



Synthesis, Characterization and Textile Application of Novel Disperse Dyes

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ABSTRACT

Two novel alkali clearable disperse dyes were synthesized. 4-fluorosulfonyl aniline and 2-nitro-4-fluorosulfonyl aniline were utilized as the diazo components and 2-naphthol as the coupling component. The DSC, FT-IR, ¹H-NMR and UV-Vis analyses were used to characterize the synthesized intermediates and dyes. The synthesized dyes were utilized to dye polyester fibers and their dyeing properties were studied. In addition, hydrolysis kinetics of the synthesized dyes in alkaline media was investigated. The results showed that the synthesized dyes had good properties on polyester fabrics. As a result of the synthesized dyes hydrolysis in alkaline media, the reduction clearing process is supposedly replaced by alkali-clearing process. The elimination of sodium hydrosulfite leads to the reduction of environmental pollution. One of the main advantages of replacing reduction-clearing method by alkali-clearing is preventing toxic and carcinogenic aromatic amines to be produced. Prog. Color Colorants Coat. 7(2014), 155-163. © Institute for Color Science and Technology.

1. Introduction

Consumption of the azo disperse dyes is more than 50% of the total amount of disperse dyes produced in the world [1]. These dyes are capable of dyeing di- and tri-cellulose acetate, polyamide, acrylic and polyester fibers [2]. The main use of disperse dyes is in dyeing polyester fibers. Disperse dyes have a limited solubility in water; therefore, they are applied as aqueous dispersions in dyeing procedures. For this purpose, anionic and nonionic dispersing agents based on fatty alcohol ethylene oxide compounds were used [3]. As a disadvantage, disperse dyes may deposit on the substrate surface and decrease the quality of dyeing, i.e. the

brightness as well as the wash and rubbing fastness of dyed fabrics will be reduced [4]. In order to solve this problem, the reduction clearing of dyed fabrics deems necessary. The process will remove loosely attached dye particulates from the fiber surface. Here, sodium hydrosulfite is used as the reducing agent. In the end, the azo groups of dyes will be reduced to either colorless or weakly colored species containing aromatic amines some of which highly toxic and carcinogenic. In addition, the presence of sodium hydrosulfite within the dye bath leads to high biological oxygen demand values [5]. In this respect, alkali-clearable azo disperse dyes have been

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synthesized and suggested for dyeing polyester fibers with acceptable fastness properties. These dyes can be hydrolyzed in alkaline media becoming water-soluble dyes. Therefore, they will no longer deposit on the fiber surface. Alkali-clearable disperse dyes minimize effluents discharge as well as energy and material consumption during the dyeing process [6]. This method is reasonably simple and substantially cheaper than reduction clearing.

Recently, synthesizing new environmental friendly dyes and modifying the dyeing methods have been considerably increased. Thus, synthesis and application of alkali-clearable azo disperse dyes have been reported [4, 6, 7]. Ester, thiophene and fluorosulfonyl groups have been applied in these three dye molecules as alkali-clearable groups. Azo disperse dyes containing phthalimide groups undergo a ring opening reaction and become soluble in water under relatively mild conditions while the azo linkage remains protected [8]. Azo dyes possessing ester groups based on naphthalimides were reported as alkali clearable disperse dyes for dyeing polyester fabrics [9]. In these dyes, the ester group is hydrolyzed and converted to a water-soluble carboxylate group by alkali hydrolysis that could be easily washed off after dyeing [10]. It has been reported that these dyes have good dyeing properties and build up with an excellent wash fastness on polyester fabrics.

