

Monoazo disperse dyes based on 2-amino-1,3,4-thiadiazole derivatives

HARI R. MARADIYA

V. P. and R. P. T. P. Science College, Vallabh Vidyanagar 388 120, Gujarat State, India

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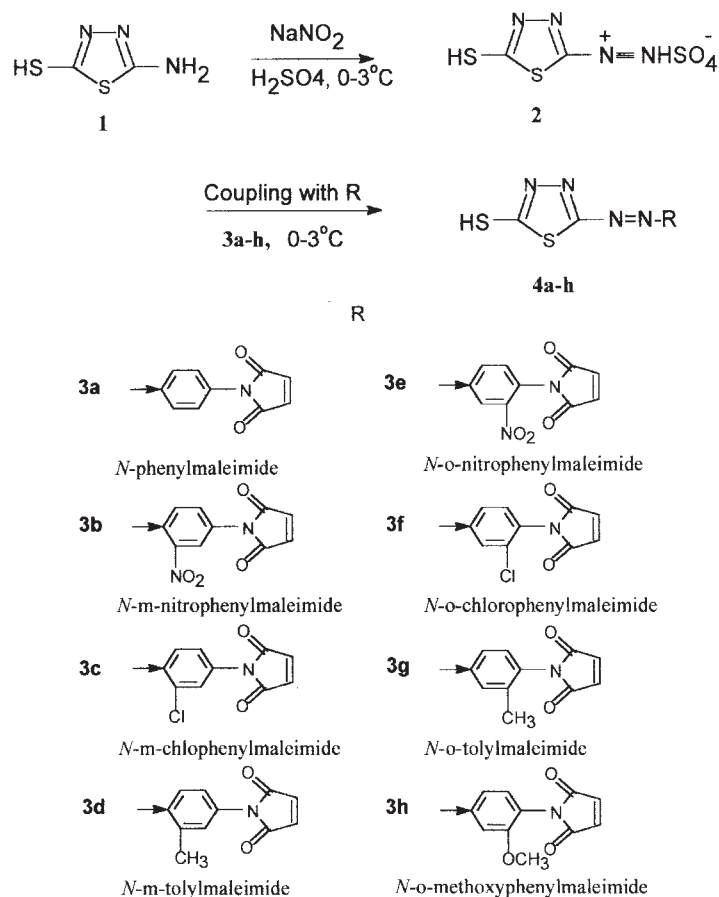
Abstract: A series of monoazo disperse dyes based on 2-amino-5-mercapto-1,3,4-thiadiazole was prepared by coupling with various *N*-arylmaleimides. The dyeing performance of these dyes was assessed on nylon fabric. The dyes were found to give yellow to brown colour shades on dyeing with good depth and levelness on nylon fabric. The dyebath exhaustion, fixation and fastness properties of the dyes were also determined. The dyed fabric showed moderate to good light fastness and very good to excellent fastness to washing, rubbing, perspiration and sublimation. The IR and visible range spectral properties of the dyes were also determined.

Keywords: 2-amino-5-mercapto-1,3,4-thiadiazole, *N*-arylmaleimides, disperse dyes, dyeing, fixation, fastness.

INTRODUCTION

Heterocycles have been put to much use in the chemistry of disperse dyes, which it has been claimed was the first area to foster the industrial exploitation of heterocyclic amines.¹ Most heterocyclic dyes of technical interest for application to textiles are derived from diazo components consisting of five-membered rings containing one sulphur heteroatom and to which a diazotisable amino group is directly attached. The ring may also possess one or more nitrogen heteroatoms. These diazo components are capable of providing red to blue disperse dyes that meet the rigorous technical and economical requirements.

