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**ORIGINAL ARTICLE**

Synthesis and dyeing performance of some novel thiazole azo disperse dyes

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Fastness

Abstract The synthesis of some novel monoazo disperse dyes derived from 5-acetyl-2-amino-4-methylthiazole using various N-alkyl derivatives of aniline and their dyeing performance as disperse dyes have been assessed on cellulose triacetate fabric. The spectral properties of these dyes were also measured. The dyed fabric show good light fastness, very good rubbing, perspiration, washing fastness and excellent sublimation fastness. These dyes have been found to give bright yellow to maroon color shade with very good depth and levelness on fabric. The dyebath exhaustion and fixation on fabric has been found to be very good.

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1. Introduction

Color Index describes various basic, direct, vat and disperse dyes wherein thiazole nucleus occurs. Early interest in heterocyclic diazo components was stimulated by the need for blue disperse dyes with improved dischargeability and gas fastness (oxide of nitrogen) for cellulose acetate. Before 1950, almost all the disperse dyes used were prepared from the anthraquinone class and had the limitations of poor dischargeability and sensitivity to oxides of nitrogen. Red and blue azo dyes

with good dischargeability for use in printing were developed. In fact investigation of the use of heterocyclic diazo components was stimulated by the discovery of Dickey of Eastman Kodak in 1950. In 1953, they prepared bright blue heterocyclic azo disperse dyes (Dickey and Towne, 1953) from 2-amino-5-nitrothiazole with adequate light fastness combined with excellent dischargeability on cellulose acetate. The commercial successes of such bright blue azo disperse dyes for cellulose acetate showing considerable interest in this class of dyes. The derivatives of 2-aminothiazole have played a significant role in the technology of disperse dyes. They have been studied owing to the marked bathochromic effect when compared with the corresponding benzenoid compounds (Weaver and Shuttleworth, 1982).

In the early days of the dyestuff industry, the majority of dyes were derived from benzene and naphthalene derived intermediates, including heterocyclic systems such as Mauveine (Meth-Cohn and Smith, 1994) are as old as the field itself, or even antedate it in cases like Indigo (Grozinger, 1996). Azo dyes with heterocyclic diazo components have been intensively

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investigated to produce bright and strong color shades ranging from red to greenish blue on synthetic fabrics. In addition to the high tinctorial strength generally characteristic of azo dyes, the brilliance of their dyeing can be increased in many cases by replacement of the aniline diazo component by a heterocyclic amine. Since 1980 a further interest has been shown in the disperse dyes derived from 2-aminothiazole derivatives (Towne and Hill, 1955; Merian, 1961; Tsien et al., 1997; Nakamura and Murata, 1993).

These dyes have very low water solubility and possess substantivity for hydrophobic fabrics. Bright blue heterocyclic azo disperse dyes prepared from 2-amino-5-nitrothiazole with adequate light fastness combined with excellent dischargeability, gas fastness and dyeability on cellulose acetate. The commercial success of such dyes for cellulose acetate which were derived from 2-amino-5-nitrothiazole resulted in considerable interest in this class of dyes. Early dyes showed moderate light fastness on cellulose acetate and polyesters, and light fastness on polyamide was very poor. Also, on polyesters the dyes had poor sublimation fastness for the thermofixation method of dyeing and showed loss of color under high temperature exhaust dyeing conditions, particularly at high pH values. Dyes with increased light fastness and/or sublimation fastness were prepared from aniline type coupling components containing one or more N-alkyl groups substituted with groups such as cyano (Gosei Senryo Gijutsu Kenkyu Kumiai, 1983a), amyl carbonyloxy (Gosei Senryo Gijutsu Kenkyu Kumiai, 1983b), dicarboximide (Weaver and Straley, 1970), alkoxy (Mitsubishi Chemical Industries Co. Ltd., 1979), pyridinium (Parton, 1978), sulphato (Sumitomo Chemical Co. Ltd., 1981), diphenylamine (Erwin et al., 1991), 1-naphthylamine (Modena and Risaliti, 1977) and N-alkyl-1,2,3,4 tetrahydroquinolines (Gosei Senryo Gijutsu Kenkyu Kumiai, 1984) were used as coupling components. These coupler types have the advantages of providing dyes, which have a neutral or green cast when exposed to artificial light, whereas the corresponding dyes from N-alkylaniline couplers have an extremely red cast. Greenish blue dyes can be prepared by choosing a coupler containing electron donating groups such as 5-acetamido-2-ethoxyaniline derivatives. Additional early work on the 2-thiazolylazo dyes resulted in violet dyes from 2-amino-4-trifluoromethyl-5-carbalkoxythiazole (Towne and Hill, 1956), 2-amino-5-formylthiazole (Egli, 1979) and even more bathochromic dyes from 2-amino-4-alkylsulphonyl-5-nitrothiazole (Towne et al., 1958).