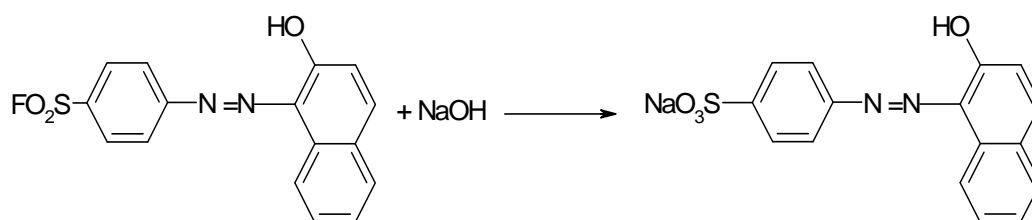
Another group of alkali-clearable dyes are fluorosulfonyl derivatives. In this type, the fluorosulfonyl group can be converted to the water soluble $-\text{SO}_3\text{Na}$ group according to equation 1 [4]. In this process, fluorosulfonyl aniline derivatives were utilized as the diazo components and aryl amines as coupling agents. Also, a series of naphthalimide based alkali-clearable azo disperse dyes containing fluorosulfonyl group prepared by 4-fluorosulfonyl anilines and their derivatives as the

diazo component and 4-hydroxy-N-3-methoxypropyl-1,8-naphthalimide as the coupler [11]. Azo naphthalimide dyes can be applied on polyester fabrics with high temperature method and give a range of color varying from red to violet hues [12]. Utilizing fluorosulfonyl group in these types of disperse dyes results in a desirable build up, excellent wash fastness while reducing BOD and production of aromatic amines through eliminating sodium hydrosulfite.

In this paper, synthesis and characterization of novel alkali-clearable azo disperse dyes are studied. These dyes have fluorosulfonyl and 2-naphthol groups in their structure. The DSC, FT-IR, $^1\text{H-NMR}$ and UV-Vis analytical techniques were used to characterize the synthesized intermediates and dyes. The synthesized dyes were applied onto polyester and fastness properties of dyed fibers were examined. Additionally, the hydrolysis kinetics of dyes in alkaline media was studied.

2. Experimental and apparatus

All materials used for synthesis of dyes were obtained from Aldrich and Merck Companies and were used without further purification. $^1\text{H-NMR}$ spectra were recorded on a Bruker AVANCE-300MHZ. UV-Vis spectra were measured using a Cecil 9200 Spectrophotometer. IR Spectra (KBr) were recorded on a Nicolet 470/670/870 spectrophotometer. The melting point of intermediates and dyes were measured by DSC 2010 TA instrument. The reflectance spectrophotometer data of dyed fabrics were obtained by Gretag MacBeth 7000A spectrophotometer. Dispersions of dyes were prepared in ball mill and were filtered off through a 5- μm Micro-Prazisons Sieve Fritsch filter. Dyeing Polyester fabrics was carried out by HT (high temperature) method using rotadyer apparatus (Nasaj sanat yazd).



Equation 1

2.2. Synthesis processes of intermediates and dyes

The synthesis procedures of intermediates and dyes are depicted in Figure 1. These processes include hydrolysis, nitration, diazotization and coupling reactions.

2.3. Synthesis of intermediates

2.3.1. Synthesis of 4-fluorsulfonylaniline

N-acetylsulfanyl chloride (50 mmol) was refluxed in a solution of potassium fluoride (18 g), water (25 ml) and *p*-dioxane (25 ml) for 3 h. The product was then drowned into ice and washed with warm water until it precipitated. The resulting precipitate was stirred in a mixture of ethanol (10 ml) and hydrochloric acid (10 ml) and again refluxed for 1 h. The mixture was drowned into ice, neutralized, and filtered off to a white powder.

2.3.2. Synthesis of 2-nitro-4-fluorosulfonyl aniline

A solution of N-acetylsulfanyl fluoride (0.01 mol) in sulfuric acid (12 ml) was prepared. This solution was cooled down to 4 °C and another mixture of nitric acid (1.3 ml) and sulfuric acid (1.4 ml) was dropwise added to it at 5 °C. After 1 h, the mixture reaction was drowned into ice. The precipitate was filtered, washed and dried. This solid was added to a solution of hydrochloric acid and ethanol (5 ml/5 ml) and refluxed for 1 h. The product of reaction was filtered off and recrystallized in ethanol.

