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Synthesis and effect of Actamido Phenyl Azo 2-6 Pyridinedione Based Dye on Polyester Fiber

A. Gour

Department of Chemical Science, Christian Eminent College, Indore, India

Corresponding Author: dranitagour@gmail.com, Tel.: +91-0731-4213665

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***Abstract*-** In order to prepare a unique hue in disperse azo dyes synthesized by the coupling component of diazonium salt 1-[4-(2-Acetamido-4-N,N-diethylamino) phenylazo] phenyl-4-(4-methoxy-3-methylphenyl)-2,6-(1H,5H)-pyridinedione to produce the corresponding different azo disperse dyes (A- E). These dyes were consumed to polyester fabric and its effect was evaluated.

Keywords **-** Disperse azo dyes, Diazonium salt , XENO test, Thermo Test, Tinctorial Power

I. INTRODUCTION

Disperse dyes were industrialized for dyeing of acetate fibres and also used for dyeing of Nylon and acrylic fibres. With the addition of swelling agents these dyes are also used in dyeing of Polyester like Terylene, Tetron, Dacron, etc. After second world war massive changes occurred in textile industry and a new generation fibres were introduced including triacetate, polyamide, polyacrylonitrile, polyester & elastane fibres.[1]

Traditionally, azo dyes are the utmost significant class of marketable dyes, occupying other than half of the dye chemistry, which comprises phenols as intermediates. Thus, the present work shows the formation of some novel disperse azo dyes synthesis by the coupling component of diazonium salt with 1-(4-Aminobenzamido)-4-(4-methoxy-3-mehylphenyl)-5-(4-nitrophenylhydrazono)-2,6-(1H)-pyridinedione to give the equivalent numerous azo disperse dyes (a-f) [2].

II. MATERIALS AND METHODS:

Preparation of 3-(4-Methoxy-3-methylphenyl)-2-pentene-1,5-dioic acid X:
To the acetone di-carboxylic 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^oC over duration of 1 hour. Stirring was further continued, for duration of 3 hours and maintain the temperature at 0-5^oC. Then 150ml cold water is were poured in the content, 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]

Melting Point : 161-162^{0}C Yield : 180 gm (32 %)
Elemental Analysis:
Found: C, 62.46 ;H, 5.62
Calc. For $C_{13}H_{14}O_5$: C, 62.40 ; H, 5.60

Preparation of 1-(4-Acetamidophenyl)-4-(4-methoxy-3-methylphenyl)-2,6-(1H,5H)-pyridinedioneY :
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 1700C for 30 minutes. The fused mass was cooled to room temperature and treated with aqueous solution of 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^{0}C Yield : 32.7 gm (90%)
Elemental Analysis :
Found : C,69.19 ;H, 5.52 ;N, 7.65
Calc. For $C_{21}H_{20}N_2O_4$: C,69.23 ; H,5.49 ;N,7.69

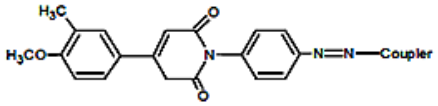
Preparation of 1-(4-Aminophenyl)-4-(4-methoxy-3-methylphenyl)-2,6-(1H,5H)-pyridinedione Z :
Compound Y (8 gm, 0.02 mole) was refluxed in hydrochloric acid : mixture of acetic acid (20 : 5ml) for 2 hours. The reaction of mixture was cooled and poured into ice water. It was neutralized with dilute solution of sodium hydroxide, white colored solid separated out. The solid was filtered and washed several times with water onwards dried. It was crystallized from ethyl alcohol [**5**].
Melting point : 225 – 229^{o}C Yield : 5.3 gm (75%)
Elemental Analysis :
Found : C, 70.86 ;H, 5.62 ;N, 8.72
Calc. For $C_{19}H_{18}N_2O_3$: C, 70.80 ;H, 5.59 ;N,8.69

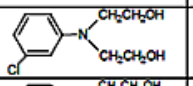
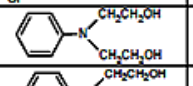
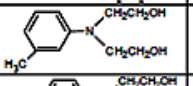
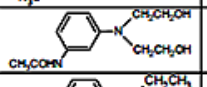
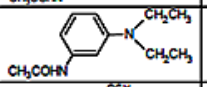
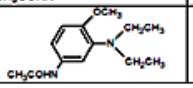
Preparation of 1-[4-(2-Acetamido-4-N,N-diethylamino) phenylazo] phenyl-4-(4-methoxy-3-methylphenyl)-2,6-(1H,5H)-pyridinedione :

A solution of Z (0.322 gm, 0.001mole) in propionic acid (1ml) and glacial acetic acid (5ml) was added drop wise with stirring at period of 45 minutes to a cold (50C) mixture of nitrosyl sulphuric acid [prepared by dissolving solid sodium nitrite (0.069gm, 0.001mole) in conc. sulphuric acid (8ml) at 70^{o}C]. The mixture was stirred for an additional period of 2 hours at 5-10^{o}C. The mixture was then added to propionic acid 2ml and acetic acid mixture 10ml under stirring. The extra nitrous acid was destroyed using 0.2gm urea. The mixture was filtered to get a clear diazonium salt solution. 3-Acetamido-4-N,N-diethylaniline (0.206gm, 0.001mole) was dissolved in propionic acid 4ml and acetic acid 20ml mixture. The resultant was externally cooled to 5^{0} C.[6] The previously cooled diazonium salt solution was slowly added to the above mixture maintained at 5-10^{o}C over 1 hour. Throughout the coupling period by addition of solid sodium acetate, the pH was maintained acidic (4-5). After addition of diazonium salt , 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-toluene mixture.[7]

