International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 08 Issue: 05 | May 2021 www.irjet.net p-ISSN: 2395-0072

Synthesis of azo disperse dyes benzene-1,4-bis(diazonium) derivatives and dyeing with polyester

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Abstract - *In this work we have synthesized four derivatives of benzene-1,4-bis(diazonium) by coupling of various naphthols with diazonium salt of p-phenylenediamine. The experimental procedure was simple and had a high reaction rate and excellent yield. These synthesized benzene-1,4-bis(diazonium) dyes were studied for dyeing on polyester fabric. The fastness results of dyed polyester were good to better comparable with that of commercial dye.*

Key Words: **azo disperse dyes; benzene-1,4-bis(diazonium) derivatives; polyester dyeing**

1. INTRODUCTION

Azo dyes represented the major production in terms of volume of dye chemistry today and the importance of them may rise in the future. These dyes were synthesized from a simple method of diazotization and coupling reaction [1]. Approximately 70% of the all the dyes used in the textile industries are azo dyes out of which 4% of them are found to be carcinogenic [2,3]. Overall, the chemical structure of an azo dye comprises of auxochromes, chromophores, solubilizing groups and the main back bone [4-8]. Azo dyes contain at least one nitrogen-nitrogen double bond (N=N); though numerous diverse structures are possible [9].

The dyeing of polyester material is a big worry to the industries in many countries around the globe. PET (Polyethylene terephthalate) fibres have a widespread range of application and are chemically resistant. However, the dense crystalline structure of polyethylene terephthalate fibres was difficult to dye with many dye types excluding disperse dyes [10]. Previously azo dispersed dyes were synthesized by using pyridine [11], dimethylamino –p-arylpropenones [12] and urethane containing groups [13].

Azo disperse dyes were also synthesized by using amino benzaldehyde and different naphthols and admirable fastness ratings were observed [14]. Disperse dyes synthesized using aromatic hydroxyl group and weakly basic amines also give promising colour fastness results [15].

On the basis of these reports in the present work we synthesized four new benzene-1,4-bis(diazonium) derivatives and studied their dyeing behaviour with polyester using HTHP (High Temperature High Pressure) dyeing method. The dyeing performance on the resultant fabric was evaluated by studying the fastness properties.

2. EXPERIMENTAL SECTION

2.1 Materials

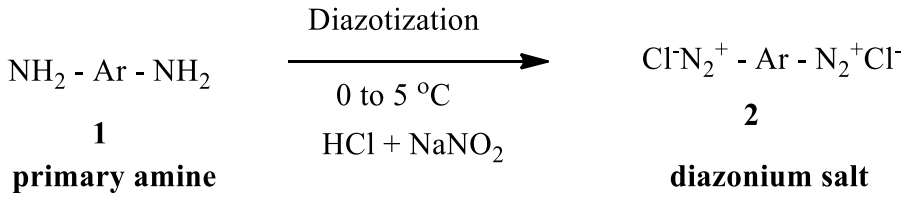
Different naphthols namely Naphthol AS-D, Naphthol AS, 1-naphthol and 2-naphthol were gifted by Department of Textiles, D.K.T.E.S's Textile and Engineering Institute, Ichalkaranji, 416 115 M. S. (India) . Para-phenylenediamine was purchased from Sigma Aldrich with 98% purity. All the solvents and other chemicals were of laboratory grade and purchased from Spectrochem, s. d. Fine chemical Limited (India). These chemicals were used as such exclusive of additional purification. which included sodium nitrite, hydrochloric acid, chloroform and methanol etc.