The yellow needle-like crystals were collected afterwards.

2.4. Synthesis of dyes

2.4.1. Diazotization

Diazotation of 4-Fluorosulfonylaniline (10 mol) was carried out in a solution of hydrochloric acid (3.4 ml), water (35 ml) and sodium nitrite (10 mmol) at 0–5 °C. After 5 h of stirring, the saturated solution of sodium acetate was added and the pH value of the diazo liquor was adjusted to approximately 5–6.

Diazotization of 2-nitro-4-fluorosulfonyl aniline was performed with nitrosyl sulfuric acid. For this purpose, 2-nitro-4-fluorosulfonyl aniline (0.02 mol) was dissolved in a solution of acetic acid and propionic acid (4:1, 50 ml), added to another solution of sodium nitrite (0.02 mol) and sulfuric acid (30 ml) at 5-10 °C. The mixture was stirred at this temperature for 3h. The obtained clear diazonium salt solution was used immediately in the coupling reaction.

2.4.2. Coupling

The diazonium liquor was added to a solution containing 10 mmol of the coupling component (2-naphthol). After 4–5 h of stirring, the coupling reaction was completed and the precipitated dyes were filtered, washed and dried. The dyes were purified by recrystallization in methanol. The final product was an orange powder.

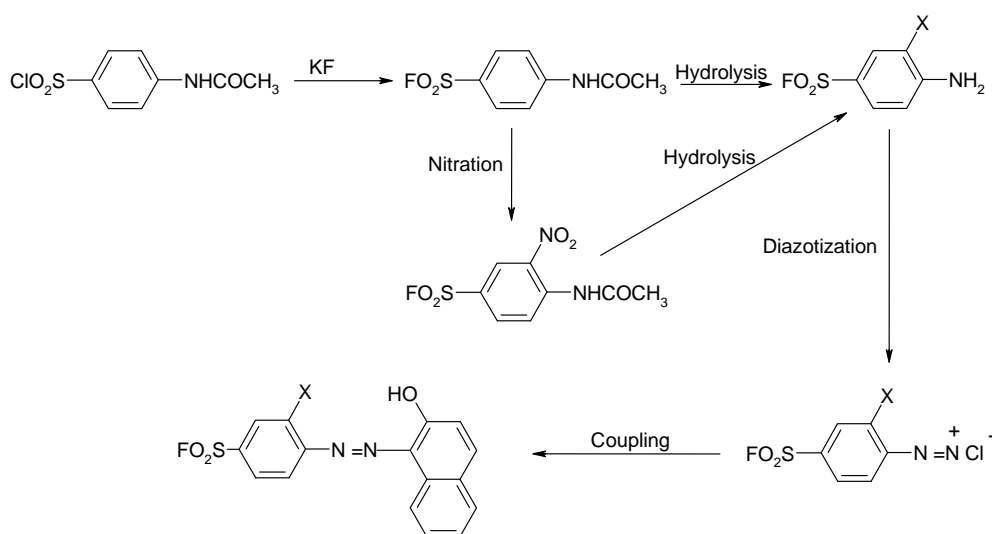


Figure 1: Chemical structure of the synthesized dyes; dye 1 (X= H) and dye 2 (X=NO₂).

2.5. Alkaline hydrolysis kinetics

For studying the hydrolysis kinetic of the synthesized dyes in alkaline media, 0.1 g of each synthesized dye samples was added to a solution of sodium carbonate 20% at 80 °C. The concentration of hydrolyzed and their corresponding parent dyes were obtained by spectrophotometry; in this process, 1 ml aliquots were withdrawn from the hydrolysis reaction every five minutes and analyzed.

2.6. Preparation of dye dispersions

Lyoprint EV, a nonionic dispersing agent, was used to prepare the dispersions. For this purpose, 1 g of pure dye, 2 g of dispersing agent and 30 ml of water were milled in a ball mill for 24 h. The obtained mixture was diluted with 100 ml of water and filtered off.

