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## ORIGINAL ARTICLE

# The comparison of spectra and dyeing properties of new azonaphthalimide with analogues azobenzene dyes on natural and synthetic polymers

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## KEYWORDS

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Polyamide fabrics;  
Dyeing;  
4-Aminonaphthalimide;  
Color fastnesses

**Abstract** The aim of the present research was to prepare new acid dyes based on naphthalimides. In this respect a series of monoazo acid dyes have been obtained using 4-amino-N-methyl (alternatively N-butyl)-1,8-naphthalimide, aniline and p-nitroaniline as diazo components. 2-Naphthol-6-sulfonic acid (Schaeffer's acid) and 1-naphthol-8-amino-3,6-disulfonic acid (H-acid) were used as coupling components. The spectrophotometric properties of the synthesized dyes were investigated in various solvents and compared with analogues azobenzene dyes. It is found, when acid dyes are applied in various solvents and different pH, additional bathochromically shifted bands of different intensity appear in the electronic spectra. This effect is caused by the occurrence of the equilibrium of azo and hydrazone forms in the dyes. The synthesized acid dyes were applied on wool fabrics in order to consider their dyeing properties, fastnesses and the obtainable color gamut. The synthesized dyes represented that they have the ability of dyeing wool and polyamide fabrics and give red to violet hues with good wash, medium light, and good milling and perspiration fastnesses.

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## 1. Introduction

In a classification method, colorants are categorized according to certain common chemical structural features. The most important organic dyes and pigments belong to the azo, carbonyl, phthalocyanine, arylcarbonium ion, sulfur, and methane and nitro chemical classes. Azo colorants constitute the most important chemical class by far. They count for about the 50–60% of the dyes used in traditional textile applications

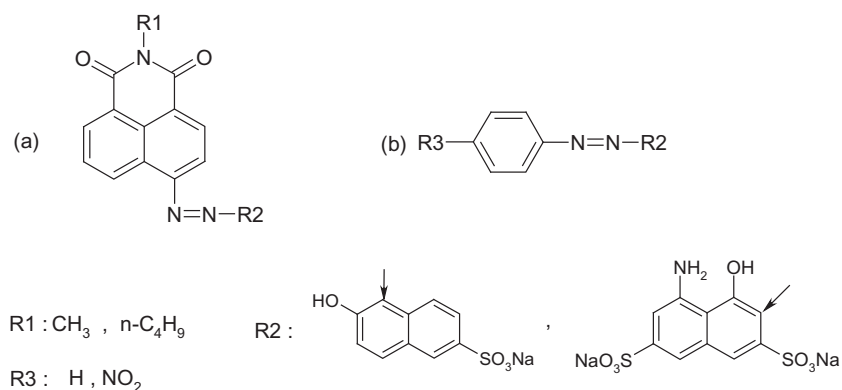
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**Figure 1** Acid dyes from 4-amino-N-alkyl-1,8-naphthalimide, aniline and paranitroaniline.

and they occupy a similarly prominent position in the range of commercial organic pigments. They are capable of providing a wide range of high intensity colors and brightness, although they give yellows, oranges and reds more readily. They are also capable of providing reasonable to very good technical properties, although in this respect they are often inferior to other chemical types (Christie et al., 1993). Azo dyes based on naphthalimides have violet to blue hue; this phenomenon is composed because of the high maximum absorption of their dyes. The strength of electron withdrawing inductive effect of naphthalimide systems is more than benzene ring systems containing nitro groups (Griffits, 1976).

Pioneering works on naphthalimides and their derivatives were thoroughly investigated and their role as intermediates for the preparation of dyes was assessed. (Khosravi et al., 2005, 2006; Gharanjig et al., 2007; Parvizi et al., 2009; Peters and Mak, 1958). Other related compounds, such as heterocyclic derivatives of naphthalimides, phenylazophthalimides and 1,8-naphthalic anhydrides have also been investigated (Peters and Bide (1986)). These compounds have been considered for the preparation of dyes which are used on textile fabrics, polymeric materials and dyes capable of copolymerization (Kraska and Blus, 1984). Investigations on naphthalimide represent that they could be applied on both synthetic and natural fibers as well as on other polymeric materials. Derivatives of N-methyl-1, 8-naphthalimide-3-sulfonic acids have been synthesized to produce a series of monoazo acid dyes. The solvatochromic effect and dyeing properties of these dyes were discussed (Wojciechowski, 1993a,b). Some of the acid dyes which are suitable for dyeing wool and polyamide fabrics were synthesized, using derivatives of arylsulphonanilides prepared from o-nitrotoluene or chloronitrotoluene. Derivatives of 1-phenyl-3-methyl pyrazolone and 2-naphthol were used as coupling compounds. Also, fastness and color properties of the prepared dyes were determined (Wojciechowski, 1993a,b). A series of monoazo acid dyes has been obtained using 4-amino-1, 8-naphthalimide and their 3-sulpho derivatives as diazo components. In this series derivatives of 1- and 2-naphthol were used as coupling components. These acid dyes have been applied on wool and polyamide fiber and their solubility and fastness properties have been evaluated (Wojciechowski, 1993a,b).

