

## GLOBAL JOURNAL OF ENGINEERING SCIENCE AND RESEARCHES ANALYSIS AND PREPARATION OF AZO DISPERSE DYE C<sub>25</sub>H<sub>21</sub>N<sub>5</sub>O<sub>5</sub>

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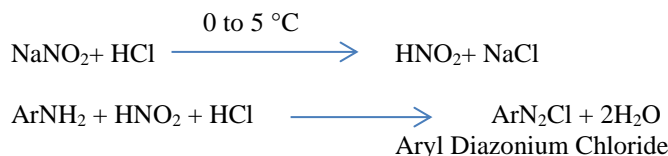
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### ABSTRACT

Disperse Dyes are aromatic heterocyclic organic compound. Advances over the last decade concerning the synthesis, properties and application of azo disperse dyes prepared from diazo components containing aromatic heterocycles are discussed here. Preparation of some novel disperse azo dyes synthesized by the coupling component of diazonium salt 1-(4-Aminophenyl)-4-(4-methoxy-3-methylphenyl)-5-((4-nitrophenylhydrazono)-2,6-(1H)-Pyridinedione (29A) to give the corresponding various azo disperse dyes (A-E). these dyes were applied to polyester fabric and their fastness properties were evaluated.

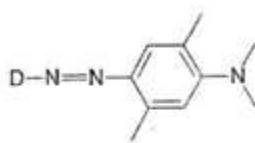
### I. INTRODUCTION

Disperse Dyes are aromatic heterocyclic organic compound. Dyes are coloured, ionising, aromatic organic compounds and as such are based fundamentally on the structure of benzene. These are synthesized by two-stage process:- Diazotisation and Coupling. In 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 HNO<sub>2</sub> at low temperature 0-5 °C.



#### 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 [1].



(1)

D = Aromatic or heterocyclic group of the diazo component.

### II. MATERIALS AND METHODS

#### 1) 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 0-50°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 [2].

**Physical Analysis**

Melting point : 161-162°C  
 Yield : 180 gm( 32 % )

**Elemental Analysis**

Found : C, 62.46 ;H, 5.62  
 Calc. For C<sub>13</sub>H<sub>14</sub>O<sub>5</sub> : C, 62.40 ; H, 5.60

**2) 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 **25** (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 [3].

**Physical Analysis**

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

**Elemental Analysis**

Found : C,69.19 ;H, 5.52 ;N, 7.65  
 Calc. For C<sub>21</sub>H<sub>20</sub>N<sub>2</sub>O<sub>4</sub> : C,69.23 ; H,5.49 ;N,7.69

**3) Preparation of 1-(4-Aminophenyl)-4-(4-methoxy-3-methylphenyl)-2,6-(1H,5H)-pyridinedione (Z)**

Compound **Y**(8 gm, 0.02 mole) was refluxed in hydrochloric acid : acetic acid mixture (20 : 5ml) for 2 hours. The reaction mixture was cooled and poured into ice cold water. It was neutralized with dilute sodium hydroxide solution, white colored solid separated out. The solid was filtered, washed several times with water and dried. It was crystallized from ethyl alcohol [4].

**Physical Analysis**

Melting point : 225 – 229°C  
 Yield : 5.3 gm (75%)

**Elemental Analysis**

Found : C, 70.86 ;H, 5.62 ;N, 8.72  
 Calc. For C<sub>19</sub>H<sub>18</sub>N<sub>2</sub>O<sub>3</sub> : C, 70.80 ;H, 5.59 ;N,8.69

**4) Preparation of 1-(4-Aminophenyl)- 4 -(4-methoxy-3-methylphenyl)- 5 -((4-nitrophenylhydrazono)-2, 6-(1H)- Pyridinedione (WA) :**

4-Nitroaniline (0.138gm, 0.001mole) was dissolved in dil. hydrochloric acid (6ml in 2ml water), in a 50ml beaker and cooled to 0-5°C. Sodium nitrite (0.075gm, 0.0011mol) dissolved in water (4ml) was gradually added to the above solution in small portions with stirring. The reaction mixture was stirred at the same temperature for 20 minutes. The excess of nitrous acid was destroyed with urea (about 0.1gm) and the mixture was filtered to get clear diazonium salt solution.