During the last fifteen years, level of interest as indicated by the patent literature has grown in the field of thiazolylazo disperse dyes (Nakamura and Murata, 1990; Noack et al., 1990; Tsien et al., 1995; Beckmann et al., 1994, 1995; Sens and Etzbach, 1994; Matsui and Ishida, 2001). There has been a considerable growth in the volume of data reporting outside of patent literature (Sekar, 1994; Peters and Gbadamosi, 1995; Bello et al., 1995; Desai et al., 1995; Bello, 1995; Keil and Hartmann, 1995; Liu et al., 1995; Griffiths and Riepl, 1998; Chikhalia and Desai, 1998; Rangnekar et al., 1998; Towns, 1999; Hallas and Choi, 1999; Rangnekar and Malankar, 2000; Elkholly et al., 2001; Helal, 2001; Maradiya and Patel, 2000; Maradiya and Patel, 2001a,b,c,d; Maradiya and Patel, 2002a,b; Maradiya, 2002; Georgiadou and Tsatsaroni, 2002; Sonawane and Rangnekar, 2002; Maradiya and Patel, 2003; Maradiya, 2004a,b). It seems that no report is available on the use of 5-acetyl-2-amino-4-methylthiazole in the synthesis of disperse dyes by coupling with various N-alkylaniline derivatives.

Hence, it was thought worthwhile to synthesize a series of disperse dyes from 5-acetyl-2-amino-4-methylthiazole **1** using various substituted N-alkyl-N-(2-hydroxyethyl)aniline derivatives **2a-e** as coupling components. The resulting dyes were applied to cellulose triacetate and evaluation of their technical properties and a color assessment were performed.

2. Experimental

2.1. Materials and methods

All the chemicals and coupling components used were of commercial grade and were further purified by recrystallization and redistilled before use. The solvents used were spectroscopic grade. Melting points were determined by open capillary method. The visible absorption spectra were measured in a Carl Zeiss UV/VIS Specord spectrometer. The elemental analysis was carried out on a Carlo Erba Elemental Analyser 1108. Infrared spectra were recorded on a Perkin-Elmer model 983 spectrometer scanning between 4000 and 400 cm^{-1} using a KBr pellets. Purification of the products was carried out by TLC (1 mm thickness glass plates 20×5 cm, using suspension of silica gel G in CHCl_3). Fastness to light, sublimation and perspiration were assessed in accordance with BS:1006-1978. The wash fastness test was carried out in accordance with IS:765-1979. The rubbing fastness test was carried out using Crockmeter (Atlas) in accordance with AATCC-1961. The detail of the method of color fastness tests was described in the literature (Maradiya, 2002). The dyeing of cellulose triacetate was carried out according to method described in the literature (Maradiya and Patel, 2001). The percentage dyebath exhaustion (%E) and percentage dyebath fixation (%F) of the dyed fabric was determined according to the known method (Maradiya, 2002).