The objective of the present research is to obtain a series of the novel monoazo acid dyes for dyeing wool and polyamide fabrics for the first time. These dyes have been provided,

utilizing 4-amino-N-methyl (alternatively N-butyl)-1, 8-naphthalimide, aniline and p-nitroaniline as the diazo components and H-acid and Schaeffer's acid as coupling components (Fig. 1). The dyeing properties of prepared acid dyes were also compared with analogous monoazobenzene dyes based on naphthalene sulfonic acids containing the hydroxyl group as coupling components. The spectrophotometric properties of the synthesized dyes in various solvents were examined. The absorption maximum of these dyes and their intensities has also been obtained. Finally their fastness and build up properties were considered on wool and polyamide fabrics. There are several good reasons to pursue the preparation of naphthalimide dyes. The uses of diazo components such as 4-aminonaphthalimides have advantageous effects on the dyeing properties of the derived disperse azo dyestuffs. The presence of the naphthalimide system also leads to a deep and intense color.

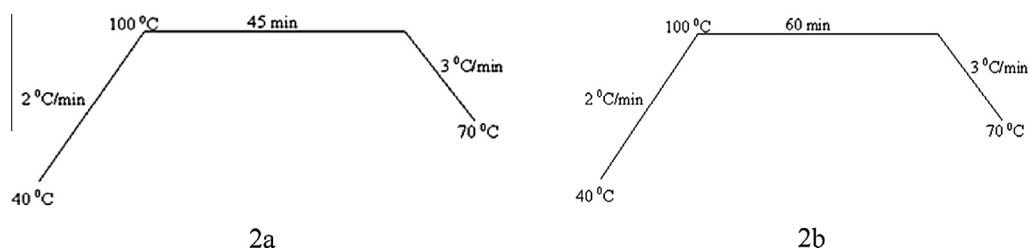
## 2. Experimental

### 2.1. Materials and apparatuses

All compounds used in this study were of analytical grade, unless stated otherwise. The novel monoazo acid dyes (Hosseinneshad et al., 2009) together with intermediate compounds were prepared in similar ways as described in the literature (Khosravi et al., 2005). Apparatuses utilized in this research are as follows: UV-visible spectrophotometry was carried out on a Cecil 9200 double beam transmission spectrophotometer. Molar extinction coefficients, absorption maximum and solvatochromic effects were also determined. The reflectance spectrophotometer data of dyed fabrics were obtained by a Grating Macbeth 7000A spectrophotometer. Light fastness was evaluated on a Xenotest 150s Hanau according to ISO 105-B02:1994(E).

### 2.2. Typical investigation of their electronic spectra

Electronic spectra of the synthesized dyes were run in various solvents such as  $\text{H}_2\text{O}$  in  $\text{pH} = 7$  and  $\text{pH} 3-4$ , mixture of N,N-dimethylformamide (DMF): $\text{H}_2\text{O}$  with solvent ratio of 50:50, and benzene. In all solvents the wavelength of maximum absorption was measured. The solvatochromic and halochromic effects of the dyes were also investigated.



**Figure 2** Diagram of dyeing wool fabrics (2a) and polyamide fabrics (2b) with synthesized dyes.

### 2.3. Dyeing procedure of wool and polyamide fabrics

#### 2.3.1. Dyeing wool fabrics

Wool fabrics (1 g) were pretreated with 5 g/l of nonionic detergent (Lotensol Hansa), at 60 °C for 20 min prior to being used for the dyeing. The samples were dyed in a dyeing apparatus using acetic acid (pH 4-5) and L: R = 40:1. The build up properties of the synthesized dyes on wool fabrics were obtained by dyeing with dye solutions 0.1%, 0.3%, 0.5%, 0.7%, 1%, 1.5%, 2% and 4% owf (*Ohio Works First*). Dyeing was carried out by raising the dye bath temperature from 30 to 100 °C at the rate of 2 °C/min, holding at this temperature for 45 min and cooling down at 70 °C at the rate of 3 °C/min (Fig. 2a). The light, wash, milling and perspiration fastnesses of each dyed fibers were determined according to ISO 105-B02:1994(E), ISO 105-C02:1989(E), ISO 105-E12:1989(E) and ISO 105-E02:1994(E), respectively.