2.2 Methods

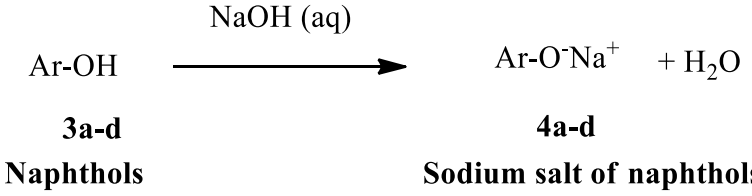
Melting points were determined in an open capillary and are uncorrected. Infrared spectra were recorded on Perkin Elmer FT-IR spectrometer FT-IR Spectra were recorded with standard light source at 45° incident angle at room temperature. Nomenclature of synthesized derivatives was done by using software ChemBioDraw ultra, version 12.0 of Cambridgesoft. Dyeing was carried out using Mathis labomat. Various fastness tests were carried out which includes color fastness to light on standard light fastness tester, color fastness to sublimation on the instrument called as sublimation fastness tester, color fastness to perspiration using perspirometer, rubbing fastness on crockmeter and washing fastness on laundrometer.

In present work new benzene-1,4-bis(diazonium) derivatives synthesized by coupling of various naphthols with diazonium salt of p-phenylenediamine. The synthesis of new derivatives of dyes involves three main steps,

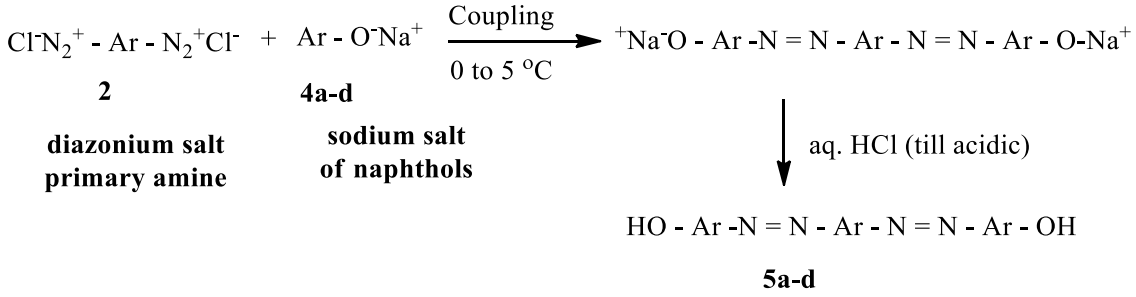
Step- 1: Preparation of benzene-1,4-bis(diazonium) derivatives from primary amine via diazotization.
Step- 2: Preparation of salt solution of naphthol.
Step- 3: Coupling of diazonium derivative of primary amine with naphthol.



Step -1 Preparation of benzene-1,4-bis(diazonium) derivatives from primary amine via diazotization.



Step -2 Preparation of salt solution of naphthol.



Step -3 Coupling of diazonium derivative of primary amine with naphthol.

2.3 General procedure for synthesis of 1,1'-(1,4-phenylenebis(diazene-2,1-diyl))bis(naphthalen-2-ol) (5a)

***Step 1 Procedure of synthesis of benzene-1,4-bis(diazonium) derivative (2)*:**
2.16 gm (0.02 mol) para-phenylenediamine taken in 250 ml beaker and to this 50 mL aq. 1:1 HCl solution is added to solubilize it. This solution further cooled to 0 to 5 ℃. After attaining the required temperature 4 gm (0.05 mol) NaNO2 was added very slowly maintaining 0 to 5 ℃ temperature. Brown fumes were released during addition of NaNO2.

***Step 2 Procedure of synthesis of sodium naphthalen-1-olate (4a)* :**
5.76 gm (0.04 mol) 1-Naphthol was dissolved in 50 mL 10 % aq. NaOH solution (if required warm and then cool and filtered through Whatman filter paper). Further the solution of naphthol cooled to maintain the temperature 0 to 5 ℃.

***Step 3 Procedure of synthesis of 2,2'-(1,4-phenylenebis(diazene-2,1-diyl))bis(naphthalen-1-ol) (5a)* :**
The cold solution of benzene-1,4-bis(diazonium) derivative (2) was slowly added to cold solution of sodium naphthalen-1-olate (4a) very slowly maintain the reaction temperature 0 to 5 ℃ over the period of 30 min. Then excess of 1:1 aq. HCl was added to make the pH acidic. The product (5a) was isolated by simple filtration. Dried the crude product at room temperature in shadow. The crude product recrystallized with 95 %.