2.7. Textile applications: dyeing

Polyester fabrics (1 g) were pretreated with a nonionic detergent, (5 g/L) at 80 °C for 20 min prior to being used for the dyeing. The samples were dyed by HT (high temperature) method in acetic acid (pH = 4-5) and a L:G ratio of 50:1. The build up properties of the synthesized dyes on polyester fabrics were obtained using different dyeing concentrations (0.1, 0.3, 0.5, 0.7, 1, 1.5, 2 and 4 % o.m.f). Dyeing was carried out by raising the dye bath temperature from 30 °C to 90 °C (2 °C/min) followed by another ascend from 90 °C to 130 °C (1 °C/min) where it will be hold for 60 min, finally cooling down to 70 °C at a 3 °C/min rate.

Some of the dyed samples were reduction cleared using sodium hydrosulfite (2 g/L), sodium hydroxide (1g/L) and detergent (1 g/L). Other samples were alkali cleared by utilizing sodium carbonate (20 g/L) and detergent (1 g/L) with an L:G of 50:1 (Figure 2).

2.8. Determination of fastness properties of dyed fabrics

The wash fastness of dyed fabrics was tested according to ISO 105C06/C2S. For this purpose, undyed cloths in contact with the dyed samples were treated with solution containing detergent (4 g/L), perborate (1 g/L) and sodium carbonate (1 g/L) at 60 °C for 30 min. For evaluating the light fastness, the samples and the wool standards were mounted on a frame partly covered on an opaque sheet, leaving the other half exposed to xenone lamp (ISO 105/B02). The heat fastness property of the samples was measured according to ISO 105/P01.

3. Results and discussion

The 4-fluorosulfanyl aniline and 2-nitro-4-fluorosulfonyl aniline were used for producing the dyes. Since 4-chlorosulfonylaniline is very unstable under nitration and fluorination reactions, the 4-fluorosulfonyl aniline was synthesized using *N*-acetylsulfanyl chloride [13]. Therefore, *N*-acetylsulfanyl chloride was reacted with potassium fluoride to obtain *N*-acetylsulfanyl fluoride. The deacetylation of product was carried out in weak acidic media using hydrochloric acid under reflux because of fluorosulfonyl group's susceptibility to alkaline medium. The pure *N*-acetylsulfanyl fluoride was obtained from recrystallization in methanol (49.6%). The synthesis of 2-nitro-4-fluorosulfonyl aniline was carried out using two methods. In first method, *N*-acetylsulfanyl chloride is nitrated in a mixture of concentrated nitric and sulfuric acids. Then several unit processes such as fluorination and hydrolysis were carried out. By this method, the yield and purity of the product is low due to the formation of by-products. In second method, the fluorination of *N*-acetylsulfanyl chloride is followed by nitration and deacetylation. Recrystallization using ethanol yielded pure compound in good yield.

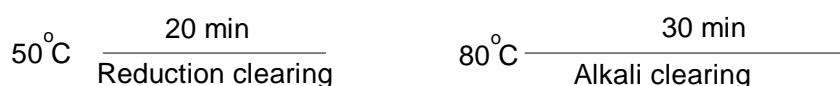


Figure 2: Profiles of reduction and alkali-clearing processes.

Characterization of both 4-fluorosulfonyl aniline and 2-nitro-4-fluorosulfonyl aniline were carried out by DSC, FT-IR and $^1\text{H-NMR}$ (Table 1).

Synthesis of dye 1 was done by diazotization of 4-fluorosulfonyl aniline using hydrochloric acid and sodium nitrite at 5 °C. The diazonium salt was coupled with 2-naphthol in weakly acid media in good yields (65%). Since 2-nitro-4-fluorosulfonyl aniline is relatively a weak base, therefore, its diazotization was carried out with nitrosyl sulfuric acid. Adding diazonium salt to 2-naphthal resulted in dye 2 with relatively good yields (62%). The FT-IR, $^1\text{H-NMR}$ and melting point of dyes are shown in Table 2.