#### 2.3.2. Dyeing polyamide fabrics

Polyamide fabrics (1 g) were pretreated with 5 g/l of nonionic detergent (Lotensol Hansa), at 60 °C for 20 min prior to being used for the dyeing. The samples were dyed in a dyeing

apparatus using acetic acid (pH 4-5) and L: R = 40:1. The build up properties of the synthesized dyes on polyamide fabrics were obtained by dyeing with dye solutions 0.1%, 0.3%, 0.5%, 0.7%, 1%, 1.5%, 2% and 4% owf. Dyeing was carried out by raising the dye bath temperature from 30 to 100 °C at the rate of 2 °C/min, holding at this temperature for 60 min and cooling down at 70 °C at the rate of 3 °C/min (Fig. 2b). The light, wash and perspiration fastnesses of each dyed fibers were determined according to ISO 105-B02:1994(E), ISO 105-C02:1989(E), ISO 105-E12:1989(E) and ISO 105-E02:1994(E), respectively.

### 3. Results and discussion

A series of monoazo acid dyes with general formula of Fig. 1, containing the sulfonic acid group in coupling components was synthesized. The acid dyes had yellow, red, orange and violet hues (Table 1). Diazotization of naphthalimides was carried out with the aid of nitrosylsulphuric acid, because naphthalimide derivatives had weak amines due to having two carbonyl groups. Aniline and p-nitroaniline considering that they have strong amines, were diazotized using hydrochloric acid

**Table 1** Acid dyes derived from various diazo and coupling components.

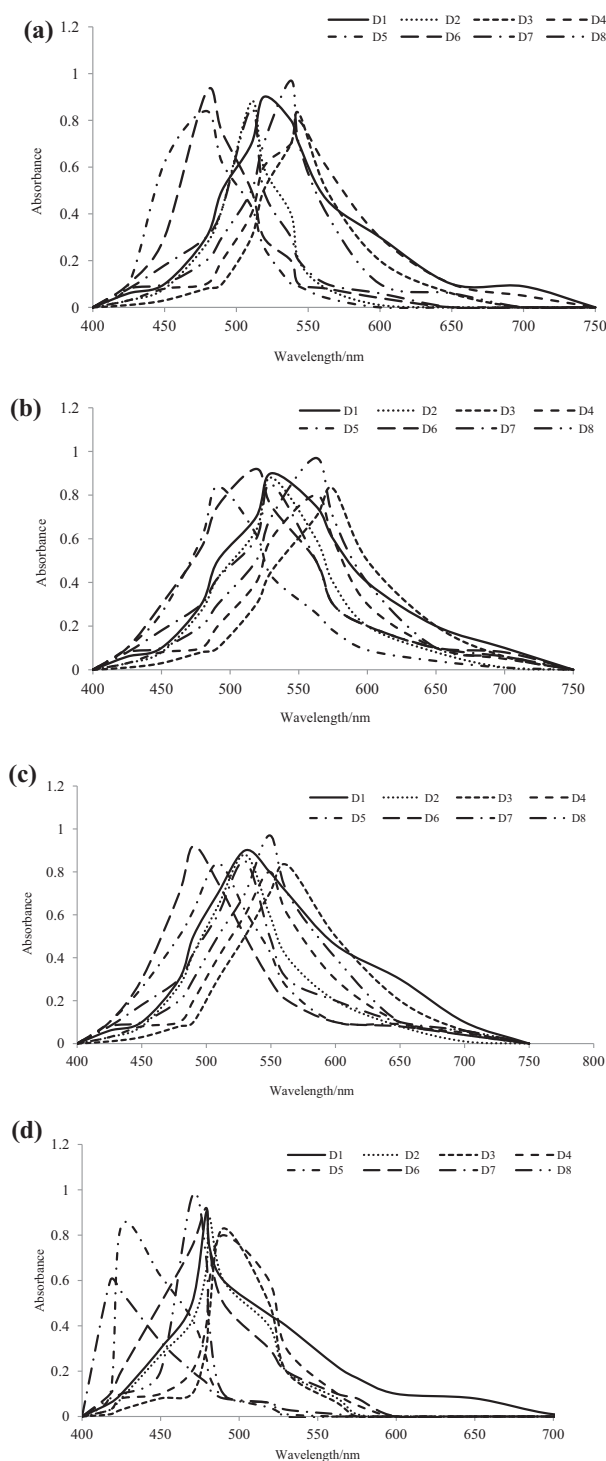
Dye	Diazo component	Coupling component	Yield (%)	Observed color
1	4-Amino-N-methyl naphthalimide	Schaeffer's acid	87	Red
2	4-Amino-N-butyl naphthalimide	Schaeffer's acid	85	Red
3	4-Amino-N-methyl naphthalimide	H-acid	87.5	Violet
4	4-Amino-N-butyl naphthalimide	H-acid	89	Violet
5	Aniline	Schaeffer's acid	91	Yellow
6	Paranitroaniline	Schaeffer's acid	90	Orange
7	Aniline	H-acid	92.5	Red
8	Paranitroaniline	H-acid	91.4	Violet