Compound **Z**(0.322gm, 0.001 mole) was dissolved in dil. Sodium hydroxide solution (15 ml) and cooled to 0-5°C. Previously prepared cooled diazonium salt solution was gradually added to the above solution maintained at 0-5°C over a period of 30 minutes. The pH of the reaction mixture was maintained 8-9 (alkaline) throughout the coupling period. The reaction mixture was further stirred for 3 hours maintained at 0-5°C. The partially separated monoazo dye was completely precipitated by neutralization with dilute hydrochloric acid, was filtered, thoroughly washed with water and dried. It was crystallized from ethanol [5].

**Physical Analysis**

Point Melting : 245 – 247°C  
 Yield : 0.296 gm (66 %)

**Elemental Analysis**

Calc. For C<sub>25</sub>H<sub>21</sub>N<sub>5</sub>O<sub>5</sub> : Nitrogen 14.86%  
Found : Nitrogen 14.80%

All the other dyes **W (B-E)** were synthesized by using the same method. The characterization data of the dyes thus prepared is depicted in **table 1**. All the other dyes ( W a- e ) were synthesised by using the same method [6].

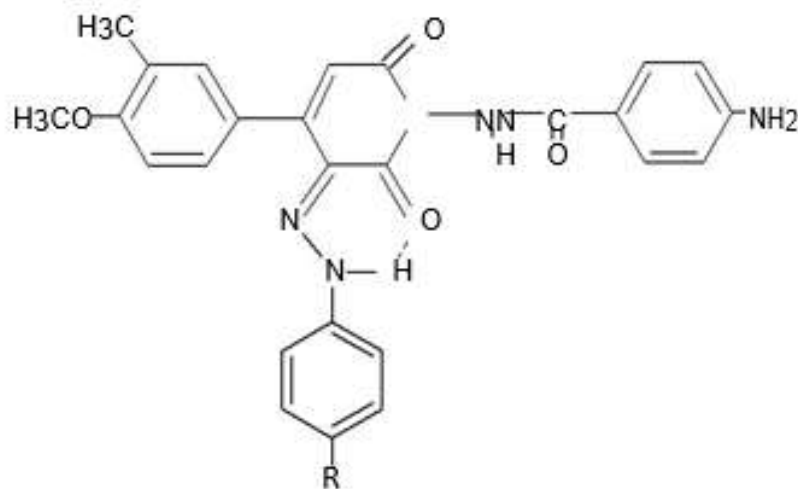


Table 1: The characterization data of the dyes thus prepared is depicted in table

Compound No.	R	Yield %	M.P. (°C)	Molecular Formula	Elemental Analysis	
					Nitrogen %	
					Calculated	Found
28 A	NO <sub>2</sub>	65	256-260	C <sub>26</sub> H <sub>22</sub> N <sub>6</sub> O <sub>6</sub>	16.34	16.37
28 B	Cl	62	263-265	C <sub>26</sub> H <sub>22</sub> ClN <sub>5</sub> O <sub>4</sub>	13.90	13.94
28 C	C <sub>8</sub> H <sub>7</sub> NS	68	283-286	C <sub>34</sub> H <sub>28</sub> N <sub>6</sub> O <sub>4</sub> S	13.63	13.68
28 D	CH <sub>3</sub>	64	251-255	C <sub>27</sub> H <sub>25</sub> N <sub>5</sub> O <sub>4</sub>	14.49	14.42
28 E	OCH <sub>3</sub>	67	280-283	C <sub>27</sub> H <sub>25</sub> N <sub>5</sub> O <sub>5</sub>	14.02	14.04

### III. RESULT AND DISCUSSION

Table 2: Visible spectral data and dyeing properties of dyes w(a – e) are shown in table

Dye No.	Colour of Dye on polyester fabric	Absorption maxima □ λ <sub>max</sub> (nm)	Log ε	Pick - up	Xeno □ □	Thermo □ □
A	yellow	435	4.143	4	2 - 3	4
B	Orange	450	4.205	5	2 - 3	4
C	Orange	458	4.281	5	3	4
D	Reddised Orange	469	4.271	5	2 - 3	4
E	Reddised Orange	480	4.290	5	2 - 3	4

where

- \* - Recorded in DMF – Methanol ( 1 : 99 )
- ♣ - For light fastness
- ⊕ - Sublimation fastness at 200°C for one min.

Monoazo dyes W(a-e) showed an absorption ranging from 435 nm to 480 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 4.143 to 4.290. The pick-up value for all dyes is found to be 5. The results for xeno test and thermo test lies in the range 2-3 and 4 respectively. The most of the dyes of these series showed moderately high absorption intensities [7,8].

### IV. CONCLUSION

Various 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.

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