

Studies on some Isoxazoline-Azo Compounds and their Colourant Performance and Fastness evaluation on Synthetic Fabric

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Abstract: Generally, heterocyclic compounds like isoxazolines are considered prolific for their biological activities. On the other hand, azo compounds are generally thought for their fascinating colourant properties. Nowadays, azo compounds having heterocyclic moiety are well-known in the colourant industry. Not only natural colourants, synthetic colourants also have added new dimensions to our lifestyle. Such compounds or their combinations add colours to life through innumerable ways. Azo group containing isoxazolines also exhibit wonderful colours. Hence, these compounds may be considered as colourants or dye molecules. Keeping such a point of view, Isoxazoline Azo Dyes(IADs) were synthesized. Their structural features were assessed by carbon, hydrogen, nitrogen-elemental analyses and by spectroscopic techniques like IR and ^1H NMR spectroscopies. After preparation of colourants, dyeing is also a step of the vital significance. Hence, IADs were dyed on synthetic fabric(polyester). Studies on light fastness and wash fastness properties of dyes were also carried out to judge the colorant parameters. Satisfactory colourant properties of synthesized substances were noticed on polyester fabric.

Key words : Colourant, IAD, polyester, dyeing, exhaustion, fixation, light fastness, wash fastness.

Introduction

From last fifty-sixty years, Isoxazolines which are heterocyclic compounds have been comprehensively studied by chemists and their biological activities are explored in detail[1-10]. These compounds are also reported as antinociceptive compounds[11].

In today's age, colouring of clothes particularly synthetic fabrics is a field of interest and activity for chemists and colour technologists. Azo group containing dye molecules have gained very strong position in the colourant market and textile industry. Good, fast and harmless colours are the needs of today's life. Azo dyes having heterocyclic moieties

can have some fascinating colours. So, isoxazoline azo dyes(IADs) were thought interesting to prepare. IADs were prepared from 1-(2-hydroxyphenyl)-3-phenylprop-2-en-1-one and dyeing was carried out on polyester fabric.

Retention of colour on the polyester depend on many factors like the structural characteristics of the colourant molecule and fiber, attachment of colourant molecule with fibers, the percentage of fixation of colourant on fibres, etc. Hence, performance of colourant is a very complex output of many microscopic and macroscopic factors. In this present study, IADs were studied for their colourant performance and fastness evaluation.

Experimental

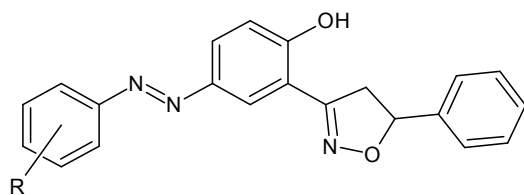


Fig.-1 General structure of IAD compound

IAD-1 : R = H, **IAD-2** : R = 4 - CH₃ , **IAD-3** : R = 4 - Cl,

IAD-4 : R = 3 - NO₂, **IAD-5** : R = 3 - Br, **IAD-6** : R = 2 - OCH₃.

For the synthesis of IADs, first of all 1-(2-hydroxyphenyl)-3-phenylprop-2-en-1-one was prepared. This reaction follows Claisen- Schmidt condensation. This compound was synthesized according to the method described in the literature[12-20]. The solution of 2-hydroxy acetophenone (0.01 mole) in absolute ethanol (40 ml) was warmed. Aldehyde (0.01 mole) was added to that warm solution and stirred to dissolve the aldehyde. Thus, the clear solution was obtained into which aqueous solution of sodium hydroxide (10 N, 1.5 ml) was poured gradually. This mixture was stirred at room temperature by a mechanical stirrer for 6-8 hours to get the mass which was decomposed with chilled HCl (50%, 4 ml). Thus, granules were obtained which were filtered. The granules were washed with aqueous solution of sodium bicarbonate (10%, 2 ml). The granules were dried and crystallized from absolute ethanol. In this way, crystals of the 1-(2-hydroxyphenyl)-3-phenylprop-2-en-1-one (C₁₅H₁₂O₂) (M.P. 87°C)(% of yield: 75%) were obtained.

In the second stage of synthesis, azo dyes were prepared according to the method described in the literature[21-30]. The general method which was adopted was as follows: Primary aromatic amine (0.01 mole) was added into the mixture of concentrated hydrochloric acid (2.8 ml to 3.6 ml) and water (2.8 ml to 3.6 ml) in a conical flask. A thermometer was placed in the flask and the flask was kept in an ice bath. The solution was gently stirred until the temperature of the solution reached to 0°C. Sodium nitrite (0.689 gm) was dissolved in water (6.9 ml). It was cooled to 0°C with the help of the ice bath. It was poured in small portions to the cold primary aromatic amine hydrochloride solution and the consequent

solution was vigorously stirred, keeping the temperature between 0°-5°C. Presence of a slight excess of nitrous acid was checked with the help of a potassium iodide-starch paper.