Aminothiadiazoles were among the first heterocyclic diazo components for disperse dyes to be described.² Such heterocyclic amines have been used commercially to produce brilliant red polyester dyes. Dyes derived from 2-amino-1,3,4-thiadiazole are of technical interest for the production of brilliant red shades.² Depending on the various substituents in the diazo component and the coupler used, these dyes exhibit brilliant scarlet to bluish-red colour.^{3–5} Commercial success has been achieved with 2-amino-5-ethylthio-1,3,4-thiadiazole in the replacement of red anthraquinone-based dyes;¹ a dye from this diazo component, C. I. Disperse Red 338, has the only structure of this type that has been disclosed to date.⁶ These, however, still do not quite match the brightness of anthraquinone dyes. In

Scheme 1. Synthetic route of dyes **4a-h**.

spite of the great number of patent applications for structures with substituents such as halogen,⁷ alkyl,⁸ trifluoromethyl⁹ and various substituted mercapto groups,¹⁰ all have fallen short of expectation as far as their commercial exploitation is concerned. Thiadiazole classes of diazo components have been extensively studied over the last ten years^{11–19} because of their brightness and good light fastness of hydrophobic fabrics. They also exhibit a generally excellent performance to wet fastness on polyester fabric. These, however, still do not quite match the brightness of the anthraquinone dyes. The great number of patent applications for structures with various substituents and couplers testify to the still high interest and bears witness to the versatility of 2-amino-1,3,4-thiadiazole derivatives.^{20–23} Relatively little research work has been published regarding this class compared to the other categories of dyes derived from five-membered sulphur-containing heterocycles.

In continuation of the work in this direction^{24–29} the present paper comprises the synthesis of disperse dyes from 2-amino-5-mercapto-1,3,4-thiadiazole (**1**) using various substituted *N*-arylmaleimide (**3a-h**) and their application on nylon fabric as disperse dyes. In

addition to the characterization of the dyes, an evaluation of their technical properties and a colour assessment were performed.

EXPERIMENTAL

Materials and methods

All the chemicals used in dye synthesis were of commercial grade and were further purified by crystallization and distillation. Melting points were determined by the open capillary method. The visible absorption spectra were measured using a Carl Zeiss UV/VIS Specord Spectrometer. A Perkin-Elmer model 983 recording infrared spectrophotometer was used for recording the IR spectra of the dyes as KBr pellets in the range between 4000 and 400 cm^{-1} . Elemental analyses were estimated by means of a Carlo Erba Elemental Analyser 1108. Purification of the products was carried out by TLC (1 mm thickness glass plates 20×5 cm, using a suspension of silica gel G in CHCl_3). The diazo component 2-amino-5-mercapto-1,3,4-thiadiazole³⁰ (**1**) and the various *N*-arylmaleimides³¹ (**3a-h**) were prepared according to the literature.

Synthesis of 2-[4'(N-phenylmaleimido)-azo]-5-mercapto-1,3,4-thiadiazole (4a). Dry NaNO_2 (1.38 g, 0.02 mol) was added in parts over a period of 30 min to concentrated sulphuric acid (1.1 ml) under stirring below 65 °C. The resulting solution was treated dropwise at 5–20 °C with 20 ml of a mixture of acetic acid-propionic acid (17:3 v/v). The resulting nitrosylsulphuric acid mixture was cooled to 0 °C, and the diazo component **1** (2.66 g, 0.02 mol) was added in portions and stirring was continued at this temperature for 2 h. The excess nitrous acid (gave a positive test on starch-iodide paper) was decomposed with the required amount of urea. The thus obtained clear diazonium salt **2** solution was used for the subsequent coupling reaction. Now *N*-phenylmaleimide (**3a**) (3.46 g, 0.02 mol), was dissolved in 10 ml acetic acid and cooled to 0 °C. Freshly prepared diazonium salt **2** solution was added dropwise to this well-stirred solution over a period of 2 h under vigorous stirring, keeping the temperature below 5 °C. The reaction mixture was stirred for 45 min at 0–3 °C, maintaining the pH at 4–5 with the help of a 10 % sodium acetate solution. The reaction mass was then diluted with 100 ml water, filtered, washed with water and dried in an oven at 50 °C. The thus obtained dye **4a** was recrystallized from a minimum amount of DMF (4.08 g, 81 %). The m.p. 208–209 °C (DMF).

The same procedure was repeated for the preparation of dyes **4b-h** using various *N*-arylmaleimide coupling components **3b-h**. The whole synthesis process is shown in Scheme 1. The characterization data for the dyes **4a-h** are given in Table I.

