

ABSTRACT

Dyes are chemical compounds that can attach themselves to fabrics or surfaces to give them colour. Most dyes are complex organic molecules and need to be resistant to weather and the action of detergents. Dyes add color to textiles. They are incorporated into the fibre by chemical reaction, absorption or dispersion. Dyes differ in their resistance to sunlight, perspiration, washing, gas, alkalies, and other agents; their affinity for different fibres; their reaction to cleaning agents and methods and their solubility and method of application. In short, dyes are coloured, ionising, aromatic organic compounds. In ancient past, Indigo, obtained from plants was being used by the Egyptians 5000 years ago, and natural dyes obtained from plants and animal sources are still used today. In 1856, Perkin developed the first synthetic dye based upon aniline. In this paper author prepared and analysis azo dye $C_{33}H_{36}N_6O_5$.

KEYWORDS: Xeno test, thermo test, tinctorial power, polyester fabric, synthesis, fastness.

I. INTRODUCTION

Actually dyes are complex organic molecules and need to be resistant to weather and the action of detergents. Dyes add color to textiles. They are incorporated into the fiber by chemical reaction, absorption or dispersion. Dyes differ in their resistance to sunlight, perspiration, washing, gas, alkalis, and other agents; their affinity for different fibers; their reaction to cleaning agents and methods and their solubility and method of application. In short, dyes are coloured, ionising, aromatic organic compounds. These are synthesized by two-stage process : Diazotisation & Coupling. Diazotisation [1-2]. Thus, the present work shows the formation of some novel disperse azo dyes $C_{33}H_{36}N_6O_5$.

II. MATERIALS AND METHODS

Preparation of 3-(4-Methoxy-3-methylphenyl)-2-pentene-1,5-dioic acid (X)

To the acetone dicarboxylic acid prepared from citric acid (400 gm, 2.25 mole) and concentrated sulphuric acid (98 %) (640 ml, d=1.83) 2-methyl anisole (140 ml, 2.10 mole) 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 $0-5^\circ\text{C}$. The contents were then poured in about 150ml cold water under stirring. The precipitated solid was filtered & washed with water. The solid thus obtained was crystallized from boiling water to get the colorless needles of the product [3].

Physical Analysis

Melting point : $161-162^\circ\text{C}$
 Yield : 180 gm (32 %)

Elemental Analysis

Calculation for $C_{13}H_{14}O_5$: C - 62.40, H - 5.60
 Found : C - 62.46, H - 5.62

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

Compound X (25 gm, 0.1 mole) was thoroughly mixed with finely powdered 4-aminoacetanilide (15 gm, 0.1 mole) 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].

Physical Analysis

Melting point	: 239 – 241 ⁰ C
Yield	: 32.7 gm (90%)

Elemental Analysis

Calculation for C ₂₁ H ₂₀ N ₂ O ₄	: C - 69.23 ; H - 5.49; N - 7.69
Found	: C - 69.19; H - 5.52; N - 7.65

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

To a solution of Y (18.2 gm, 0.05 mole) in 25ml of acetic acid, 10 ml of N,N-dimethylformamide and 2 drop of conc. HCl acid maintained at 0-50C, solution of sodium nitrite (3.45 gm, 0.05 mole) 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 120C. After the sodium nitrite was added, the mixture was stirred further for a period of 2-3 hours. The temperature of the reaction mixture was then allowed to reach to room temperature and was stirred for another 4 hours. 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.

Oxime thus obtained was dissolved in about 30 ml acetic acid and cooled to 15⁰C. To this solution zinc dust (3.40 gm, 0.052 mole) was added in portion with vigorous stirring. The rate of addition was regulated so that temperature never rises above 60⁰C. After the addition was complete, the reaction mixture was refluxed for 2 – 3 hours, 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].

Physical Analysis

Melting Point	: 282- 285 ⁰ C
Yield	: 9.85 gm (52 %)

Elemental Analysis

Calculation for C ₂₁ H ₂₁ N ₃ O ₄	: C, 66.49; H, 5.54; N, 11.08.
Found	: C, 66.55; H, 5.49; N, 11.05.

Preparation of 5- [(2-Acetamido-4-N,N-diethylamino)phenyl] hydrazono-1-(4- acetamidophenyl) - 4 - (4-methoxy-3-methylphenyl) -2, 6-(1H)- pyridinedione (We) :

Compound Z (0.379 gm, 0.001 mole) was dissolved in propionic acid (1 ml) and acetic acid (4 ml) mixture. The solution was cooled to about 10⁰C. It was added drop wise to a cold solution (5⁰C) of nitrosyl sulphuric acid [prepared by dissolving solid sodium nitrite (0.075 gm, 0.001 mole) in conc. sulphuric acid (8 ml) at 70⁰C]. The added to propionic acid – acetic acid mixture (2ml-10 ml) under stirring. The excess of nitrous acid was destroyed using urea (about 0.2 gm). The mixture was filtered to get a clear reaction mixture was stirred further, for an additional period of 2 hours at 5-10⁰C. The reaction mixture was then diazonium salt solution.