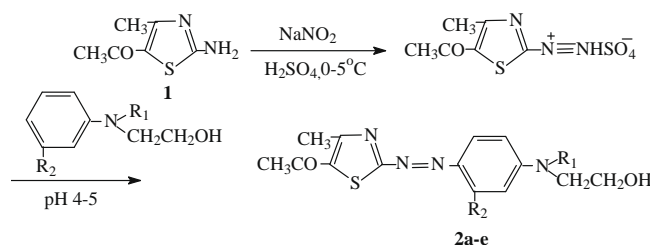
2.2. Preparation of 5-acetyl-2-amino-4-methylthiazole **1**

The title diazo compound was synthesized by the method reported in the literature (Kochergin, 1956).

2.3. Preparation of dye **2a**

Diazotization of 5-acetyl-2-amino-4-methylthiazole **1** was effected with nitrosylsulphuric acid as follows.

Dry NaNO_2 (1.38 g, 0.02 mol) was slowly added with stirring to concentrated H_2SO_4 (1.1 ml) with stirring while allowing the temperature to rise to 65 °C. The solution was then cooled to 5 °C and 20 ml of a mixture of acetic acid and propionic acid (17:3) was added dropwise with stirring, allowing



Scheme 1

Table 1 Absorption maxima, intensities, characterization data, exhaustion and fixation of dyes.

Dye no.	Substituents		Melting point (°C)	Yield (%)	Absorption maxima		log ϵ (DMF)	(%E)	(%F)	R_f value
	R ₁	R ₂			λ_{\max}/nm (DMF)	λ_{\max}/nm (Conc. H ₂ SO ₄)				
2a	H	H	208–209	82	498	460	4.13	82	88	0.81
2b	CH ₃	H	215–216	85	510	460	4.80	83	90	0.79
2c	C ₂ H ₅	H	192–194	81	508	465	4.82	82	91	0.74
2d	H	CH ₃	186–188	80	510	470	4.83	84	89	0.80
2e	H	Cl	198–199	84	495	475	4.56	83	90	0.92

the temperature to raise 15 °C, and the diazo component 5-acetyl-2-amino-4-methylthiazole **1** (3.12 g 0.02 mol) was added portionwise and stirring was continued at this temperature for 2 h. The excess nitrous acid (the presence tested by starch-iodide paper) was decomposed with the required amount of urea. The clear diazonium salt solution thus obtained was used immediately in the coupling reaction (Scheme 1).

2.4. General coupling procedure

The N-(2-hydroxyethyl)aniline (2.74 g, 0.02 mol), was dissolved in 10 ml of acetic acid, then cooled in an ice-bath at 0 °C. The diazonium solution previously prepared was added dropwise over 1 h with vigorous stirring at 0–3 °C. The reaction mixture was stirred for further 2 h at 0–5 °C, and then 10% sodium acetate solution was dropped in slowly until the pH became 4–5. The product was then filtered off, washed with water until acid-free, dried at 50 °C in an oven to give azo dye **2a**, which was recrystallized three times from ethanol (4.98 g, 82%). The m.p. 208–209 °C. The characterization data are given in Table 1.

The same procedure was repeated for the preparation of the dye **2b–e**. The synthetic scheme is shown in Scheme 1.

3. Results and discussion

3.1. Preparation of dye 2

5-acetyl-2-amino-4-methylthiazole **1** was diazotized satisfactorily at 0–5 °C by nitrosyl sulphuric acid in acetic acid. In order to determine the end point of diazotization, it was found useful to check for the presence of unreacted diazo component on thin layer chromatography (TLC), by sampling the diazotization mixture. Thus, when unreacted diazo component no longer persisted on TLC, the diazotization ended. The diazonium salt solution was used immediately since this decomposed on standing, even when cold. Subsequent coupling reactions took

place readily upon the addition of the diazonium salt continuously to the solution of the coupling component in acetic acid. Coupling was usually accompanied by some evidence of decomposition; however, 80–85% yield of the dye were usually obtained by careful addition of the diazonium salt solution at 0–3 °C to a solution of the coupling component in an acetic acid. To complete the coupling particularly for reaction using nitrosylsulphuric acid in the previous diazotization, the pH of the reaction mixture was adjusted to approximately 4–5. Thus, an appropriate amount of 10% sodium acetate solution was slowly added below 5 °C.