Finely powdered 1-(2-hydroxyphenyl)-3-phenylprop-2-en-1-one (0.01 mole) was added gradually in the solution of sodium hydroxide (0.564 gm) in water (15.8 ml) and absolute ethanol (15.8 ml). The mixture was stirred for 40-50 minutes. It was heated gently up to 50°-55°C with vigorous stirring to obtain clear solution and it was then cooled with intensive stirring. To the well-stirred solution of 1-(2-hydroxyphenyl)-3-phenylprop-2-en-1-one, the diazonium salt solution was added slowly maintaining the temperature between 0°-5°C. The mixture was then stirred for 3 hours at 0°-5°C to yield the dye. It was then filtered, washed with water, dried and was crystallized from absolute ethanol.

In the third stage, isoxazolines were prepared according to the procedure available in the literature[31-34]. Azo dye of previous stage(0.01 mole), hydroxylamine hydrochloride (0.02 mole) and potassium hydroxide (1.122 gm) in ethanol (35 ml) were refluxed for 11 to 12 hours. The reaction mixture was cooled, slightly acidified with glacial acetic acid and added into ice-cold water. The product thus obtained was filtered, washed with water, dried and crystallized from absolute ethanol.

The melting points of all these compounds were taken in open capillary glass tubes using paraffin bath and are uncorrected. The elemental analyses of carbon, hydrogen and nitrogen was performed using Carlo-Erba Analyser. The results satisfy the predicted structure of dyes. IR spectra were run on Shimadzu FTIR-8300 spectrophotometer and Perkin Elmer spectrophotometer using KBr pellets technique. Proton-NMR Spectra were recorded on a Bruker-Ultrashield(300 MHZ) spectrophotometer using DMSO-d₆ as a solvent and TMS as internal standard. The elemental analysis is shown in **Table-1**.

The spectral characteristics were as below :

IAD-1: IR: 3285, 3040.33, 1605.3, 1579, 1230 cm⁻¹, ¹H NMR : δ = 8.42 (s, 1H, -OH), 6.88-7.8 (m, 13H, Ar-H), 5.68-5.73, 5.15-5.21, 3.91-4.02 [three dd, -CH₂ - CH (isoxazoline)].

IAD-2: IR: 3230, 3031, 1606.4, 1570, 1240 cm⁻¹, ¹H NMR: δ = 2.3 (s, 3H, -CH₃), 8.5 (s, 1H, -OH), 6.72-8.38 (m, 12H, Ar-H), 5.66-5.72, 5.15-5.20, 3.90-4.01 [three dd, -CH₂ -CH(isoxazoline)].

IAD-3: IR: 3187, 3049.6, 1617, 1572, 1241.3 cm^{-1} , $^1\text{H NMR}$: δ = 8.28 (s, 1H, -OH), 7.1-8.1 (m, 12H, Ar-H), 5.8-5.87, 5.28-5.39, 4.11-4.18 [three dd, -CH₂-CH (isoxazoline)].

IAD-4: IR: 3300, 3050.86, 1613.6, 1575.81, 1233.8 cm^{-1} , $^1\text{H NMR}$: δ = 8.32 (s, 1H, -OH), 6.87-8.25 (m, 12H, Ar-H), 5.75-5.79, 5.13-5.22, 3.8-4.03 [three dd, -CH₂-CH (isoxazoline)].

IAD-5: IR: 3301.18, 3014, 1599.8, 1579.2, 1243.1 cm^{-1} , $^1\text{H NMR}$: δ = 8.29 (s, 1H, -OH), 6.91-7.84 (m, 12H, Ar-H), 5.14-5.21, 5.56-5.68, 3.8-3.88 [three dd, -CH₂-CH (isoxazoline)].

IAD-6: IR: 3205.47, 3026.11, 1607.5, 1581, 1235 cm^{-1} , $^1\text{H NMR}$: δ = 3.4 (s, 3H, -OCH₃), 8.3 (s, 1H, -OH), 6.5-7.8 (m, 12H, Ar-H), 5.60-5.68, 4.8-5.03, 3.76-3.86 [three dd, -CH₂-CH (isoxazoline)].

Dyeing is a very crucial facet in determining the performance of a colourant on fabric. The IADs which were synthesized used for dyeing on synthetic fabric (polyester). The general method that was adopted for the dyeing polyester is as follows: In a conical flask containing distilled water (100 ml), synthetic fabric pattern (2.0 gm) was introduced. $\frac{2}{3}$ portion of the flask was immersed into a thermostate bath and temperature was maintained at 80°C for 10 minutes. The fabric pattern was then taken out of the flask, squeezed very

well and dried. The pretreated polyester fabric thus prepared was used further in the process of dyeing.