3-Acetamido-4-N,N-diethylamine coupler (0.206gm, 0.001 mole) was dissolved in propionic acid – acetic acid mixture (4ml-20 ml). The solution was externally cooled to 5⁰C. The previously cooled diazonium salt was slowly added to the reaction mixture maintained at 5-10⁰C over a time period of 1 hour. The pH of reaction mixture 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 hours. The mixture was neutralized with sodium acetate solution. The separated monoazo dye was filtered, washed thoroughly with cold water and dried. The monoazo dye was crystallized from DMF-Methanol mixture [6].

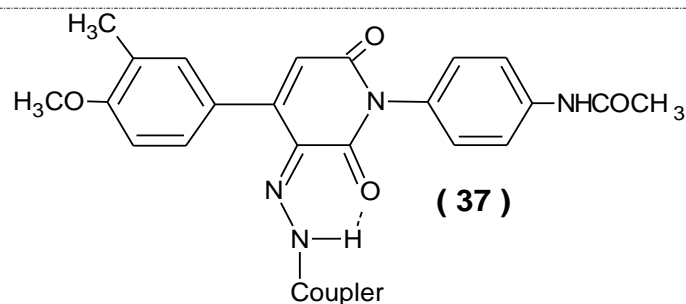
Physical Analysis

Melting Point	: 260-263 ⁰ C
Yield	: 0.187 gm (58%)

Elemental Analysis

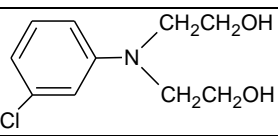
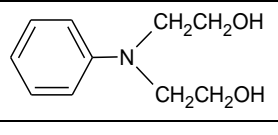
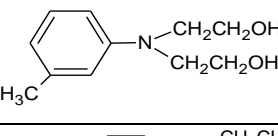
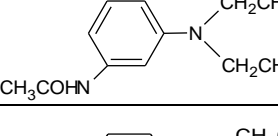
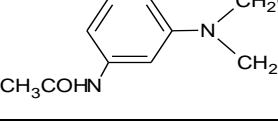
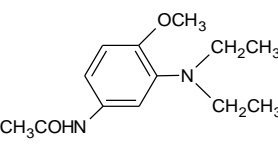
Calc. for C ₃₃ H ₃₆ N ₆ O ₅	: Nitrogen 14.09 %
Found	: Nitrogen 14.15 %

All the other dyes (W a- f) were synthesised by using the same method.



The characterization data of the dyes thus prepared is depicted in table 1.

Table 1: Characterization data of the dyes

Compound No.	Coupler	Yield (%)	M.P. (°C)	Molecular Formula	Elemental Analysis	
					Nitrogen %	
					Calculated	Found
37a		57	192-197	C ₃₁ H ₃₂ ClN ₅ O ₆	11.56	11.52
37b		55	196-202	C ₃₁ H ₃₃ N ₅ O ₆	12.25	12.29
37c		61	205-209	C ₃₂ H ₃₅ N ₅ O ₆	11.96	11.92
37d		60	188-192	C ₃₃ H ₃₆ N ₆ O ₇	13.37	13.40
37e		58	173-179	C ₃₃ H ₃₆ N ₆ O ₅	14.09	14.15
37f		62	205-210	C ₃₄ H ₃₈ N ₆ O ₆	13.41	13.46

III. RESULT AND DISCUSSION

Visible spectral data and dyeing properties of dyes {w(a – f)} are shown in table 2.

Table 2: Visible spectral data and dyeing properties

Dye No.	Colour of Dye on polyester fabric	Absorption maxima * λ_{max} (nm)	Log ϵ	Pick - up	Xeno [♣]	Thermo Θ
A	Golden Yellow	440	4.060	2	2-3	3-4
B	Golden Yellow	443	4.085	2	2-3	3-4
C	Golden Yellow	447	4.102	2	2-3	3-4
D	Yellowish Orange	450	4.112	2	2-3	3-4
E	Yellowish Orange	452	4.135	2	2-3	3-4
F	Yellowish Orange	456	4.154	2	2-3	4

where

- * - Recorded in DMF – Methanol (1 : 99)
- ♣ - For light fastness
- Θ - Sublimation fastness at 200⁰C for one min

Monoazo dyes **W(a-f)** showed an absorption ranging from 440 nm to 456 nm. It was observed that the value of absorption maxima increased with increasing number of electron donating substituents on dye molecule. These dyes also showed absorption intensities in the range of 4.06 to 4.15. The pick-up value for all dyes is found to be 2. The results for xeno test and thermo test lies in the range 2-3 and 3-4 respectively. The most of the dyes of these series showed moderately high absorption intensities.

IV. CONCLUSION

Numerous possibilities to modify the azo dyes synthesis is being utilized to produce desired range of hue of industrial use. In current scenario of Make in India, above synthesis research could be developed to produce economic dyes in our country and we could save significant foreign investment through import.

V. REFERENCES

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CITE AN ARTICLE

Gour, A., Imam, S., & Shivhare, N. (n.d.). ANALYSIS AND PREPARATION OF AZO DISPERSE DYE C33H36N6O5. *INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY*, 6(12), 543-546.