TABLE I. Characterization data of dyes **4a-h**

Dye no.	Coupling component R	Yield %	mp (DMF) °C	$\lambda_{\text{max}}/\text{nm}$ (DMF)	$\lambda_{\text{max}}/\text{nm}$ (conc. H_2SO_4)	log ϵ	Exhaustion %	Fixation %	R_f
4a	<i>N</i> -Phenylmaleimide	81	208–209	480	395	3.36	72	81	0.88
4b	<i>N-m</i> -Nitrophenylmaleimide	84	225–227	440	390	3.11	80	85	0.82
4c	<i>N-m</i> -Chlorophenylmaleimide	72	192–193	450	395	3.29	86	85	0.90
4d	<i>N-m</i> -Tolylmaleimide	78	186–188	430	400	4.10	83	80	0.92
4e	<i>N-o</i> -Nitrophenylmaleimide	70	217–218	450	400	4.60	73	78	0.87
4f	<i>N-o</i> -Chlorophenylmaleimide	68	236–237	440	415	3.29	78	83	0.81
4g	<i>N-o</i> -Tolylmaleimide	69	200–201	410	485	4.70	68	81	0.84
4h	<i>N-o</i> -Methoxyphenylmaleimide	71	195–196	450	415	4.47	81	80	0.88

Dyeing method

Dyeing at 90–135 °C temperature under high pressure (24–30 psi) is a convenient method for dyeing nylon fiber in the laboratory. A laboratory model glycerin-bath high-temperature beaker dyeing machine was used. A paste of finely powdered dye (40 mg) was prepared with the dispersing agent Dodamol (80 mg), wetting agent Tween-80 (5 mg) and water (1 ml) in a ball mill for 10 min. To this paste, water (99 ml) was added under stirring and the pH was adjusted to 4.5–5 using acetic acid. This dye suspension (100 ml) was added to a beaker provided with a lid and a screw cap. Before closing the lid and tightening the metal cap over the beaker, a wetted pattern of nylon fiber was rolled into the beaker. The beaker was then placed vertically on the rotatory carrier inside the tank and the clamp plate firmly tightened. The rotatory carrier was then allowed to rotate in the glycerin-bath, the temperature of which was raised to 110 °C (for nylon fiber) at the rate of 2 °C/min. The dyeing was continued for 1 h under pressure. After cooling for 1 h, the beaker was removed from the bath and washed with water. The pattern was thoroughly washed with hot water at 50 °C, and then with cold water, and finally dried at room temperature.

TABLE II. Elemental analysis of dyes **4a-h**

Dye no.	Molecular formula	Molecular weight	C %		H %		N %	
			Calcd.	Found	Calcd.	Found	Calcd.	Found
4a	C ₁₂ H ₇ N ₅ O ₂ S ₂	317	45.42	45.02	2.20	2.10	22.80	21.95
4b	C ₁₂ H ₆ N ₆ O ₄ S ₂	362	39.77	39.10	1.65	1.45	23.20	23.08
4c	C ₁₂ H ₆ N ₅ O ₂ S ₂ Cl	351.5	40.96	40.46	1.70	1.60	19.91	19.68
4d	C ₁₃ H ₉ N ₅ O ₂ S ₂	331	47.12	46.89	2.71	2.60	21.14	21.00
4e	C ₁₂ H ₆ N ₆ O ₄ S ₂	362	39.77	39.10	1.65	1.45	23.20	23.08
4f	C ₁₂ H ₆ N ₅ O ₂ S ₂ Cl	351.5	40.96	40.46	1.70	1.60	19.91	19.68
4g	C ₁₃ H ₉ N ₅ O ₂ S ₂	331	47.12	46.89	2.71	2.60	21.14	21.00
4h	C ₁₃ H ₉ N ₅ O ₃ S ₂	347	44.95	44.80	2.59	2.40	20.17	20.06

Fastness properties

The fastness to light, sublimation and perspiration was assessed in accordance with BS:1006–1978. The rubbing fastness test was carried out using a crockmeter (Atlas) in accordance with AATCC-1961 and the wash fastness test in accordance with IS:765–1979. Details of the various fastness tests are mentioned below.

Light fastness test

The light fastness study was carried out using a Microscal light fastness tester having a straight mercury vapour lamp (MB/V 400 W). The dyed fiber was exposed to light along with standard dye patterns of specific rating. Such standard samples are the blue wool standard manufactured by Microscal Ltd and identified by the numerical designation 1 to 8. The higher the rating, the better is the light fastness. The results are given in Table III.