- Melting point : 232-237^{o}C Yield : 0.296gm (55%)
- Elemental Analysis :
- Found : Nitrogen 12.92%
- Calc. For $C_{31}H_{33}N_5O_4$: Nitrogen 12.98%

All the other dyes (a-f) were synthesized by using the same method. The characterization data of the dyes prepared is depicted in table below [8]:



Compound No.	Coupler	Yield %	M.P. (^{0}C)	Molecular Formula	Elemental Analysis Nitrogen %	
					Calculated	Found
a		54	202-205	$C_{29}H_{29}ClN_4O_5$	10.20	10.25
b		57	212-217	$C_{29}H_{30}N_4O_5$	10.89	10.85
c		60	231-235	$C_{30}H_{32}N_4O_5$	10.60	10.64
d		53	224-228	$C_{31}H_{33}N_5O_6$	12.25	12.21
e		55	232-237	$C_{31}H_{33}N_5O_4$	12.98	12.92
f		59	246-250	$C_{32}H_{35}N_5O_5$	12.30	12.36

III. RESULTS AND DISCUSSION

EVALUATION OF DYES – TERMS AND RATINGS :

A. Terms of pick-up (Tinctorial Power)

The pick-up values were determined by comparing the dyeing with standard dyeing (Hifstypen standard depths)

5 = 1 x standard depth (excellent)

4 = 1/3 x standard depth (very good)

3 = 1/4 x standard depth (good)

2 = 1/6 x standard depth (moderate)

1 = 1/12 x standard depth (poor)

B. Terms for light fastness (Xeno test)

Light fastness was evaluated by exposing a part of dyed fabric to high energy radiation (Xenon arc lamp) in a fade-o-meter and compared with undyed fabric.

Rating	Grade	Rating	Grade
8	Outstanding	4	Fairly good
7	Excellent	3	Fair
6	Very Good	2	Poor
5	Good	1	Very poor

C. Terms for sublimation fastness (Thermo test)

Sublimation fastness was determined by keeping a composite specimen of dyed polyester and undyed polyester pieces in a precision press at 200^{0}C for 1 minute.

Rating	Grade
5	Excellent
4	Good
3	Fair
2	Poor
1	Very Poor

VISIBLE SPECTRAL DATA AND DYEING PROPERTIES OF DYES {a – f}

Dye No.	Colour of Dye on polyester fabric	Absorption maxima * λ max (nm)	Log ε	Pick - up	Xeno♣	Thermo Θ
a	Pale Golden Yellow	440	4.033	2 - 3	4	4
b	Pale Golden Yellow	443	4.056	2 - 3	4	4
c	Pale Golden Yellow	446	4.085	2 - 3	4 - 5	4 - 5
d	Golden Yellow	451	4.106	2 - 3	4 - 5	4 - 5
e	Reddish Orange	453	4.108	3	4 - 5	4 - 5
f	Reddish Orange	455	4.113	3	4 - 5	5

* –Recorded in DMF – Methanol (1 : 99)

♣ – For light fastness

Θ –Sublimation fastness at 200^{0}C for one minute

IV. CONCLUSION

The pyridone skeleton as diazo component and different N,N-dialkylsubstitutedaniline derivatives as couplers of monoazo dyes a-f, showed an absorption range 408 nm to 435 nm and 440 nm to 455 nm respectively. It was observed that the value of the absorption maxima increased with increasing number of electron donating substituents on dyes. In these chromophore systems coupler serves as donor end, while the pyridone skeleton is the acceptor end. Monoazo dyes a-f showed absorption intensity in the range of 4.01 to 4.10 and 4.03 to 4.11 respectively. Most of the dyes of these series demonstrated moderately high absorption intensities [9].

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VI. REFERENCES

[1]. MF. Comelo "*Coloration Technology*", Willey, Vol.32, Issue.1, pp.1–12, 2002

[2]. J. Shore, "*Blends Dyeing*", SDC, Bradford, pp.10–19, 1998.

[3]. HS. Freeman, J. Sokolowska, "*Developments in dyestuff chemistry from Book* "Coloration Technology, Vol.29, Issie.1, pp.8-22, 1999.

[4]. H Toshio , H Inoue , W Seto, *"Yellow disperse dye mixture which has a high level of light fastness*", Dystar Textilfarben Gmbh & Co Deutschland Kg, UA, pp.1-29, 2006.

[5]. DM. Lewis, PJ. Broadbent, "*A universal dye for all fibres - are disperse dyes capable of fulfilling this vision?",* Journal of the Society of Dyers and Colourists, Vol.113, Issue.5, pp.159-64,1997.

[6]. AI. Vogel, "*A Text-Book of Practical Organic Chemistry*", Longman Scietific and Technical, Englend, pp.1-1000, 1948.

[7]. J. W. COOK, "*A Text-Book of Practical Organic Chemistry*", Nature, Vol.161, pp.995-995, 1948.

[8]. Lecher, "The Chemistry of Synthetic Dyes and Pigments". Journal of the American Chemical Society, Vol.77, Issue.22, pp.6089-6089, 1995.

[9]. D. W. Rangnekar, P.Y. Kamath, "*synthesis of 3-amino-2-arylazo-4,6 dimethyl thieno[2,3]pyridines and their application on polyester fibres as disperse dyes*", Indian Journal of Fiber and Textile Research,Vol.17 pp.98-100, 1992.

AUTHORS PROFILE

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 Assistant Professor in Department of Chemical Science at Christian Eminent College Indore from Aug 2011.

