

Synthesis of several new pyridine-2(1H) thiones containing an arylazo function and their applications in textile printing

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Abstract

Purpose – To synthesise several new pyridine-2(1H)thione containing an arylazo function and their applications in textile printing.

Design/methodology/approach – To prepare substituted 5-arylazopyridine-2(1H)thione by the coupling of diazonium salts of aniline derivatives with dibenzoyl methane (1,3-diphenyl-1,3-propanedione) to form arylhydrazone of dibenzoylmethane which react with cyanothioacetamide in boiling ethanolic sodium ethoxide to yield the 5-arylazo-2(1H)-pyridinethione derivatives. The printing properties by screen printing or transfer printing technique of these dyes are reported by using sodium alginate as thickening agent.

Findings – The structure of 5-arylazopyridine-2(1H)thione dyes were established and confirm for the reaction products on the basis of their element analysis and spectral data (MS, IR and ¹H-NMR). The suitability of the prepared dyestuffs for either heat transfer printing or traditional printing on polyester and nylon 6 fabrics has been investigated. The prints obtained from dyes possess high-colour strength as well as good overall fastness properties.

Research limitations/implications – Synthesis of azo dyes containing pyridinethiole moiety has never been reported in textile printing.

Practical implications – Production of simple and less costly dyes.

Originality/value – 5-arylazo pyridine-2(1H)thione, the result of our work aimed to define the scope of and limitation of our procedures for the synthesis of novel 5-arylazopyridine-2(1H)thione.

Keywords Dyes, Textiles, Printers

Paper type Research paper

Introduction

The synthesis of pyridone azo compounds has been extensively studied (Schaezter and Ger. Offen, 1995). Many derivatives of this type were proven to be excellent dyes (Rether and Russ, 1995). In spite of the large number of reports on the use of these compounds in dye industry, according to our knowledge, only few of their corresponding pyridinethione had been reported. In continuation of our interest in synthesis of arylazopyridine-2(1H)-thiones (Elgemeie and Fatty, 1995; Elgemeie and Hussain, 1994; Elgemeie *et al.*, 1990, 1992a, b, 1993, 1995, 1997, 2002). The present paper reports synthesis and printing properties of arylazo pyridine-2(1H)thione.

There are currently two major printing processes for fabrics: silk screen printing and heat transfer printing. Silk screen printing consists of lithographic printing and circular screen printing. Both require the use of a stencil to obtain the desired patterns. In order to fix the ink, the fabrics needs to go through the processes of the steaming, fixation, washing and

rinsing. Such printing technique is well-developed. A conventional machine can print up to 60m/min, but it produces a large amount of wastewater, as every 100m of fabric could use up to 5m³ of water. As far as water consumption is concerned, heat transfer printing, developed in the 1960s, offers an alternative and has found extensive applications in various industrial sectors. Dyestuff is used to colour patterns printed on heat transfer paper, which is then transferred onto the fabric surface to fix the patterns under certain temperature. Such method saves the finishing process and eliminates the problem of wastewater. The downside, however, is an increased consumption of paper, and the method can only apply on synthetic fabrics.

The present work was carried out with the following two objectives:

- 1 synthesis and identification of some novel substituted arylazopyridine-2(1H)thione dyes; and
- 2 the possibility of the use of the prepared dyestuffs for either heat transfer printing or traditional printing of polyester (PE) and nylon 6 fabrics.

Experimental

Materials

The following materials were employed in the study reported here:

- *Fabrics*. PE knitted fabric of 150 g/m², supplied by a private sector company, was treated with a solution containing 1 g/l non-ionic detergent at 70°C for 1/2 h,

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thoroughly washed, and air dried at room temperature.

Polyamide 6 of 212 g/m² supplied by El-Nasr Company for spinning, weaving and knitting, was treated with a solution containing 5 g/l soap at 50°C for 15 min, then thoroughly washed and dried at room temperature.

- *Thickener.* Commercial sodium alginate of high viscosity (alginate THV 30000) supplied by Françoise de colloids Company.
- *Chemicals.* Malononitrile, 2,4-dimethoxy aniline, α -naphthylamine, 2,4-dichloro aniline 2-amino-4,6-dinitrophenol, p-chloroaniline, p-nitroaniline, o-chloro-p-nitroaniline, p-Bromoaniline, di-benzoyl methane Acetic acid, sodium acetate, ethanol, sodium metal, hydrochloric acid, iron sulphate.