Wash fastness test

Fastness to washing was assessed using an automatic Lander-O-meter under the following conditions.

The washing solution contained neutral soap at 5 g/l and soda ash 2 g/l, a washing temperature was 95 °C, the duration of washing was 30 min; the material-to-liquor ratio was 1:50. After washing, the specimen was removed and rinsed with hot water (35 °C) until the rinsed water showed no alkalinity with phenolphthalein. It was then squeezed and air dried. The effect on the colour was expressed and defined by reference to the international geometric grey scale. The results are given in Table III.

Rubbing fastness test

The specimen is fastened in a crockmeter, which causes a piece of standard white cloth to rub against the coloured specimen under controlled conditions of pressure and speed. The rubbing fingers are covered with white cloth, both for the dry test and the wet test, and slide back and forth for twenty rubbing strokes. The

colour transferred to the white cloth is compared with a grey scale. The grey scale used for the alteration of colour consisted of grade 1–5. The results are given in Table III.

TABLE III. Quality of dyeing

Dye no.	Colour shade on nylon	Light fastness	Wash fastness	Rubbing fastness		Perspiration fastness		Sublimation fastness
				Dry	Wet	Acid	Alkaline	
4a	Dark yellow	3–4	5	5	5–4	5	5	5
4b	Dark brown	4–3	5	5	5	5	5	5
4c	Yellow	4–3	5	5–4	5	5	5	5
4d	Yellow	4–5	4–5	5–4	5–4	5	5	5
4e	Brown	3–4	5	5	5	5	5	3–4
4f	Reddish yellow	4–3	5	5	5	5	5	5
4g	Yellowish orange	4–5	5	5	5	5	5	4–5
4h	Deep brown	3–4	5	5–4	5	5	5	5

Perspiration fastness test

A specimen of the textile in contact with specified adjacent fibers is treated in two different solutions containing histidine, drained, and placed between two plates of 11.5 × 6 cm size and of 0.15 cm thickness under the pressure of a weight-piece of mass 5 kg in a testing device consisting of a stainless steel frame. It was kept in an oven for 4 h at 37 ± 2 °C. The specimen and the adjacent fibers are dried separately. The change in colour of each specimen and the staining of the adjacent fibers are assessed with grey scales.

Acid perspiration test

The freshly prepared acid solution contained 5 g/l sodium chloride, 2.2 g/l sodium dihydrogen orthophosphate (NaH₂PO₄·2H₂O) and 0.5 g/l L-histidine mono-hydrochloride mono-hydrate (C₆H₉O₂N₃HCl·H₂O). The solution is brought to pH 5.5 with a 0.1 M acetic acid solution. The results are given in Table III.

Alkaline perspiration test

A freshly prepared alkaline solution contained 5 g/l sodium chloride, 0.5 g/l L-histidine mono-hydrochloride mono-hydrate (C₆H₉O₂N₃HCl·H₂O) and 2.5 g/l disodium hydrogen orthophosphate (Na₂HPO₄·2H₂O). The solution is brought to pH 8 with a 0.1 M sodium hydroxide solution. The results are given in Table III.

Sublimation fastness test

The composite specimens were placed in a heating device (Sublimation Tester, Model STE) equipped with two heating plates with an electrical heating system and kept there for 30 s at 150 ± 2 °C. The dried fiber was then removed and kept in air for 4 h. The change in colour of the specimen was assessed with grey scales. The results are given in Table III.

Determination of the percentage of exhaustion and fixation (Dissolution method)

The dyebath exhaustion percentage (%E) and fixation percentage (%F) of the dyed fiber are determined according to the method described below.

Exhaustion

This method involves two steps, *i.e.*, (a) the construction of a calibration curve and (b) estimation of the dye.

(a) *Calibration study*: The calibration study involves the preparation of a dye solution of known concentration and the measurement of the absorption at a specified wavelength. A solution of known weight (40 mg) dye was prepared by grinding very well with the dispersing agent Dodamol (20 mg) and 2–3 drops of wetting agent, Tween-80 in a small mortar with pestle and diluting with the required amount of distilled water to make

the final volume 100 ml (stock solution). Now, four dilutions of different concentration were prepared by using 2, 5, 7 and 10 ml each of the stock solution with the required amount of distilled water to make the final volume 100 ml. A solution of 20 mg of the dispersing agent and 2–3 drops of wetting agent in 100 ml water was used as the reference solution. The absorption of stock solution was measured at various wavelengths ranging from 350 to 700 nm and the position of λ_{max} value was ascertained. At the λ_{max} value, the absorption of the four diluted solutions were measured and a plot of absorption *versus* concentration was made for each dye. This plot is known as a calibration curve.