**Table 2** UV-visible electronic spectral data of synthesized acid dye (1-8).

Dye	$\lambda_1$ in H <sub>2</sub> O (nm) pH = 7	$\lambda_2$ in H <sub>2</sub> O (nm) pH = 3-4	$\Delta\lambda = \lambda_2 - \lambda_1$	DMF:H <sub>2</sub> O = 50:50		$\lambda_4$ in benzene (nm)
				$\lambda_3$ (nm)	$\epsilon_{max}$ (l/mol cm)	
1	519.0	530.0	11.0	530.0	38995	479.0
2	510.5	529.0	18.5	533.0	41925	481.5
3	543.5	575.5	32	563.5	39562	490.0
4	549.5	562.5	13	554.5	49080	490.5
5	479.0	491.6	12.6	509.5	38233	425.5
6	478.0	519.5	41.5	482.5	33196	475.5
7	507.0	528.6	21.6	529.0	36983	418.5
8	538.5	560.5	22	549.5	37637	470.5

(Bukharina et al., 2002; Gharanjig et al., 2008; Singh et al., 2003). The correlation between the color and constitution is a highly interesting aspect of the development of the dyes and commercialization. In this respect, the wavelength of maximum absorption, the molar extinction coefficient and absorption profile are important for dyes (Griffits, 1984). The role of the sulfonic group in acid dyes is mentioned in the literature data. It has been observed that this group can affect the color of the dye, position of the maximum absorption ( $\lambda_{\max}$ ) and brightness of the shades. The absorption spectra of the acid dyes were measured in benzene, H<sub>2</sub>O at pH 7, H<sub>2</sub>O at pH 3–4 and DMF:H<sub>2</sub>O with the solvent ratio of 50:50, respectively which are summarized in Table 2.

The spectrophotometric data of the synthesized dyes in various solvents indicated that, the maximum absorption of the acid dyes was 482.5 to 563.5 nm in DMF:H<sub>2</sub>O = 50:50 (Fig. 3d) and molar extinction coefficient was about 33196–49080 l/mol cm. In the present study as illustrated in Fig. 1, the naphthol residue acts as the donor group and the two carbonyl groups of naphthalimide residue act as acceptor groups, and following this line of reasoning, the dyes 1–4 give a bathochromic shift effect as well. All synthesized dyes based on naphthalimide are more bathochromic, compared to synthesized dyes based on azobenzene (Table 2) and the reason for that is because the two carbonyl groups of naphthalimide residue are more acceptor than the phenyl and nitrophenyl groups. The presence of the electron donor group in coupler and the electron acceptor group in diazo component causes strong bathochromic effect in the prepared dyes (Gharanjig et al., 2008; Singh et al., 2003). The UV-vis analysis indicated bathochromic shift in all compounds. The effect of the –NO<sub>2</sub> group on the diazo component in the synthesized acid dyes (dyes 6 and 8) is more bathochromic compared to unsubstituted diazo components (dyes 5 and 7) as mentioned in Table 2. The absorption maximum of compound 3 reflected a bathochromic shift (red shift) by over 33 nm, compared to compound 1. This bathochromic shift vividly indicates that charge transfer from the donor group is delocalized across the acceptor systems, thereby increasing the conjugation length between donor and acceptor (Vijayalakshmi and Kalyanaraman, 2013; Bartkowiak and Lipkowski, 2005). The absorption maximum for compound 3 in DMF:H<sub>2</sub>O = 50:50 being recorded at 563 nm and that for compound 1 at 530 nm (Table 2) clearly exemplify the charge transfer of the acceptor and that it is more effective in compound 3 due to a higher shift. A contribution toward the bathochromic shift is generally expected when the molecules in the CT excited state have higher polarization than the ground state (Vijayalakshmi and Kalyanaraman, 2013). For such molecules, high polar solvents produce a better stabilization in the excited state than the ground state. The excitation energy is significantly decreased by the bathochromic shift. This leads to the increase of the first order hyperpolarizability owing to the bathochromic shift of the CT absorption band (Vijayalakshmi and Kalyanaraman, 2013). The measurement of maximum absorption of the acid dyes in benzene, H<sub>2</sub>O (pH 7) and DMF:H<sub>2</sub>O = 50:50 (Table 2 and Fig. 3a-d) showed that all dyes have positive solvatochromic effect, which is caused when the polarity of the solvent was changed from low to high which is related to dielectric constants of the solvents. This phenomenon is generally known as solvatochromism, however, it is not limited to solvated polarity only, it could also be observed in solid and on various