All chemical used were in reagent grades.

Dyestuff synthesis

Synthesis of 5-aryloxy-3-cyano-4,6-diphenylpyridine-2(1H) thiones derivatives.

General procedure

A mixture of arylazo of dibenzoylmethane (1,3-diphenyl-1,3-propanedione) (0.01 mole) 2 and cyanothioacetamide 1 (0.01 mole) were dissolved in ethanol (30 ml) containing sodium ethoxide (0.01 mole). The mixture was refluxed for 1 h and then allowed to cool to room temperature and acidified with dilute hydrochloric acid. The resulting solid product 5 was collected by filtration and crystallised from the ethanol (Figure 1).

Printing methods

Transfer printing experiments

Print paste recipe for printing the paper:

- Dye – 3 g.
- Sodium alginate – 3 g.
- Water – 94 g.

A good quality paper was manually printed with the above formulation using silk screen and then air dried.

Samples of PE and nylon 6 were transfer printed using the previously screen printed paper. The heat source was a 40 × 25 cm² flat-bed press. Printing time was 45 s, and the temperatures used were 150, 170, 190 and 210°C.

Silk screen printing experiments

Print paste recipe:

- Dye – 3 g.
- Sodium alginate – 3 g.
- Acetic acid – 1 g.
- Dispersing agent – 1 g.
- Water – 92 g.

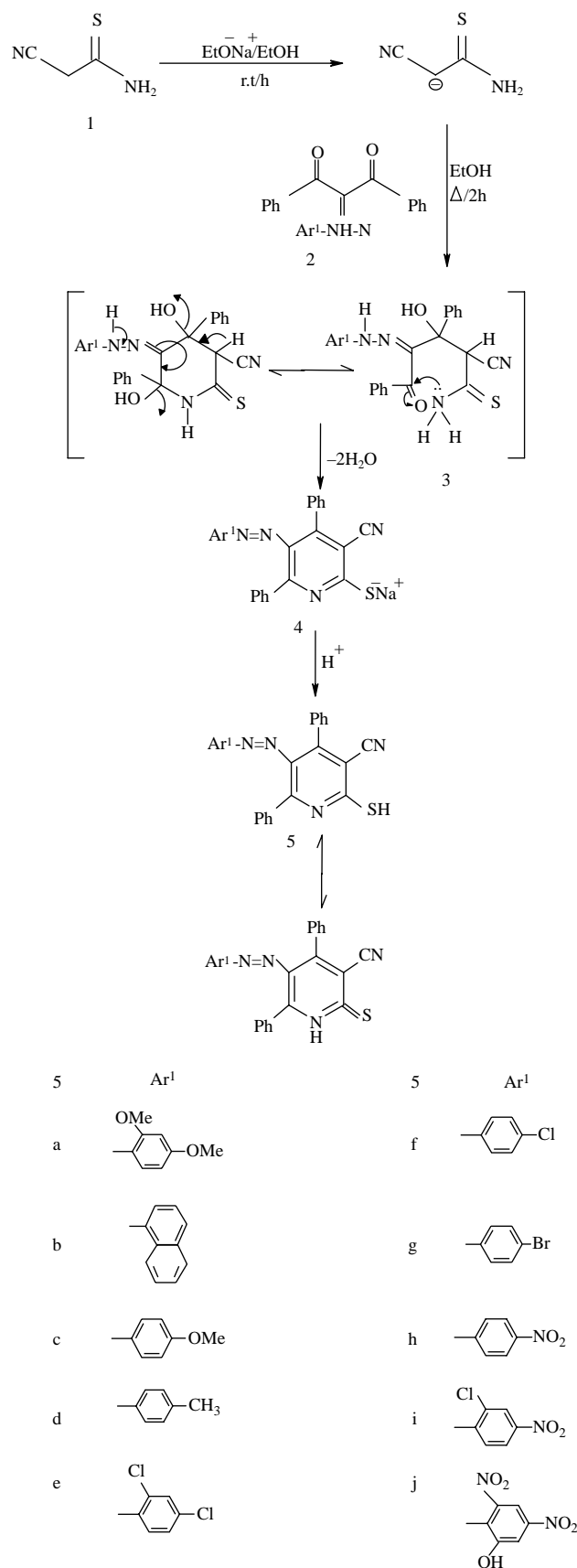
Samples of PE and nylon 6 fabrics were silk screen printed using the above printing paste, the printed fabrics were dried and fixed at different temperatures 150, 170, 190 and 210°C, then washed twice with cold water, then twice with hot water and finally rinsed with cold water, then air dried.