3.2. Physical properties of dyes

The purity of the dyes was checked by TLC using methanol:water:acetic acid (12:3:7 v/v) as the solvent system. When adsorbed onto silica chromatography plates, the dyes produced a single orange-red color spot. All the recrystallized dyes exhibited well-defined melting points characteristics of pure compounds but it would be unwise to attempt to explain in detail their relative values because of the complex dependence of the melting points on a number of factors (e.g. polarity, size, geometry, molecular interactions etc.). The characterization data are given in Table 1. Elemental analysis data are given in Table 2.

3.3. Spectral properties of dyes

The adsorption maxima λ_{\max} of dyes **2a–e** were recorded in concentrated H₂SO₄ and DMF solution and are shown in Table 1. As far as absorption maxima are concerned, λ_{\max} values are directly proportional to the electronic powder, the nature and position of the substituent in the phenyl ring of coupler moiety. The value of the logarithm of molar extinction coefficient (log ϵ) of the dyes **2a–e** were in the range of 4.13–4.83, consistent with their high intensity of absorption. One cause of the increased intensity might be attributed to the greater

Table 2 Elemental analysis of dyes.

Dye no.	Substituents		Molecular formula	Molecular weight	C%		H%		N%	
	R ₁	R ₂			Require	Found	Require	Found	Require	Found
2a	H	H	C ₁₄ H ₁₆ N ₄ O ₂ S	304	55.26	55.10	5.26	5.08	18.42	18.28
2b	CH ₃	H	C ₁₅ H ₁₈ N ₄ O ₂ S	318	56.60	56.48	5.66	5.51	17.61	17.51
2c	C ₂ H ₅	H	C ₁₆ H ₂₀ N ₄ O ₂ S	332	57.83	57.75	6.02	5.91	16.86	16.71
2d	H	CH ₃	C ₁₅ H ₁₈ N ₄ O ₂ S	318	56.60	56.45	5.66	5.60	17.61	17.52
2e	H	Cl	C ₁₄ H ₁₅ N ₄ O ₂ SCl	338.5	49.63	49.49	4.43	4.32	16.54	16.54

Table 3 Dyeing properties of dyes.

Dye no.	Color shade on cellulose triacetate	Light fastness	Wash fastness	Perspiration fastness		Sublimation fastness	Rubbing fastness	
				Acid	Alkaline		Dry	Wet
2a	Yellowish-orange	4-5	4-5	4-5	4-5	5	4-5	4-5
2b	Maroon	4-5	4-5	4-5	5-4	5	4	4-5
2c	Pink	4-5	4-5	5-4	4-5	5	4	4-5
2d	Dark maroon	4-5	4-5	4-5	4-5	5	4	4
2e	Light pink	4	4-5	4-5	5-4	5	4-5	4-5

planarity of the dyes, because of the lower steric interaction of a five-membered ring. The introduction of electron-donating or electron-attracting group at suitable position in the diazo component affect the absorption characteristics of the dyes. As can be seen from the data in Table 1, the bathochromic shift can be obtained by enhancing the electron donor properties of the coupled ring, e.g. by introducing a methyl group ortho to the azo group, as in dye **2d**. This gave considerable shift of 15 nm relative to dye **2e**. Thus, electron displacement can be enhanced by electron donating substituents on the coupled ring. The resulting increase polarizability should result in bathochromism (Table 1).

3.4. Infrared spectra of dyes

The characteristic band at 3400–3420 cm^{-1} indicates the N–H and O–H stretching. The strong bands observed at 1600–1619 cm^{-1} indicate stretching vibration of the C=O. The bands at 820–830 cm^{-1} and 1320–1360 cm^{-1} corresponding to para disubstituted ring and –C–N–C stretching, respectively. The azo group is confirmed at 1515–1520 cm^{-1} . The band at 1450–1460 cm^{-1} is due to the alkyl chain. The methyl group is confirmed at 2895–2900 cm^{-1} (Table 2).