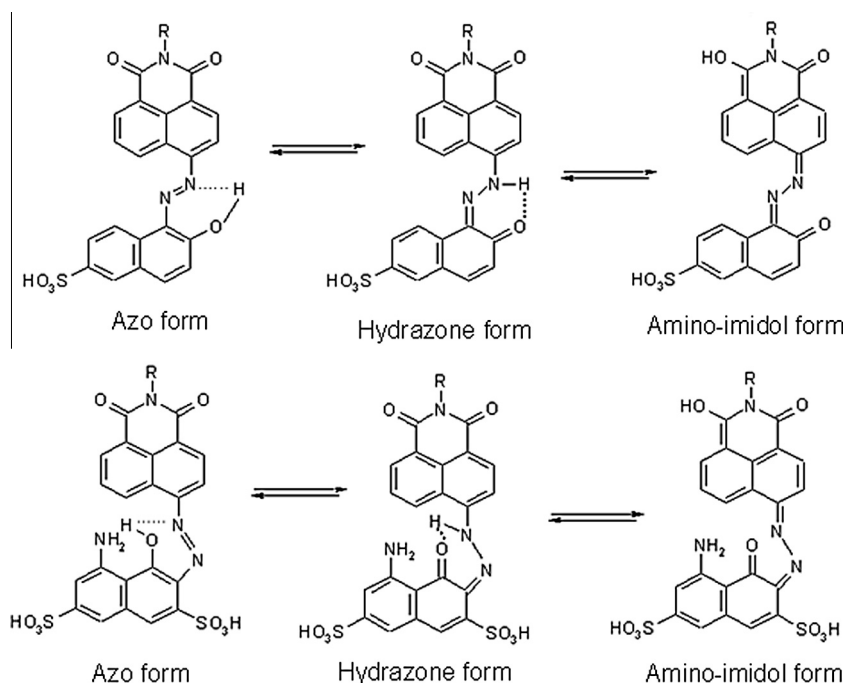
**Figure 3** UV-Vis absorption spectra of D1–D8 dyes in (a) H<sub>2</sub>O:pH = 7; (b) H<sub>2</sub>O:pH = 3–4; (c) H<sub>2</sub>O/DMF; (d) benzene.

surface/interfaces. Solvatochromism is caused by differential solvation of the ground and first state of the light absorbing molecules. Based on its ability to detect the bulk or local polarity, solvatochromism is commonly used in many fields of chemical and biological research (Racz et al. (2013); Bouvrain et al., 2010). In general, it can be concluded that the highest maximum absorption belongs to the mixture of DMF:H<sub>2</sub>O solvent and minimum absorption belongs to the non polar

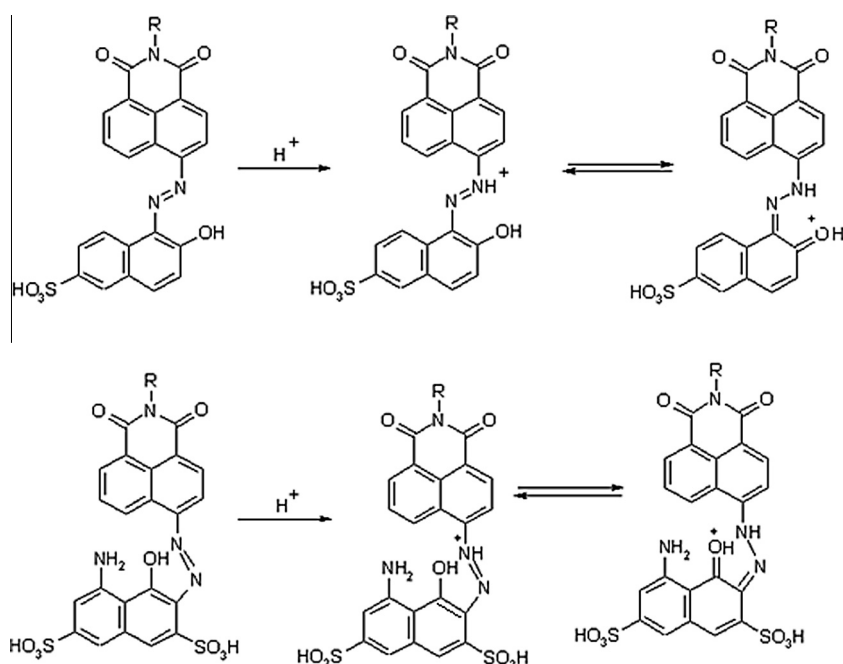
solvent of benzene (Table 2; Fig. 3c and d). However, solvatochromism still remains largely unknown due to the complex coupling of these many different interactions (Racz et al. (2013); Bag et al., 2010).