(b) *Estimation of the dye.* The combined solution of dye liquor and washing of the dyed pattern was diluted to 100 ml with distilled water. Then 5 ml of the solution was further diluted to 50 ml with distilled water. The amount of dye exhaustion on the fiber can be calculated by measuring the absorption of the above solution at λ_{max} and reading the corresponding concentration from the calibration curve.

Fixation study

This method also involves two steps. *i.e.*, (a) construction of a calibration curve and (b) estimation of the dye.

(a) *Calibration curve.* This follows the same procedure as above. A known weight of dye (5 mg) was dissolved in A.R. grade concentrated sulphuric acid (50 ml) and four different dilutions were prepared using 1, 2, 3 and 4 ml to the stock solution. The optical density of the stock solution was measured at various wavelengths of the range from 350 to 700 nm. At the position of λ_{max} , the optical density of the four diluted solutions were measured. A calibration curve was prepared by plotting optical density against concentration.

(b) *Estimation of the fixation of the dye on the fiber.* A sample of undyed fiber (0.1 g) was dissolved in concentrated sulphuric acid (25 ml) and this solution was used as a reference solution in the colorimetric estimation. Then dyed fiber (0.1 g) was dissolved in 15 ml concentrated sulphuric acid. This required about 10–15 min. The solution was then diluted to 25 ml with concentrated sulphuric acid. The optical density of this solution was measured at the λ_{max} value. Using the calibration curve, the concentration of the dye was measured and hence the amount of dye fixed on the fiber was estimated.

The fixation of the dye on a fiber is based on its exhaustion. It can be seen from Table I, dye **4a** has 81 % fixation. This means 81 % fixation on the basis of the corresponding 72 % exhaustion. The fixation value is not directly based on the amount of dye taken in the dyebath but is based on the amount of the dye exhausted on the fiber. The fixation value may be higher or lower than the exhaustion value.

The exhaustion and fixation values were calculated using the following relations:

$$\% \text{ Exhaustion} = \frac{\text{Amount of dye exhausted on the fiber from dyebath} \times 100}{\text{Total amount of dye taken in dyebath}}$$

$$\% \text{ Fixation} = \frac{\text{Amount of dye fixed on the dyed fiber} \times 100}{\text{Amount of dye exhausted on the fiber from the dyebath}}$$

Suppose 40 mg dye **4a** had been taken in the dyebath for dyeing purpose. If 28.80 mg dye was found to be exhausted on the fiber. *i.e.*

$$\% \text{ exhaustion} = \frac{28.80 \times 100}{40} = 72 \%$$

Now, suppose 23.32 mg dye **4a** had been fixed on the fiber (out of 28.8 mg). *i.e.*

$$\% \text{ fixation} = \frac{23.32 \times 100}{28.80} = 81\%$$

RESULTS AND DISCUSSION

Diazotization and coupling

2-Amino-5-mercapto-1,3,4-thiadiazole (**1**) was synthesized from thiosemicarbazide by reacting with carbon disulphide, either with or without the presence of a base. The ring

closure of the dithiocarboxylate salt occurs upon heating under the reaction conditions. Weak amines, such as compound **1**, required the use of nitrosylsulphuric acid, and it was diazotized satisfactorily at 0 °C by adding to nitrosylsulphuric acid in acetic acid-propionic acid mixture. The resulting diazonium salt **2** solution was generally used within a few hours since it decomposed on standing, even in the cold. The subsequent coupling reactions took place readily on adding the diazonium salt solution continuously to a solution of the coupling component in acetic acid. Coupling was usually accompanied by some evidence of decomposition; however, by careful addition of the diazonium salt **2** solution at 0–3 °C to a solution of the coupler in acetic acid, high yields (68–84 %) of the dye were usually obtained. To complete the coupling, particularly for reactions using nitrosylsulphuric acid in the diazotization, the pH of the reaction mixture was eventually adjusted to approximately 4–5. Thus, an appropriate amount of 10 % sodium acetate solution was slowly added below 3 °C. In order to determine the end point of diazotization, it was found useful to check for the presence of the unreacted diazo component by TLC. Thus, when the unreacted diazo component was no longer visible by TLC, the diazotization was ended.

