from ethanol.⁶

Melting Point : 263-265°C Yield : 2.76 gms (51 %)

Elemental Analysis :

Found : C, 69.85 ; H, 5.24 ; N, 11.59

Calc. for C₂₁H₁₉N₃O₃ : C, 69.80 ; H, 5.26 ; N, 11.63

Preparation of 7-{4-[2-Chloro-4-N,N-di (2-hydroxyethyl) amino]phenylazo} phenyl-6-oxo-2-methyl-4-(4-methoxy-3-methylphenyl)-oxazolo [5,4-b] pyridine:

A solution of compound **W** (0.361 gms, 0.001 moles) in glacial acetic acid (5 ml) and propionic acid (1 ml) was added drop wise with stirring over a period of 45 minutes to a cold (5°C) mixture of nitrosyl sulphuric acid [prepared by dissolving solid sodium nitrite (0.075 gms, 0.001 moles) in conc. sulphuric acid (8 ml) at 70°C]. The reaction mixture was stirred for an additional period of 2 Hrs. maintained at 5 – 10°C. The reaction mixture was then added to propionic acid – acetic acid mixture (2-10 ml) under stirring. The excess of nitrous acid was removed using urea (about 0.2 gms). The mixture was filtered to get a clear diazonium salt solution.

3-Chloro-4-N,N-di (2-hydroxyethyl) aniline (0.215 gms, 0.001 moles) was dissolved in propionic acid – acetic acid mixture (4-20 ml). The solution was externally cooled at 5°C. The previously cooled diazonium salt solution was slowly added to the mixture maintained at 5-10°C over a time period of 1 Hr. The pH of the reaction was maintained acidic (4 – 5) throughout the coupling period by addition of solid sodium acetate. After the addition of diazonium salt solution was over, the reaction mixture was stirred further, for a period of 3 Hrs. The mixture was neutralized with acetate solution. The separated monoazo dye was filtered, washed thoroughly with cold water and dried. The monoazo dye was crystallized from DMF- methanol mixture.

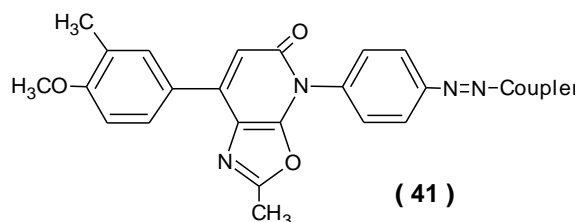
Melting Point : 232-236°C Yield : 0.340 gms (58 %)

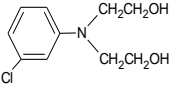
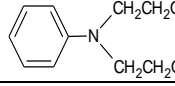
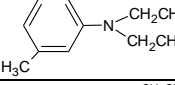
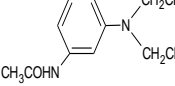
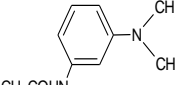
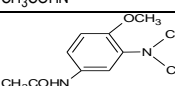
Elemental Analysis

Found : Nitrogen 11.95 %

Calc. for C₃₁H₃₀ClN₅O₅ : Nitrogen 11.91 %

All the other dyes (**b - f**) were synthesised by using the same method. The characterization data of the dyes thus prepared is depicted in table ⁶



| Compound No. | Coupler | Yield % | M.P. (°C) | Molecular Formula | ELEMENTAL ANALYSIS | |
|--------------|---|---------|-----------|---|--------------------|-------|
| | | | | | Nitrogen % | |
| | | | | | Calculated | Found |
| a |  | 58 | 232-236 | C ₃₁ H ₃₀ ClN ₅ O ₅ | 11.91 | 11.95 |
| b |  | 60 | 221-225 | C ₃₁ H ₃₁ N ₅ O ₅ | 12.65 | 12.60 |
| c |  | 55 | 202-206 | C ₃₂ H ₃₃ N ₅ O ₅ | 12.34 | 12.38 |
| d |  | 53 | 211-215 | C ₃₃ H ₃₄ N ₆ O ₆ | 13.77 | 13.79 |
| e |  | 54 | 225-229 | C ₃₃ H ₃₄ N ₆ O ₄ | 14.53 | 14.58 |
| f |  | 59 | 245-250 | C ₃₄ H ₃₆ N ₆ O ₅ | 13.81 | 13.86 |

III. RESULTS AND DISCUSSION:

VISIBLE SPECTRAL DATA AND DYEING PROPERTIES of Dyes

| Dye No. | Colour of Dye on polyester fabric | Absorption maxima max (nm) | Log | Pick - up | Xeno | Thermo |
|---------|-----------------------------------|--------------------------------|-------|-----------|-------|--------|
| a | Yellow | 451 | 4.090 | 3 | 3 – 4 | 4 |
| b | Yellowish Orange | 460 | 4.120 | 3 | 3 – 4 | 4 |
| c | Yellowish Orange | 466 | 4.152 | 3 | 3 – 4 | 4 – 5 |
| d | Yellowish Orange | 471 | 4.194 | 4 | 4 | 4 – 5 |
| e | Reddish Orange | 476 | 4.212 | 4 | 4 | 4 – 5 |
| f | Reddish Orange | 482 | 4.282 | 4 | 4 | 5 |

Recorded in DMF – Methanol (1 : 99)

For light fastness Sublimation fastness at 200°C for one minute

IV. CONCLUSION

Monoazo dyes **a-f** showed an absorption ranging from 451 nm to 482 nm. These dyes shows absorption intensities in the range of 4.09 to 4.28. Most of the dyes of these series showed moderately high absorption intensities.

Monoazodye **a – f** gave yellow to reddish orange shades on polyester. The pick-up of these dyes were good to very good. The light fastness of these dyes was in the range of 3 – 4 i.e. fair to fairly good and sublimation fastness was good to excellent.

V. ACKNOWLEDGEMENT:

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Dr. Mrs. Anita Abhishek Singh Gour received her graduation and post-graduation in chemistry from DAVV INDORE. She obtained Ph.D. degree from DAVV INDORE in 2007. She has published /presented 4 research papers in various international journals and conferences. She is fellow member of ICCE and also fellow member ISROSET[®], She has more than 20 Years of Teaching. Recently she is Asstt. Professor in Department of Chemical Science at Christian Eminent College Indore from Aug 2011.