The color of the acid dyes is attributed to azo-hydrazone tautomerism, since the two tautomeric forms generally will have different spectroscopic properties. It is found experimentally that the hydrazone forms always absorb at longer

wavelengths than their azo tautomers. These two forms are illustrated in Fig. 4. In aqueous solutions of pH 7, the naphthol derivatives usually occur in the azo form. The absorption of the hydrazone form has a significant bathochromic shift in acid medium compared to the azo form (Griffits, 1976). As mentioned above, the dyes can occur in two different hydrazone forms, viz, either with the azo system (azo-hydrazone tautomerism) or with the imido system (amido-iminol



**Figure 4** Azo-Hydrazone forms in acidic pH environment.



**Figure 5** Halochromic effect in synthesized acid dyes.

tautomerism), in Fig. 4, according to Wojciechowski results (Wojciechowski, 1993a,b). The measurement of maximum absorption of the acid dyes in H<sub>2</sub>O at pH 7 and pH 3-4 (Fig. 3a and b) showed that all dyes have halochromic effects, when the pH of solvent was changed from neutral to acidic condition the maximum absorption ( $\lambda_{max}$ ) of the acid dyes was changed as represented in Table 2. The halochromic effect is illustrated in Fig. 5. These observations are matching with the Griffiths (Griffiths, 1976) and Wojciechowski results (Wojciechowski, 1993a,b). The intra molecular hydrogen bond leads to the rise of a greater charged, excited state due to the electrostatic interaction between the adjacent groups. This interaction in turn produces a bathochromic shift in the UV-vis Spectrum (Vijayalakshmi and Kalyanaraman, 2013; Delower et al., 2010).

To investigate the dyeing properties of the synthesized acid dyes on wool fabrics, solutions of dyes (0.2 g/l) in H<sub>2</sub>O were prepared. These solutions with 8 different concentrations were used for dyeing wool fabrics. Dyes 1-4 gave red to violet hue, with excellent build up, while dyes 5-8 with phenylazo structures gave yellow to violet hue on wool fabrics. In general,

the bathochromic shift in dyes 1-4 is related to the presence of naphthalimide ring in diazo components. Fig. 6 shows the build up properties of acid dyes based on naphthalimides and azobenzene series on wool fabrics. The color strength of dyed wool and polyamide fabrics reached saturation at the dye concentration of about 1% owf.

To investigate the dyeing properties of the synthesized acid dyes on polyamide fabrics, solutions of dyes (0.2 g/l) in H<sub>2</sub>O were prepared (Hosseinezhad et al., 2009). These solutions with 8 different concentrations were used for dyeing polyamide fabrics. Dyes 1-4 gave red to violet hue with excellent build up, while dyes 5-8 with phenylazo structures gave orange to violet hue on polyamide fabrics. In general, the bathochromic shift in dyes 1-4 is related to the presence of the naphthalimide ring in diazo components. Fig. 7 shows the build up properties of acid dyes based on naphthalimides and azobenzene series on polyamide fabrics.