Measurements

Melting point

All melting points of the synthesised dyes were determined in open glass capillaries on Gallenkamp melting point apparatus and were uncorrected.

Figure 1 Scheme 1



IR spectra

The infra red of the synthesised dyes was measured using a Perkin Elmer/1650 FT-IR instrument.

¹H – NMR spectra

The ¹H – NMR spectra of the synthesised dyes were measured with a Varian 400 or Wilmad 270 MHz spectrometer for (CD₃)SO solutions using SiMe₄ as internal standard.

Mass spectra

Mass spectra were recorded in a Varian MAT112 spectrometer. Analytical data were obtained from the micro analytical data centre at Cairo University.

Spectrophotometric measurements

The optical absorbance of the dyes was measured in the ultraviolet-visible region between 300–700 nm, using a UNICAM UV spectrophotometer using 1 cm quartz cell. The dyes were dissolved in absolute ethanol at a concentration of 10⁻⁴ mole/l.

Colour measurements

The colour strength (K/S) of the printed samples was determined (Jdd *et al.*, 1975) by reflection measurements using an automatic filter spectrophotometer.

Fastness properties measurements

Fastness to washing, rubbing, light and perspiration were assessed according to standard methods (DINEN ISO, 1996, 1997; AATCC, 2002).

Results and discussion

The present paper reports synthesis and textile printing properties of several arylazo-pyridinethiones. Moreover, the results of our work aimed to define the scope and limitation of our procedures for the synthesis of novel arylazopyridine printing dye stuff are also reported. Thus, it has been found that arylhydrazones of dibenzoylmethane 2 reacted with cyanothioacetamide 1 in boiling ethanolic sodium ethoxide to yield the 5-arylozo-2(1H)-pyridinethione derivatives 5. The structures of 5 were established and confirmed for the reaction products on the basis of their elemental analysis and spectral data (MS, IR, ¹H NMR). The analytical data for 5c revealed a molecular formula C₂₅H₁₈N₄SO (M⁺ = 422). ¹H NMR revealed a broad bond at = 11.0 ppm assignable to SH group, a multiplet at 7.1–8.3 ppm assigned for aromatic protons. The formation of 5 is assumed to proceed via intermediary of 3, which loses two molecules of water to yield the stable sodium salts 4. Subsequent addition of water and acidification gave the final isolable 5-arylozopyridine-2(1H)-thiones 5.

The structures of the new dyes are established based on analytical data as well as IR and ¹H-NMR and mass spectral data (Tables I and II).

The effect of the structural configuration on the UV-visible absorption maxima of the dyes synthesis

The electronic absorption spectra of the title dye, as well as the dye fibre binding mechanism, have been studied. Thus, the UV spectra of 5-arylozo-3-cyano-4,6-diphenylpyridine-2(1H)thiones derivatives lie in the range 400–490 nm. Theoretically, pyridinethione has two tautomeric formula I and II (Figure 2). Tautomer I has two resonating structures A and B. Tautomer II has four resonating structures C–F.

A parent structure B of the pyridinethione ring plays an important role in the resonance hybrids of the dyes. Optimum conjugation is observed in the resonating system B, where the lone pair electron of the substituent X interacts efficiently with aromatic system. Consequently, such a high-energy dipolar arrangement will be stabilised when X is an electron-donating group. Concerning the tautomer II of the pyridinethione, the parent structure contributes to four resonance hybrids C–F. Structure F can be taken as a reasonable approximation to excited state. This arrangement is stabilised when X is an electron-donating group and in the aromatic ring (Figure 2).

The electronic and spectral data of the investigated dyes indicate the following:

- The pyridinethione dyes absorbed at higher absorption maxima due to the mobility of electronic transmission across the resonating system (structure F).
- The dye systems comprising phenyl moiety absorbed bathochromically compared to other analogues due to a longer conjugation which stabilised charge separation in the corresponding stabilised hybrids.
- The cyano group in the third position had no shift in absorption. This indicated that, the cyano group did not play an important role in attracting electrons through the conjugated system. Therefore, the charge separation between the electron-donating and the electron – attracting centres were not favoured.

Colour characteristics of the printed fabrics

The colour characteristics of the printed fabrics PE and nylon 6 were investigated and are shown in Tables III and IV.