3.1. Spectrophotometric properties

The correlation between the color and composition is a highly interesting aspect of dye development and commercialization [14]. In this respect, the wavelength of maximum absorption (λ_{max}), the molar extinction coefficient (ϵ_{max}) and absorption profile are important factors for dyes. The spectrophotometric properties of the synthesized dyes were recorded in chloroform and DMF solutions (Table 3). The λ_{max} of dyes showed that the presence of nitro as an electron acceptor group on diazo component caused a bathochromic shift. It is well known that the color of dyes is influenced by the introduction of additional donor groups in the coupling component and acceptor groups on diazo components [15, 16]. The molar extinction coefficients of dyes 1 and 2 were 14941 and 15194 $\text{L mol}^{-1} \text{cm}^{-1}$, respectively.

Table 1: FT-IR, $^1\text{H-NMR}$ data and melting points of intermediates.

Materials	FT-IR	$^1\text{H-NMR}$	Melting point (°C)
4-fluorosulfonyl aniline	FT-IR ν (cm^{-1}) (KBr): 3397, 3492 (N-H str. Primary Amine), 1595, (C=C str. Aromatic).	$^1\text{H-NMR}$ (CDCl_3): δ 4.2 (2H,s,NH ₂), 6.72-6.75 (2H, 2-H, 6-H), 7.76-7.79 (2H, 3-H, 5-H).	70
2-nitro-4-fluorosulfonyl aniline	FT-IR ν (cm^{-1}) (KBr): 3368, 3481 (N-H str. Primary amine), 1559(C=C str Aromatic), 1403, 1514 (NO ₂ str. Unsym. and sym.).	$^1\text{H-NMR}$ (CDCl_3): δ 4.8 (2H,s,NH ₂), 7.0-7.03(1H, 6-H), 7.87-7.9(1H,5-H), 8.86 (1H, 3-H).	150.3

Table 2: FT-IR, $^1\text{H-NMR}$ data and melting points of dyes.

Dye	FT-IR	$^1\text{H-NMR}$	Melting point(°C)
1	FT-IR ν (cm^{-1}) (KBr): 3451 (OH str.), 1411(N=N str.).	$^1\text{H-NMR}$ (CDCl_3): δ 6.6-6.7(1H, 5-H), 7.4-7.5(1H, 4-H), 7.5-7.6(2H, 6-H, 7-H), 7.6-7.7(1H, 8-H), 7.7-7.8(2H, 2'-H, 8'-H), 8.3-8.4(1H, 3-H), 16(1H, OH).	219
2	FT-IR ν (cm^{-1}) (KBr): 3440 (OH str.), 1400(N=N str), 1493, 1353 (NO ₂ str. Unsym. and sym.).	$^1\text{H-NMR}$ (CDCl_3): δ 6.6-6.7(1H, 5-H), 7.4-7.5(1H, 4-H), 7.5-7.6(2H, 6-H, 7-H), 7.6-7.7(1H, 8-H), 8.1-8.2(1H,3-H), 8.3-8.4(1H, 6'-H), 8.6-8.7(1H, 5'-H), 9(1H, 3'-H), 16.5(1H, OH).	138

Solvatochromism implies the effect of a solvent upon the color of a dye. The solvatochromism is determined by the λ_{\max} difference of a dye in two different solvents. Generally, there are two kinds of solvatochromism, i.e. positive and negative. In positive solvatochromism, the first excited state of dye has more polarity than the ground state. Negative solvatochromism is relatively rare and observed when the first excited state has less polarity than the ground state. However, comparing λ_{\max} values of dyes in chloroform and DMF shows a negative solvatochromism for dyes **1** and **2** ($\lambda_{\max} = -17$ and -45 for dyes **1** and **2**, respectively). This finding is in good agreement with the work of other research groups in case of azonaphthalimides that their coupler is 2-naphthol [17].