The same procedure was used to synthesis the other three dye derivatives product 5b-c (table 1). The prepared product (5a) was first applied on cotton by exhaust method dyeing was carried out but the results were not satisfactory. Hence the product (5a) examined for disperse dyeing method. The appearance of dyed fabrics in day light are shown in table 2.

2.4 General Procedure for Disperse Dyeing

The polyester fabric was taken from Ratanmoti tex fab of 82 GSM having 170 and 80 EPI and PPI respectively and dye was applied through HTHP method by mathis labomat which included preparation of 1% disperse dye by weighing exactly 1gm of dye powder and pasting it with small quantity of dispersing agent and little amount of acetic acid. Small quantity of warm water is added and paste is prepared. Then volume is made up to 100ml by addition of water. Afterwards dyeing is carried out by setting the dye bath with 1:20 MLR and maintaining 4.5-5.5 pH by adding acetic acid. Then fabric is added at room temperature and worked for 10 minutes. Then temperature rises to 130°C in 60 minutes. At this temperature dyeing is carried out for 30 minutes. After this bath is cooled down to 80°C. Fabric is removed and rinsed properly with cold water followed by reduction clearing with 2gpl sodium hydrosulphite, 4cc/Lt. NaOH at 70°C and later soaping with 1.5gpl soap at boil for 10 minutes. This dyeing method gave satisfactory dyeing, so we proceeded with polyester for further dyeing and testing.

3. RESULTS AND DISCUSSIONS

We have synthesized four derivatives 5a-d. The product 5a and 5b are naphthol derivatives and required time of reaction completion was less than 70 minutes, however the substituted derivatives 5c and 5d required more than 100 minutes for completion of reaction. The product 5d have melting point 195 ℃ while other three derivatives 5a-c are having the melting point less than 100 ℃ (table 1).

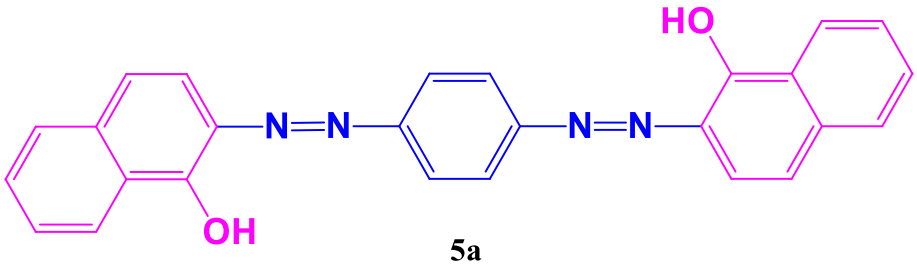
Table 1 Synthesis of benzene-1,4-bis(diazonium) derivatives. [a]

Product	Time (min.)	Observed Melting point (℃)	Yield [a] (%)
5a	65	87	92
5b	60	86	90
5c	120	97	91
5d	120	195	93

a = isolated yield of crude product.

The structures of isolated products and images of dyed polyester fabric samples are tabulated below (table 2). The first two derivatives 5a and 5b showed the light brown colored dyeing of polyester fabric, while the product 5c and 5d showed purple color dyeing on polyester fabric.

Table 2 Dye structure and color appearance of fabric after dyeing

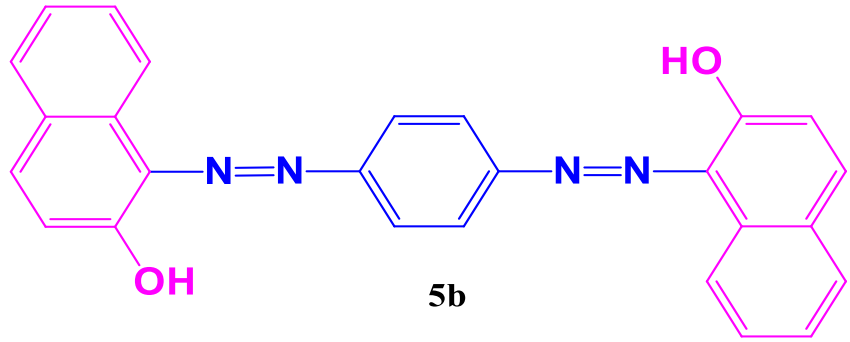


5a

2,2'-(1,4-phenylenebis(diazene-2,1-diyl))bis(naphthalen-1-ol)