Physical properties of the dyes

The dyes **4a–h** were dark reddish-brown coloured solids and gave deep brown coloured solutions in DMF. The purified dyes **4a–h** were all found to have satisfactory elemental analyses. All the dyes required purification to eliminate contaminants arising from decomposition of the diazo and/or coupling components used. The purity of the dyes was evaluated by TLC using ethyl acetate–benzene (1:4 v/v) as the solvent system. When adsorbed onto silica chromatography plates, each dye produced a single coloured spot. All the recrystallized dyes exhibited well-defined melting points characteristic of pure compounds but it would be premature 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, geometry, size and molecular interactions).

Infrared spectra of the dyes

The infrared spectra of the dyes **4a**, **4c** and **4g** are shown in Fig. 1. The unsaturation is indicated by the C–H stretching vibrations that appear at 3085–3200 cm^{-1} and by the one appearing at 825–835 cm^{-1} due to out-of-plane deformation vibrations. The band at 700–730 cm^{-1} is an out-of-plane deformation vibration characteristic of HC=CH. The vibration band of the skeletal C–C bond appears at 1570 cm^{-1} . The band appearing at 1580–1595 cm^{-1} is due to the stretching vibration of C=C, which is conjugated with the C=O. The strong bands observed at 1700–1721 cm^{-1} result from the stretching vibration of the C=O of the cyclic maleimide ring. The band at 630–680 cm^{-1} and 570–580 cm^{-1} can be attributed to an in-plane and an out-of-plane deformation vibration of the C=O. Bands appear at 820–860 cm^{-1} corresponding to a para disubstituted ring. The bands at 1370 cm^{-1} and 1350 cm^{-1} are due to a stretching vibration of –C–N–C, the first being asymmetrical and the second symmetrical. The azo group is confirmed at 1585–1605 cm^{-1} . The

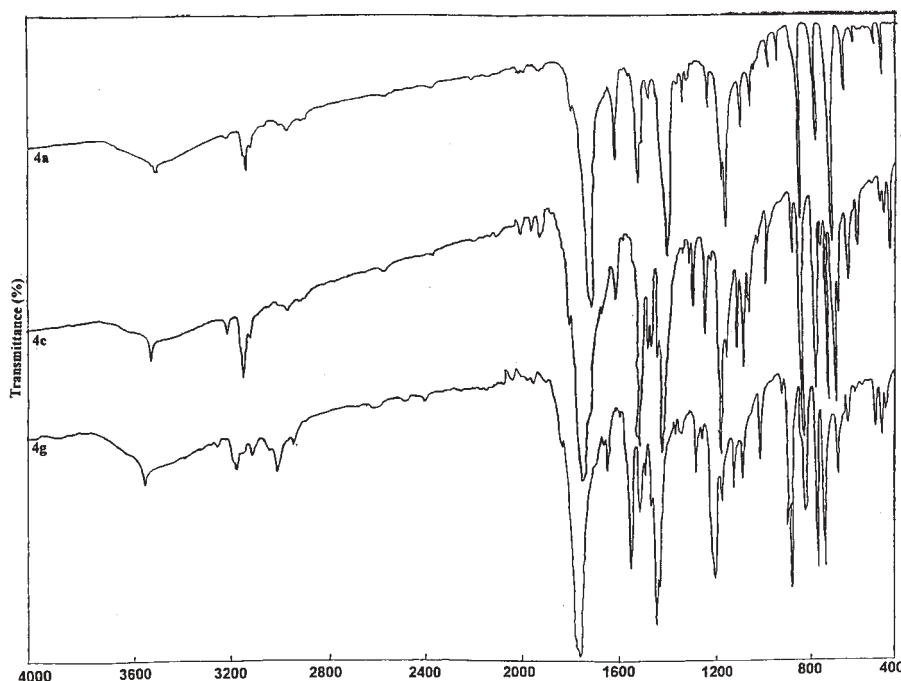


Fig. 1. IR spectra of dye **4a**, **4b** and **4c**.