It could be seen that the colour characteristics of the printed fabrics (expressed as K/S) varied due to difference in the nature of substituents present on the dye molecules. It is also affected by the type of printing method, for example.

In case of transfer printing technique

The printed PE and nylon 6 using the prepared dyes 5_{a-j} was heated at different temperature (150, 170, 190 and 210°C) at a constant time of 45 s (Figures 3 and 4).

It is noticed, from Figures 3 and 4, that the colour strength increases as the transfer temperature increases for every dye. The dye 5_a possesses a higher colour strength and dye 5_i possesses the lowest colour strength. This may be attributed to the difference in between the molecular weight of the two dyes where molecular weight of dye 5_a is 452 and molecular weight of dye 5_i is 471.5 and also may be due to the difference in the nature of substituents present on the dye molecules, where dye 5_j contains non-ionic groups in its molecular structure (NO₂, Cl). Dye 5_j has no ability for transfer printing and this may be due to its high-molecular weight (498) and the presence of two nitro groups within its molecular structure which are non-ionic polar group (Griffiths and Jones, 1977).

In case of screen printing technique

The printed PE and nylon 6 using the prepared dyes 5_{a-j} were thermo fixed at different temperatures 140, 160, 180 and 200°C, the fixation time was 2 min (Figures 5 and 6).

It is shown that, as the fixation temperature increases the colour strength increases for every dye. The bathochromic shift is observed in case of dye 5_j and this may be attributed to its molecular structure which possesses electron withdrawing groups (two nitro groups) and hydroxyl group.

Table I Physical and analytical data of synthesized dyes 5_{a-j}

Compound No.	Colour	Solvent	Yield (per cent)	m.p. °C	Molecular formula	M.Wt	λ max	FT-IR (KBr, μ cm ⁻¹)		Element analysis							
								SH	CN	Required (per sent)				Found (per cent)			
									C	H	N	S	C	H	N	S	
5 _a	Brown	Ethanol	80	227	C ₂₆ H ₂₀ N ₄ SO ₂	452	447	2,250	2,219	69.03	4.42	12.38	7.08	69.00	4.14	12.35	7.0
5 _b	Brown	Ethanol	82	247	C ₂₈ H ₁₈ N ₄ S	442	436	2,250	2,217	75.85	4.29	12.64	7.22	75.83	4.20	12.50	7.10
5 _c	Orange	Ethanol	78	252	C ₂₅ H ₁₈ N ₄ SO	422	432	2,450	2,216	71.09	4.27	13.27	7.58	71.00	4.21	13.10	7.50
5 _d	Yellow	Ethanol	89	222	C ₂₅ H ₁₈ N ₄ S	406	410	2,448	2,216	73.89	4.43	13.79	7.88	73.61	4.42	13.60	7.72
5 _e	Yellow	Ethanol	78	243	C ₂₄ H ₁₄ N ₄ SCl ₂	462	430	2,486	2,221	62.34	3.25	12.12	6.93	62.11	3.20	12.09	6.81
5 _f	Yellow	Ethanol	80	249	C ₂₄ H ₁₅ N ₄ SCl	426.5	425	2,448	2,215	67.53	3.52	13.13	7.50	67.20	3.41	13.01	7.39
5 _g	Yellow	Ethanol	85	287	C ₂₄ H ₁₅ N ₄ SBr	471	422	2,493	2,218	61.15	3.18	11.89	6.79	61.10	3.15	1.81	6.69
5 _h	Brown	Ethanol	81	267	C ₂₄ H ₁₅ N ₅ SO ₂	437	470	2,449	2,216	61.08	2.97	14.85	6.79	61.05	2.94	14.80	6.69
5 _i	Brown	Ethanol	83	244	C ₂₄ H ₁₄ N ₅ SCl	471.5	477	2,450	2,220	57.83	2.8	16.87	6.43	57.80	2.81	16.85	6.42
5 _j	Brown	Ethanol	87	288	C ₂₄ H ₁₄ N ₆ SO ₅	498	490	2,453	2,222	57.83	2.81	16.87	6.43	57.78	2.76	16.80	6.44