The spectroscopic data of the wool and polyamide fabric dyeing treatments indicated that dye 4 has a strong bathochromic effect in comparison to dyes 1-3, but shows the lowest build up as well as dye 8. The presence of H-acid as a coupling

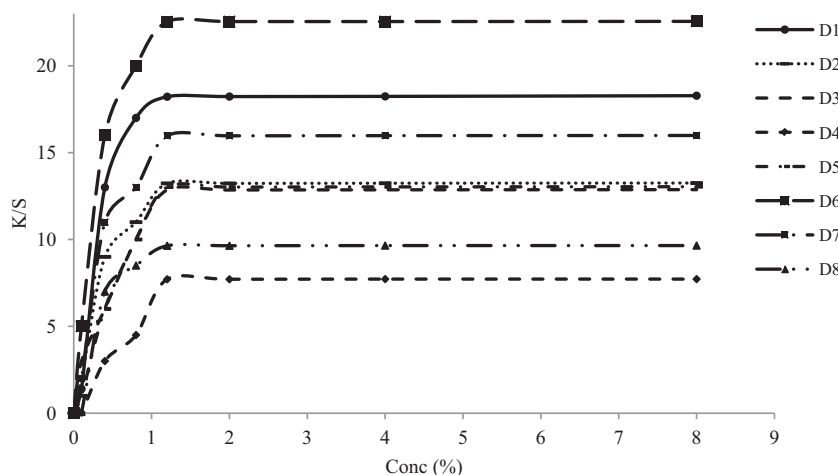


Figure 6 Build up property of synthesized dyes on wool fabrics.

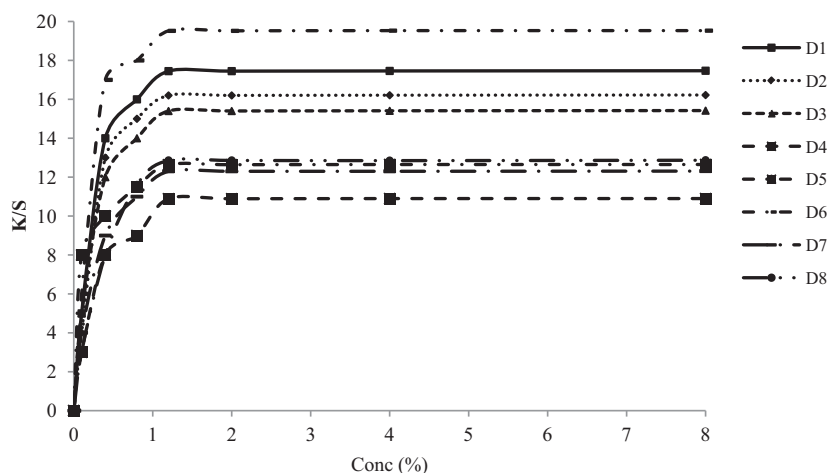


Figure 7 Build up property of synthesized dyes on polyamide fabrics.

component due to having two electron donor groups (Dyes 3, 4, 7 and 8) relative to Schaeffer's acid which has only one hydroxyl group as an electron donor (Dyes 1, 2, 5 and 6) causes bathochromic effect but the presence of two sulfonic acid groups causes hydrophilic effect and finally causes low build up.

The spectrophotometric properties of wool and polyamide dyeing treatments in terms of the CIE Lab system are shown in Tables 3 and 4, respectively. Depending on the presence of substituents in coupling component, the wool and polyamide fabric dyeing processes varied in color from red to violet. Naphthalimide derivatives which possess positive  $a^*$  and negative  $b^*$ , have blue-red hues. Whereas the spectrophotometric data of dyeing represented that all dyes based on dyed fabrics with azobenzene derivatives have yellow-red hues due to positive  $a^*$  and  $b^*$ . Of course, in these series, dyed fabrics with dyes 7 and 8 have bluish red hues, because these dyes contain H-acid in their chemical structures. According to the results of the build-up curves of wool and polyamide fabrics (Figs. 6 and 7), the synthesized acid dyes based on naphthalimide and azobenzene were suitable for dyeing of natural and synthetic polymers. The chromaticity values of the synthesized acid dyes on wool and polyamide fabrics showed that acid dyes have the same hue.

The wash fastness of dyed wool and polyamide fabrics was tested according to ISO 105-C02:1989(E). The data demonstrated that washing fastness of all samples was very good

**Table 3** The spectrophotometric data of the synthesized dyes on wool fabrics (1% owf).

Dye	L*	a*	b*	c*	h°	K/S
1	35.79	41.06	-6.76	43.77	335.98	18.22
2	30.47	50.30	-6.37	36.19	222.23	13.23
3	37.8	10.05	-17.68	20.14	315.82	12.85
4	36.34	10.55	-17.92	16.98	184.57	7.71
5	43.17	8.03	26.75	26.72	311.58	13.02
6	46.32	44.06	27.42	56.19	250.72	22.54
7	31.21	43.76	-6.72	44.74	293.72	15.97
8	30.18	30.18	-17.99	19.23	252.61	9.64