S–H stretching vibration of the mercapto group appears at $600\text{--}660\text{ cm}^{-1}$ and $675\text{--}700\text{ cm}^{-1}$. The band appearing at 1070 cm^{-1} is due to the stretching vibration of --C--N . The characteristic bands at 2850 cm^{-1} , $640\text{--}700\text{ cm}^{-1}$ and $1390\text{--}1450\text{ cm}^{-1}$ are due to --C--H bending vibrations of --OCH_3 , C--Cl and C--CH_3 stretching vibration, respectively. The band at $1320\text{--}1360\text{ cm}^{-1}$ is due to the symmetric stretching of the --NO_2 group.

Dyeing properties of dyes

The disperse dyes **4a-h** were applied at 2% depth on nylon fabric. Their dyeing properties are given in Table III. These dyes gave yellow to brown colours with brighter and deeper shades with high tinctorial strength and excellent levelness on the fabric. The variation in the shades of the dyes fabric results from alteration in the coupling components. The dyeings showed moderate (3–4) to good (4–5) light fastness, very good (5–4) to excellent (5) washing, rubbing, perspiration and sublimation fastness properties. The remarkable degree of levelness and brightness after washing indicated good penetration and excellent affinity of these dyes for the fabric.

Colour and spectral properties of dyes

The visible absorption spectra of dyes **4a-h** were recorded in DMF solution and concentrated sulphuric acid. The absorption maxima and extinction coefficients are presented in Table I. The introduction of electron-donating or electron-attracting groups at suitable position in the coupler ring affects the absorption characteristics of the dyes. The dyes,

listed in Table I, are reddish-brown to deep brown having absorption at 410 to 480 nm in DMF. The values of the logarithm of molar extinction coefficient ($\log \epsilon$) of the dyes were in the range of 3.11–4.70, consistent with their high intensity of absorption.

Exhaustion and fixation properties of dyes

Table I shows the exhaustion and fixation of the dyes on the fabric were very good. The exhaustion values depend on the structure of the fiber. Consequently, diffusion of the dye within the fabric proceeds rapidly under the given dyeing conditions, thereby affecting the exhaustion value.

CONCLUSION

A series of thiadiazole azo disperse dyes containing various *N*-arylmaleimides residues were prepared by conventional methods and their properties examined both in solution and on application to nylon fabric. Nitrosylsulphuric acid was needed for satisfactory diazotization of 2-amino-5-mercapto-1,3,4-thiadiazole. Within the range of dyes investigated, a gamut of colour variation from yellow to brown on nylon fabric with excellent affinity and intensity of colour were observed. Furthermore these dyes show level dyeing, excellent dispersability and dyeability (additional dyeing properties, not exhibited by the carbocyclic azo and anthraquinone disperse dye structure). Exhaustion and fixation of these dyes are very good. This indicates that the dyes have good compatibility with the fabric.

The remarkable degree of levelness after washing indicates good penetration and affinity of these dyes to the fabric. The small size of such diazo components provides better dyeability. The intrinsic conjugation in the diazo structure provides for very good colour strength. The compactness of the structure provides excellent sublimation fastness. It is these properties which have aided the rise to prominence of such diazo components as replacement for the benzenoid colourants usually used on nylon fabric.

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ИЗВОД

МОНОАЗО ДИСПЕРЗНЕ БОЈЕ ЗАСНОВАНЕ НА ДЕРИВАТИМА 2-АМИНО-1,3,4-ТИЈАДИАЗОЛА

HARI R. MARADIYA

V. P. and R. P. T. P. Science College, Vallabh Vidyanagar 388 120, Gujarat State, India

Синтетизована је серија моноазо дисперзних боја заснованих на 2-амино-5-меркапто-1,3,4-тијадиазолу купловањем са различитим *N*-арилмалеимидима. Способност бојења ових боја проучавана је на најлонским тканинама. Одређене су такође и исцрпљивост купатила, фиксација и отпорности ових боја. Обојене тканине показале су умерену до добру стабилност на светлост, као и врло добру до одличну стабилност у односу на прање, трљање, зној и сублимацију. Одређене су и спектралне особине ових боја у ултраљубичастој и видљивој области.

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