 Table II ¹H-NMR and mass spectra of the synthesised dyes 5_{a-j}

Compound no.	¹ H-NMR (ppm)	MS (M ⁺)
5 _b	7.3-8.2 (m, 17H, 2 C ₆ H ₅ , C ₁₀ H ₇), 12.9 (s, 1H, SH)	–
5 _c	2.5 (s, 3H, OCH ₃), 7.1-8.3 (m, 14H, 2 C ₆ H ₅ , C ₆ H ₅), 11.0 (s, 1H, SH)	421
5 _g	6.8-8.0 (m, 12H, 3 C ₆ H ₄), 11.7 (s, 1H, SH)	471
5 _i	7.3-8.0 (m, 13H, 2 C ₆ H ₅ , C ₆ H ₃), 12.8 (s, 1H, NH), 11.8 (s, 1H, SH)	471
5 _j	7.0-8.5 (m, 12H, 2C ₆ H ₅ , C ₆ H ₂), 8.4 (s, 1H, OH), 12.8 (s, 1H, SH)	–

Fastness properties

Fastness properties of both transfer and screen printed PE and nylon 6 are shown in Tables III and IV.

Wash fastness

It can be seen, from Tables III and IV, that the nature and/or the number of the substituents on the aromatic moieties of the dye molecules determined the wash-fastness of the printed fabrics, the wash fastness for all printed fabrics was between 3-4 for nylon and 4-5 for PE.

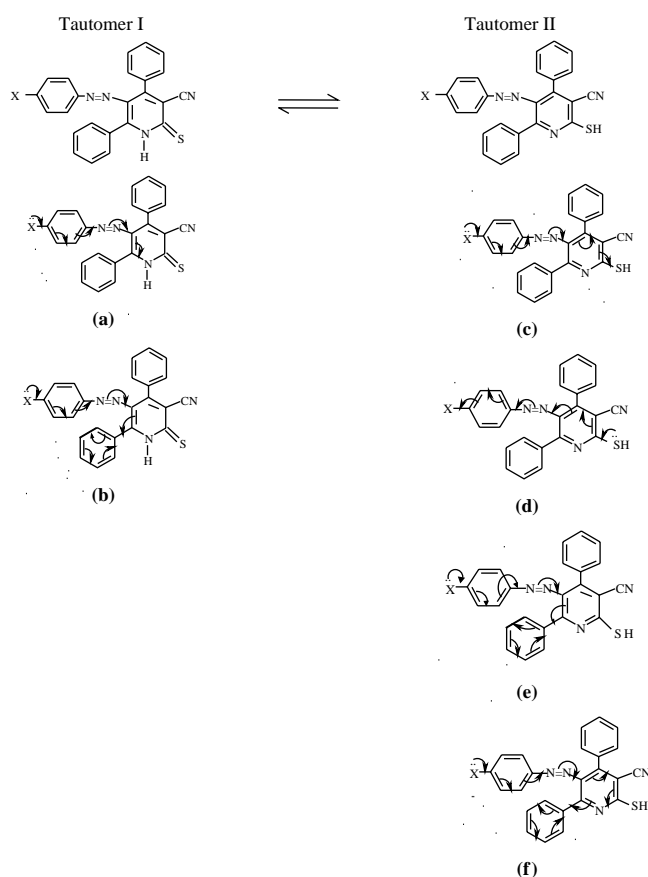
Perspiration fastness

The magnitude of the dye removal from PE and nylon fibres under influence of perspiration solutions (alkali and acidic) are shown in Tables III and IV. The result indicates that the dye removal could be dependent on the molecular weight of the dye and the binding forces between the dye and the fibre. Thus, dyes 5_g, 5_i and 5_j having higher molecular weight compared to other synthesised dyes, had greater fastness in perspiration solution, dyes 5_g, 5_i and 5_j had perspiration values 4-5 for nylon and 5 for PE.

Rub-fastness

Rub-fastness is an indication of the quantity of loosely adhered dye molecules on fibre. It can be seen, from Tables III and IV, that the higher value of rubbing fastness are associated with high-molecular weight of the dye, dyes 5_g, 5_i and 5_j had higher rubbing fastness than the other dyes. The value of rubbing fastness for dyes 5_g, 5_i and 5_j is 4 for nylon and 4-5 for PE.

Figure 2 Scheme 2



Light fastness

Light fastness is largely affected by the dye aggregation within the fibre. Generally, light fastness increases with the increase of the colour strength. It was found that the light fastness of most dyes range between 4 and 5.