**Table 4** The spectrophotometric data of the synthesized dyes on polyamide fabrics (1% owf).

Dye	L*	a*	b*	c*	h°	K/S
1	32.79	40.78	-7.46	44.40	321.65	17.45
2	29.47	48.95	-6.30	35.12	256.96	16.20
3	35.5	9.81	-16.51	16.19	302.65	15.40
4	33.21	10.06	-16.39	17.40	209.31	10.89
5	40.17	10.26	20.41	21.50	261.52	12.64
6	41.12	32.12	21.36	44.63	250.63	19.52
7	35.68	33.26	-9.84	40.21	301.56	12.30
8	33.67	24.21	-11.45	26.74	302.56	12.86

**Table 5** Wash, light, milling and perspiration fastness properties of the synthesized acid dyes on wool fabrics (1% owf).

Dye	W.F			L.F	M.F			P.F					
	Ch	C	W		Ch	C	W	Alkaline			Acid		
								Ch	C	W	Ch	C	W
1	4-5	5	5	4-5	4-5	5	5	4-5	4-5	5	4-5	4-5	5
2	4-5	5	5	4	4-5	5	5	4-5	4-5	5	4-5	4-5	5
3	4-5	4-5	5	4-5	5	4-5	5	5	4-5	5	5	4-5	5
4	4-5	4-5	5	4-5	5	5	5	5	5	5	5	5	5
5	5	5	5	5	5	5	5	4-5	5	5	4-5	5	5
6	4-5	5	5	4-5	4-5	5	5	5	4-5	5	5	4-5	5
7	5	4-5	5	4-5	5	4-5	5	5	5	5	5	5	5
8	5	5	5	4-5	5	5	5	4-5	5	5	4-5	5	5

W.F, wash fastness; L.F, light fastness; M.F, milling fastness; P.F, perspiration fastness; Ch, change; C, staining on cotton; W, staining on wool.

**Table 6** Wash, light, milling and perspiration fastness properties of the synthesized acid dyes on polyamide fabrics (1% owf).

Dye	W.F			L.F	P.F					
	Ch	C	W		Alkaline			Acid		
					Ch	C	W	Ch	C	W
1	5	5	5	4	4-5	4-5	5	4-5	4-5	5
2	5	5	5	3	4-5	4-5	5	4-5	4-5	5
3	5	4-5	5	3	5	4-5	5	5	4-5	5
4	5	4-5	5	3-4	5	5	5	5	5	5
5	5	4-5	5	4	4-5	5	5	4-5	5	5
6	5	5	5	4	5	4-5	5	5	4-5	5
7	5	4-5	5	3-4	5	5	5	5	5	5
8	5	5	5	3	4-5	5	5	4-5	5	5

W.F, wash fastness; L.F, light fastness; P.F, perspiration fastness; Ch, change; C, staining on cotton; W, staining on wool.

(4-5) (Tables 5 and 6). For testing light fastness, the samples of standard fabric were mounted on a frame partly covered on opaque sheet, leaving the other half exposed under Xenone lamp ISO105-B02:1994(E). The dyes showed medium light fastness on wool and polyamide fabrics. The perspiration fastness of dyed fabrics was tested according to ISO 105-E02:1994(E). The dyed fabrics showed good perspiration fastness (Tables 5 and 6). The milling fastness of dyed wool fabrics was examined according to ISO 105-E12:1989(E). The dyed fabrics showed very good milling fastness (Tables 5 and 6).

#### 4. Conclusion

Two series of monoazo acid dyes based on 4-aminonaphthalimide and aminobenzene derivatives were prepared. Their spectrophotometric and their dyeing properties were investigated and compared with each other. The results pointed toward that the synthesized dyes based on aminonaphthalimide derivatives are more bathochromic than aminobenzene derivatives. The synthesized dyes showed that they have different dyeing ability on wool fabrics and they reached saturation at the concentration of 1% owf. From this point of view the acid dyes containing naphthalimide derivatives presented nearly similar dyeing behavior to aminobenzene derivatives.

The spectrophotometric properties of all of the synthesized monoazo acid dyes in various solvents were examined. It was found that these acid dyes have positive solvatochromic effect.

The dyeing fastnesses of all the prepared acid dyes on wool substrate were determined. The results demonstrated that wash fastness, perspiration fastness and milling fastness of all dyes were good while the light fastness of all dyes was medium. The chromaticity diagram of the synthesized dyes on wool fabrics was analyzed. The results indicated that the synthesized acid dyes based on naphthalimides have red to violet and aminobenzene derivatives have yellow to violet color gamut.

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