1 gpl	
2 gpl	

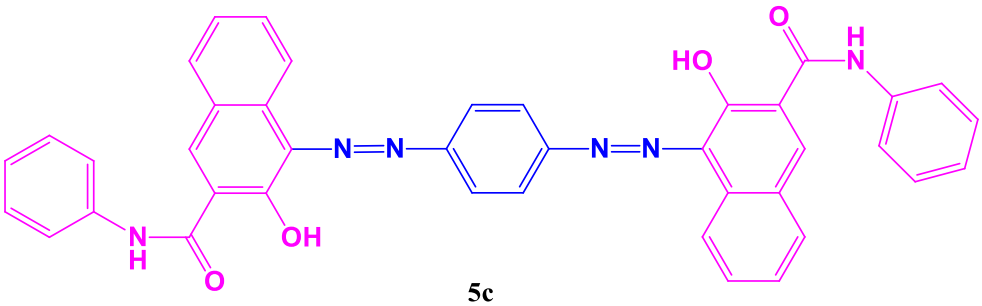
	



5b

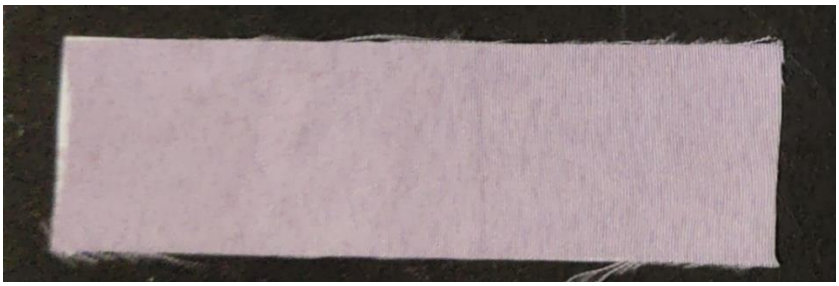
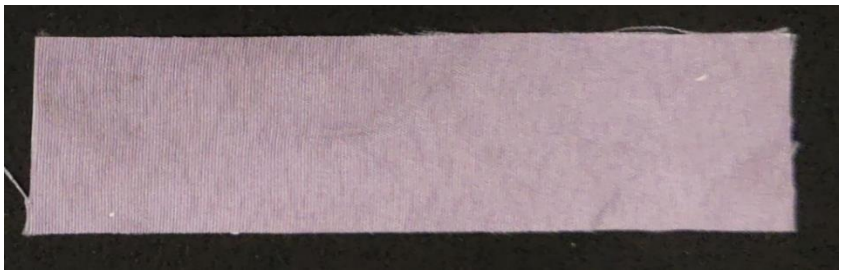
1,1'-(1,4-phenylenebis(diazene-2,1-diyl))bis(naphthalen-2-ol)

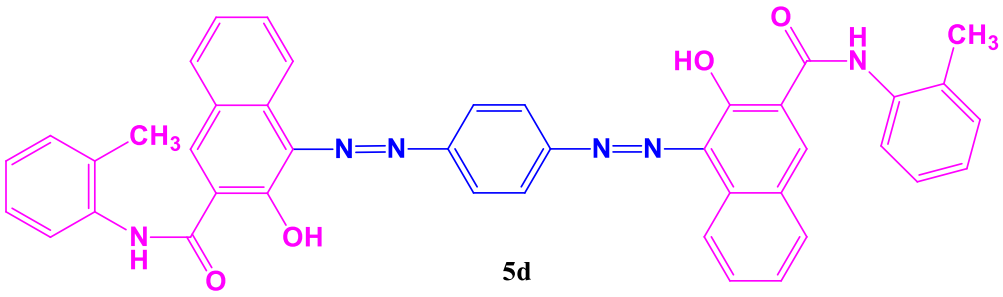
1 gpl	
2 gpl	



5c

4,4'-(1,4-phenylenebis(diazene-2,1-diyl))bis(3-hydroxy-*N*-phenyl-2-naphthamide)