Conclusion

New substituted 5-arylazopyridine-2(1H)thiones have been synthesised by the coupling of diazonium salts of aniline

Table III Colour strength and fastness properties of transfer printed polyester^a and nylon 6^b fabrics, using the synthesized dyes (5_{a-j})

Dye no.	K/S		Washing				Rubbing				Perspiration								Light fastness		
	Nylon	Poly-ester	Nylon		Polyester		Nylon		Polyester		Nylon				Polyester				Nylon	Poly-ester	
			Alt	St	Alt	St	Dry	Wet	Dry	Wet	Acidic	Alkali	Acidic	Alkali	Acidic	Alkali	Acidic	Alkali			
5 _a	5.38	5.29	3	3	4	4	3	3	4	4	4	4	4	4	4	5	4	4	5	4	4-5
5 _b	4.99	5	3	4	4	4	3	3	4	4	4	4	4	4	5	4	4-5	4-5	4	4	
5 _c	3.6	4.2	3	4	4	4	4	3	4	4	4	4	4	4	5	5	4	5	4	4	
5 _d	3.95	4.0	3	3	4	4	3	3	4	4	4	4	4	4	5	4	5	4-5	4	4	
5 _e	3.8	3.86	3-4	4	4	4	3	3	4	4	4	4	4	4	5	5	5	5	4	4	
5 _f	4.1	3.64	3	3-4	4	4	4	4	4	4	4	4	4	4	5	5	4	5	4	4	
5 _g	3.4	3.3	3	3	4	4	3	3	4	4	4	4	4	4	5	4-5	5	4-5	4-5	4-5	
5 _h	2.6	2.6	3	3-4	4	4	3-4	3	4	4	4	4	4	4-5	5	5	5	5	5	5	
5 _i	2	1.99	4	3-4	4-5	4-5	4	3-4	4	4	4	4	4	4-5	5	5	5	5	5-6	5-6	
5 _j	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	

Notes: ^aTemperature of transfer – 210°C; time of transfer – 45 s; ^btemperature of transfer – 190°C; time of transfer – 45 s

Table IV Colour strength and fastness properties of screen printed ^apolyester and nylon 6 fabrics, using the synthesised dyes (5_{a-j})

Dye no.	K/S		Washing				Rubbing				Perspiration								Light fastness	
	Nylon	Poly-ester	Nylon		Poly-ester		Nylon		Poly-ester		Nylon				Polyester				Nylon	Poly-ester
			Alt	St	Alt	St	Dry	Wet	Dry	Wet	Acidic	Alkali	Acidic	Alkali	Acidic	Alkali	Acidic	Alkali		
5 _a	6.0	5.9	4	4	5	5	4	3	5	4	4	4	4	4	5	4	5	4	4	4
5 _b	5.8	5.6	4	3	5	4	4	3	5	4	4	4	4-5	4	5	5	4	5	4	4
5 _c	5.1	4.8	4	4	4	4	4	3	5	4	4	4	4	4	5	5	5	4	4	4
5 _d	5	4.6	4	5	5	5	4	4	4	4	4	4	4	4	5	4	4	5	3-4	3-4
5 _e	5.1	4.7	3-4	4	5	4	4	3	5	4	4	4	4	4	5	5	5	5	4	4
5 _f	4.6	4.5	4	4	4	5	4	3	5	4	4	4	4	4	5	4	4	4	4	4
5 _g	4.4	4.2	3	4	5	5	4	4	5	5	4	4	4	4	5	4	5	5	4-5	4-5
5 _h	6.9	6.5	3	4	5	5	4	4	5	5	4	4	4-5	4	5	5	4	4	5-6	5-6
5 _i	7.3	7.0	4	4	5	5	4	3	4	4	4	4	4	5	5	5	4	5	5	5
5 _j	9.3	9.0	4	3-4	5	5	4	3	5	5	4	4	4-5	4-5	5	5	5	5	5	5

Notes: ^aFixation temperature – 200°C; fixation time – 2 min

Figure 3 Effect of transfer printing temperature on the color strength of the transfer printed PE using screen printed paper containing prepared dyes (5_{a-j}) transfer time at 45 s