3.2. Dyeing and fastness properties

The synthesized dyes were applied to polyester fibers under high temperature conditions and different

concentrations of 0.1, 0.3, 0.5, 0.7, 1, 1.5, 2 and 4% of dyes on the weight of fiber. 0.5% Acetic acid (pH= 4.5-5) was added and the dyeing was carried out in accordance with Figure 2. For investigating the dyeing properties of the synthesized dyes on the polyester fabrics, a dispersion of dyes was prepared. These dispersions were used for dyeing polyester fibers by HT method. For elaborating the buildup of the synthesized dyes, the K/S was calculated by using the Kubelka-Monk Equation (equation 2) [16, 17]:

$$K/S=(1-R)^2/2R \quad (2)$$

where K, S and R are absorption coefficient, scatter coefficient and reflectance of dyed samples, respectively. Figure 3 shows the build up values of dyes on polyester fabrics.

Table 3: The spectrophotometric properties of the synthesized dyes.

Dye	Chloroform	DMF	Chloroform	DMF
	λ_{\max} (nm)	λ_{\max} (nm)	ε (L/mol. cm)	$\Delta\lambda_{1/2}$ (nm)
1	479	434	14941	105
2	490	473	15194	125

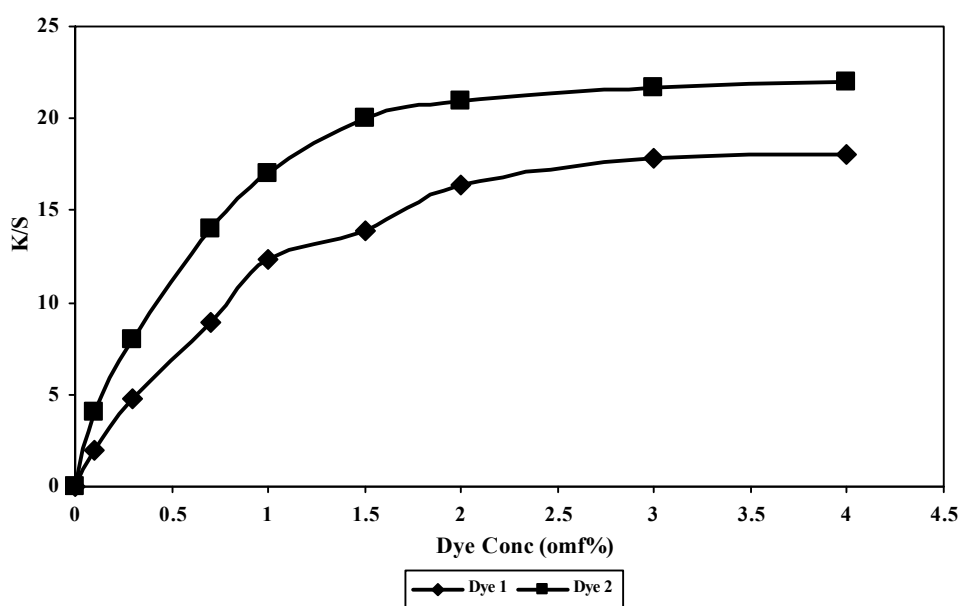


Figure 3: Build up of the synthesized dyes.

The color strength of the dyed fabric reached the saturated state at the dye concentration of ~2% omf. The dyed fabrics with both of dyes showed relatively good build up and levelness.