1 gpl	
2 gpl	



5d

4,4'-(1,4-phenylenebis(diazene-2,1-diyl))bis(3-hydroxy-*N*-(*o*-tolyl)-2-naphthamide)

1	

gpl	
2 gpl	

3.1 Color Fastness Tests

3.1.1 Color Fastness to Washing

Color fastness to washing was determined by using the test method ISO 105 C05, after carrying out the test we found that all the synthesized dye i.e. dye 5(a-d) shows excellent washing fastness (table 3).

3.1.2 Color Fastness to Perspiration

The test for perspiration fastness was carried out based on acidic and alkaline media in accordance with test method ISO 105 E 04, after carrying out the test we found that all the dyes exhibit excellent acidic perspiration fastness with rating 5 and moderate to excellent alkaline perspiration fastness with rating 4-5 (table 3).

3.1.3 Color Fastness to Crocking (Rubbing)

The crocking fastness to rubbing of the dyed samples was carried out according to test method ISO 105 X12, after carrying out the test it is found that the dry rubbing fastness is slightly poor than the wet rubbing fastness. Among all the dyes; dye 5a (1%), 5c (1%), 5d (1%), 5d (2%) show excellent wet rubbing fastness (table 3).

Table 3 Washing, perspiration and rubbing fastness of the synthesized dye

Dye	Shade (%)	Washing	Perspiration		Rubbing	
			Alkali	Acidic	Dry	Wet
5a	1	5	5	5	4-5	5
	2	5	4	5	4	4-5
5b	1	5	4-5	5	4-5	4-5
	2	5	5	5	4-5	4-5
5c	1	5	4-5	5	4-5	5
	2	5	4-5	5	4-5	4-5
5d	1	5	5	5	4-5	5
	2	5	5	5	4-5	5

3.1.4 Color Fastness to Sublimation

The test for the sublimation fastness is carried out according to test method ISO 105 P01, after carrying out the test we found that the dye 5(c-d) show excellent sublimation fastness in both the concentration. We also found out that the dye 5(a-b) show poor sublimation fastness at 210 °C*in both the concentration. (table 4)*

3.1.5 Color Fastness to Light

The test for light fastness was assessed using the test method ISO 105 BO2, after carrying out the test we have found that the all the dyes exhibit poor light fastness with rating 3-4 and among those dyes dye 5b with 1% shade exhibit fastness rating 2-3 (table 4).

Table 4 Sublimination and light fastness of the synthesized dyes

Dye	Shade (%)	Sublimation			Light
		150 ℃	180 ℃	210 ℃	
5a	1	5	4-5	3	3-4
	2	5	4	2-3	3-4
5b	1	4-5	4	2-3	2-3
	2	4-5	4	2-3	3-4
5c	1	5	5	5	3-4
	2	5	5	5	3-4
5d	1	5	5	5	3-4
	2	5	5	5	3-4

4. CONCLUSION

The study described the synthesis and application of disperse-azo dyes onto PET fibers. The dyes showed outstanding characteristics on textile substrates and washing fastness properties. There was little variation in rubbing and perspiration fastness properties.

ACKNOWLEDGEMENTS

We gratefully acknowledge financial support from the Shivaji University, Kolhapur-416004, (MS), India for granting us the research project under Research Initiation Scheme 2018. Ref: SU/C&U.D.Section/26/174; Dated 15th May 2018 and Department of Textiles, D.K.T.E.S's Textile and Engineering Institute, Ichalkaranji, 416 115 M. S. (India)

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