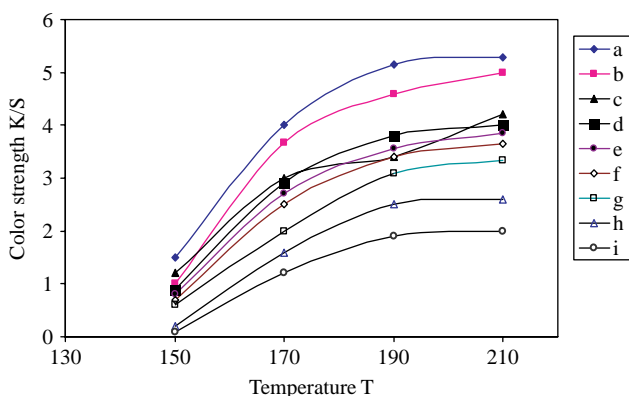


Figure 4 Effect of transfer printing temperature on the color strength of the transfer printed nylon 6 using screen printed paper containing prepared dyes (5_{a-j}) transfer time at 45 s

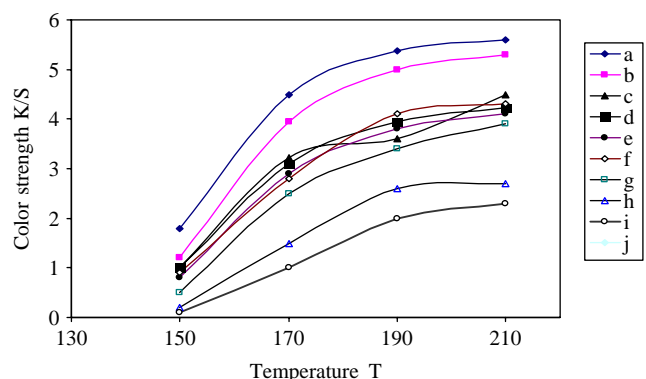


Figure 5 Effect of fixation temperature of the color strength of screen printed nylon 6 using silk screen printing technique of dyes (7_{a-j}) fixation time 2 min

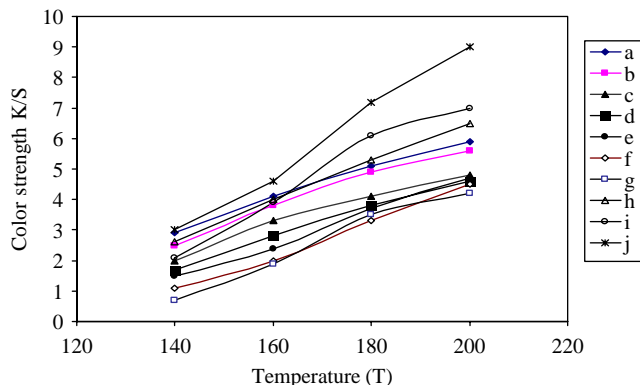
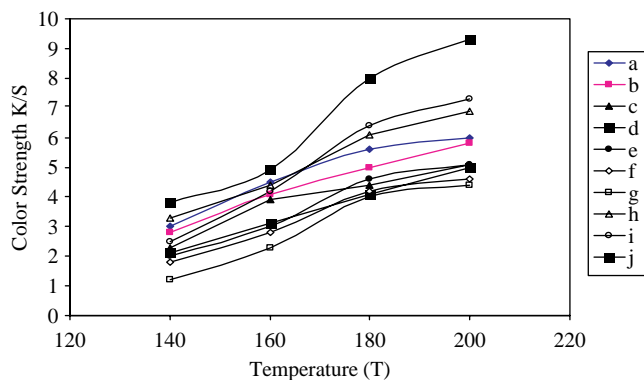


Figure 6 Effect of fixation temperature on the color strength of screen printed nylon 6 using silk screen printing technique of dyes (5_{a-j}) fixation time 2 min



derivatives with dibenzoyl methane (1,3-diphenyl-1,3-propanedione) to form arylhydrazone of dibenzoylmethane which reacts with cyanothioacetamide in boiling ethanolic sodium ethoxide to yield the 5-arylaazo-2(1H)-pyridinethione derivatives.

The structure of the substituted 5-arylazopyridine-2(1H)thione dyes has been established and confirmed on the basis of elemental analysis and IR spectra. The synthesised dyes are produced in good yield with different colours.

The substituents on the aromatic moiety of the synthesised dyes have a considerable effect on the colour strength. Such an effect depends on the electron mobility through the resonating system, which in turn depends on the type of the substituent.

Dye 5_a possesses the highest colour strength (K/S) and dye 5_i possesses the lowest colour strength (K/S). This may be attributed to the difference in structure and molecular weight of the two dyes. It has been found that transfer and screen printing of pyridinethione azo dyes 5_{a-j} on PE and nylon fabrics give good colour strength, good washing, rubbing and perspiration fastness.

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