The results showed that the dyed polyester fabrics exhibit relatively good light fastness and high heat properties. Because fluorosulfonyl group of these dyes has been converted to water-soluble sulfonic acid group by alkali-hydrolysis which can be easily removed from fiber surface. The mechanism of dye hydrolysis in alkali media is illustrated in Figure 4. In this reaction, fluorosulfonyl group of dyes can be hydrolyzed to the corresponding acid by nucleophilic bimolecular substitution (SN²) mechanism under alkaline conditions [17]. In comparison with reduction- and alkali-cleared samples, the uncleared dyed samples showed relatively poor wash fastness (Table 4). Hydrolysis of dyes with

fluorosulfonyl group in alkaline media results in the replacement of reduction clearing process. In other words, elimination of sodium hydrosulfite will cause the reduction of environmental pollution. The advantage of replacing reduction-clearing method by alkali-clearing process eliminates the production of toxic and carcinogenic compounds [18-20].

3.3. Kinetic study of dye hydrolysis

Since the chemical compounds with sulfonyl fluoride group can be hydrolyzed to the sulfonic group under alkaline media (Figure 1), the synthesized dyes were treated with sodium carbonate (20 g/L) at 80 °C. The decrease of primary dye concentration was measured by spectrophotometric method. The absorption maxima for dyes 1 and 2 were 430 and 467 nm, respectively.

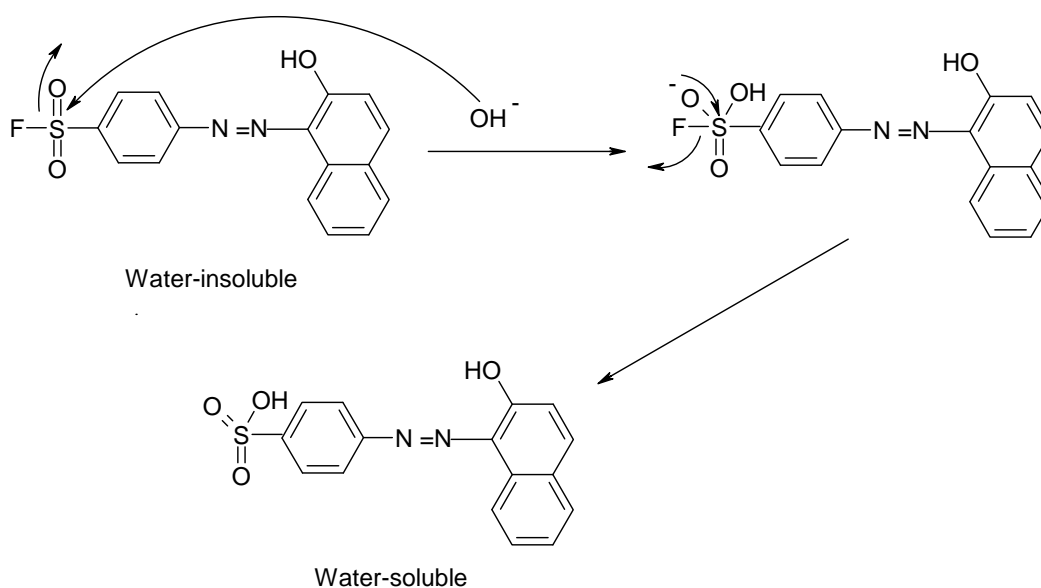


Figure 4: Hydrolysis mechanism of the synthesized dyes in alkaline media

Table 4: Color fastness of dyed fabrics (A: wash fastness (staining), B: wash fastness (change), C: cotton, P: polyester, N: nylon, L.F. = light fastness, H.F. = heat fastness, RC= reduction cleared, AC= alkali cleared).

Dye	None			B	RC			B	AC			H.F.	L.F.	
	A				A				A					
	C	P	N		C	P	N		C	P	N			
1	4	4	2	3-4	5	5	4	5	5	5	3-4	5	3-4	5
2	4	4	2-3	3-4	5	5	4	5	5	5	4	5	4	5