3.5. Dyeing properties of dyes

All the disperse dyes **2a–e** were applied at 2% depth on cellulose triacetate fabric. Their dyeing properties are given in Table 3. These dyes gave a narrow range of color varying from yellow to maroon with excellent brightness, depth on fabric and deeper shade with high tinctorial and excellent levelness. The variation in the shade of the dyed fabric results from alteration in the coupling components. The dyed fabric have fair (4) to fairly good (4–5) light fastness. All the dyes show very good (4–5) to excellent (5) fastness to wash, rubbing, perspiration and sublimation on fabric. A remarkable degree of levelness and brightness after washing indicates good penetration and excellent affinity of dyes to the fabric. All the fastness properties shown in Table 3 are interrelated since they depend, among other things, on the rate of diffusion of dye in the fabric. This rate is a function of the geometry of the dye molecule. The concentration of dye in the fabric appeared to be the most influential factor in the fastness of the dyeing.

The β -hydroxyalkyl group, which has been widely used in disperse dyes, generally gives moderate light fastness. In attempting to trace the relationships between chemical structure and light fastness, one needs to appreciate that there is no absolute value for the light fastness of a dye. The value ob-

tained for a given colorant in any fading test depends on many factors, the most important of which are: concentration and/or degree of aggregation of dye within the fabric (deep sample shade); nature of fabric in which it is dispersed; the characteristics of the incident radiation, molecular structure and substantivity. The substantivity of the dyes for fabric such that they almost conferred similar depth of shade (close fixation values), which had the same rating, implying that the intrinsic photostabilities of dyes on the fabric are similar. This suggesting that smaller molecular size and higher substantivity factors hold more importance than that concerning the slight different in molecular structure of dyes.

In order to obtain a satisfactory combination of light and sublimation fastness, a judicious choice of substituents is required. The introduction of polar groups can adversely affect the dyeing properties of a dye but the hydroxyalkyl group appears to be particularly useful in increasing polarity without excessive undesirable side effects. The free hydroxyl group and also short alkyl chain favour formation of compact aggregates of dye, which confers low surface activity and thus cause dye to spread as multilayer on the internal surface of the fabric; the multilayer is less susceptible to fading. Also, the smaller molecules penetrate deeper into the fabric structure, which close on them after dyeing and so protects them from attack when irradiated. The compactness of structure improves sublimation fastness.

Table 1 shows that exhaustion and fixation of all the dyes on fabric. The very good exhaustion on the fabric may be expected due to the diffusion of the dye within the fabric proceed rapidly under the dyeing condition. Hence, the rate of diffusion of the dye molecules into fabric is higher, which increases the exhaustion value. Also the smaller thiazolyazo dye molecule penetrates deeper into the fabric which closes easily on fabric after dyeing and so protects them from sublimation. The sublimation fastness has been observed that all the dyes exhibit excellent (5) rating on fabric.

4. Conclusion

The diazo components **1** possessed sufficient basicity to warrant the employment of various conditions for diazotization. It was found that efficient diazotization can only be achieved using nitrosylsulphuric acid obtained from sodium nitrite and concentrated sulphuric acid. A series of heterocyclic azo disperse dyes containing various N-alkyl-N-(2-hydroxyethyl)aniline coupling moiety have been synthesized by conventional methods and their color properties examined both in solution and on application to cellulose triacetate fabric.

A gamut of color shades narrow ranging from yellow to maroon was obtained by applying the variously substituted dyes. The small size of thiazolylazo dyes provides good dyeability. The intrinsic conjugation in the dye structure results in very good color strength. The nature of the substituent in coupling components has a little influence on the visible absorption. These dye give very good exhaustion, fixation and economical (as compared to anthraquinones which could only provide up to blue shades). An outstanding characteristic of these dyes is that they give level dyeing and excellent dispersability. A remarkable degree of levelness after dyeing indicates good penetration and affinity of these dyes to the fabric. These dyes, however, are noteworthy in its excellent affinity and intensity of color. Outstanding characteristics of these dyes are that they give deep and bright hues with leveling dyeing. The bright hue might be attributed to the greater planarity of the thiazole dyes, because of the lower steric interaction of a five membered ring.

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