For 2% dyeing, IAD under study (40 mg) was dissolved in possible minimum quantity of DMF. The IAD solution was then added with continuous stirring into a dye pot containing the solution of dispersing agent (sodium lauryl sulphate, 100 mg) in distilled water to obtain a fine aqueous dispersion of the IAD. The total volume of the solution in the dye bath was 100 ml. Thus, the MLR was maintained 1:50. The dye bath was set at 60°C and the same temperature was maintained for 10 minutes. The temperature was then raised up to 70°C. The temperature from 70°C to 130°C was raised within 1 hour at the rate of 1°C per minute. Dyeing was carried out at this temperature for 1 hour. After this stage, the dye bath was cooled. The dyed fabric pattern was washed well with water (100 ml). This water was collected in a volumetric flask containing DMF (40 ml) and the residual dye liquor was collected from the dye bath. The total volume of the solution in the flask was made 250 ml by the further dilution with water. 25 ml of this solution was pipetted out and further diluted to 50 ml with water. 1 ml of this consequent solution was further diluted to 10 ml with DMF. The absorbance of the resultant solution was measured. Thus, the dyed polyester fabric was obtained. It was then rinsed and scoured in a detergent (Lissapol) solution (100 ml, 0.2 %) at 50°C for 20 minutes. The % of exhaustion and fixation were determined according to the known methods[35-38].

Table - 1 : IAD Compounds

Comp.	Molecular Formula	M.P. (°C)	% of yield	Elemental Analysis Found(Calcd.)%		
				C	H	N
IAD-1	C ₂₁ H ₁₇ N ₃ O ₂	73	58	73.40(73.45)	5.01(4.99)	12.22(12.23)
IAD-2	C ₂₂ H ₁₉ N ₃ O ₂	69	58	73.95(73.93)	5.37(5.32)	11.72(11.75)
IAD-3	C ₂₁ H ₁₆ N ₃ O ₂ Cl	158	72	66.73(66.75)	4.27(4.26)	11.11(11.12)
IAD-4	C ₂₁ H ₁₆ N ₄ O ₄	108	65	64.95(64.94)	4.16(4.15)	14.40(14.42)
IAD-5	C ₂₁ H ₁₆ N ₃ O ₂ Br	137	61	59.72(59.73)	3.83(3.81)	9.91(9.95)
IAD-6	C ₂₂ H ₁₉ N ₃ O ₃	128	58	70.71(70.76)	5.14(5.12)	11.21(11.25)

Comp. : Compound, M. P. : Melting Point, Calcd. : Calculated.

Table – 2 : Colourant performance and fastness properties on polyester fabric

Compound	Shade on fabric	% of exhaustion from dye bath	% of fixation on fabric	Light Fastness	Wash Fastness
IAD-1	Light orange	74.12	65.69	4	3
IAD-2	Mid orange	70.75	67.98	3	3
IAD-3	Mid orange	69.25	60.83	3-4	2-3
IAD-4	Deep orange	81.15	62.04	4	3
IAD-5	Yellowish orange	74.87	74.62	3-4	3
IAD-6	Light orange	73.12	63.93	4	2-3

For the present study, light fastness and wash fastness tests were performed on dyed synthetic fabrics and fastness values were obtained. For determining light fastness, general method according to British Standards(BS) 1006-1978 was followed[39-49] and for the grade determination of the wash fastness, general method according to Indian Standards : IS : 765-1979 was followed[50-54]. All the IADs under study were rated in the different grades of light and wash fastness. The data thus obtained about shades of colourants, extent of consumption of colourants during dyeing, amount of fixation of colourants on polyester and fastness grades of colourants are given in Table - 2.

Results and Discussion

Good colourant substance should be consumed very well from the dye bath. All professional and highly skilled colourists are always agree on this point, but a healthy consumption from the dye pot is not everything in colour quality determination. The colourant material should be fixed properly on the synthetic fabric. Thus, the colour of the dyed fabric must be able to survive against two normal external factors like light and washings. High-quality colour should not fade easily against these parameters. So, fastness of colourants on the fabric towards these external agencies is also an imperative criterion in the determination of quality of colourant material. Under

the light of these facts, results of this research work can be described as follows: The IADs used to colour synthetic fabrics gave orange shades. The exhaustion of IAD from the dye bath on polyester fabric was in the range of 69.25% to 81.15%. The fixation study of dyes on polyester fibres exhibits good fixation characteristics of the IADs. The percentage of IADs fixed on the polyester fabric range from 60.83 % to 74.62%. So, IADs showed good exhaustion and fixation characteristics for polyester fabric.

The fastness properties of colourants are affected by numerous pre-consumer and post-consumer factors. All the colourants possess unique fastness properties. The usage of fabric also determines the importance of particular fastness property, e.g. Generally, for the clothing synthetic fabrics, higher importance is given to wash fastness while for upholstery synthetic fabrics, higher weight is given to light fastness.

Here, the light fastness for IAD on the polyester fabric was found from moderate fastness to fair fastness. The wash fastness for IADS on polyester fabric was found from fair to good.

In this way, IADs showed a good penetration and affinity for polyester and nylon fabrics. A remarkable levelness of the colour was observed after washing and drying the fabrics. These all properties indicate that IADs may be used as colourant material for polyester.

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