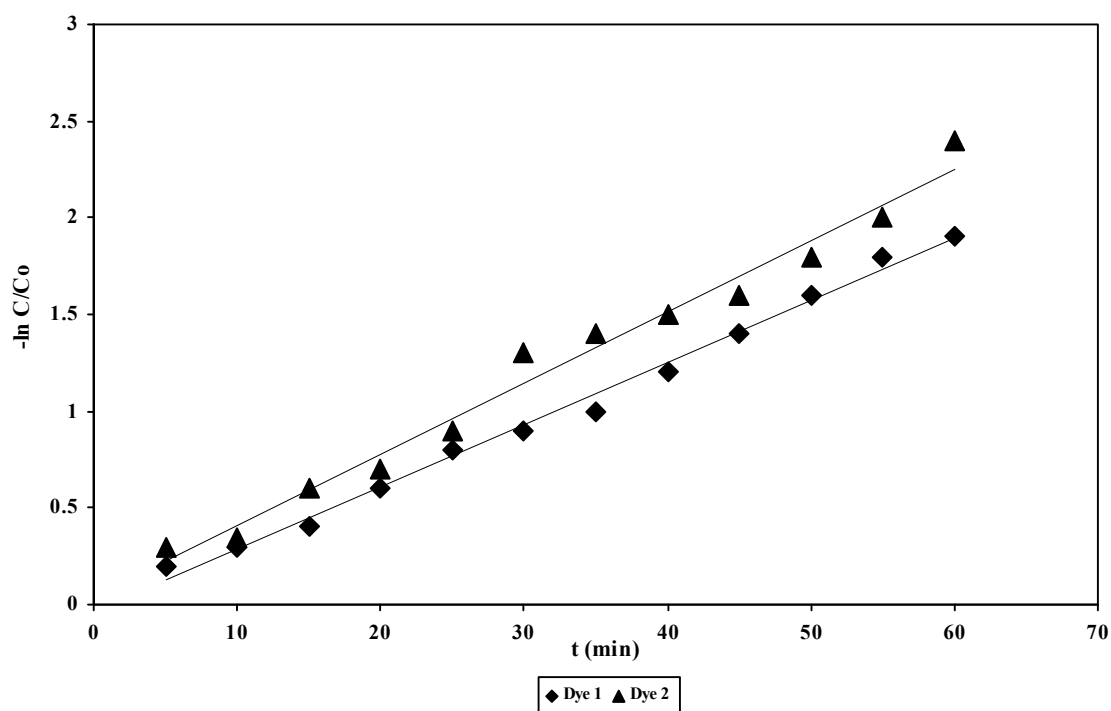


Figure 5: Kinetic study of the hydrolysis reaction of the synthesized dyes.

The rate of concentration decrease of the primary dye in the alkaline solution can be illustrated by the first-order kinetics (equation 3):

$$-d[C]/dt = k[C] \quad (3)$$

where $[C]$ and k are the dye concentration and rate constant of hydrolysis, respectively.

$$-\ln(C/C_0) = k t \quad (4)$$

where C_0 and C are the dye concentration at the beginning and moment t of the reaction, respectively.

As it is clear from Figure 5, the dye concentration decreases linearly towards the elapse of time. This means that the first-order kinetics to dye is operative. The rate constants of the synthesized dyes 1 and 2 in alkaline media were $0.040 \text{ (min}^{-1}\text{)}$ and $0.0328 \text{ (min}^{-1}\text{)}$, respectively.

4. Conclusions

Two novel azo disperse dyes were synthesized and their spectral properties were investigated. These dyes have a fluorosulfonyl group in their structures. The UV-Vis absorption data for the synthesized dyes showed that the presence of a nitro group in diazo component caused bathochromic shift. The dyeing of polyester fibers with the synthesized dyes using HT (high temperature) method resulted in shades from orange to orange-red with good color fastnesses and build up properties.

The hydrolysis kinetics of the dyes in alkaline media showed that the both dyes have potentially alkali clearable properties with different rates of reaction. Hydrolysis of the synthesized dyes with a fluorosulfonyl group in their structure in alkaline media provides replacement of reduction clearing process. The elimination of sodium hydrosulfite will cause reduction of environmental pollution.

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