Synthesis and application of novel monoazo disperse dyes based on N-ester-1,8-naphthalimide on polyester

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ABSTRACT

A series of novel monoazo disperse dyes based on N-ester-1,8-naphthalimide were synthesized. 4-Nitro-1,8-naphthalic anhydride was reacted with ethyl glycinate and reduced to 4-Amino-N-ethyl glycinate-1,8-naphthalimide, then diazotized and coupled with a number of couplers. The absorption characteristics of the synthesized dyes were investigated and extinction coefficient, absorption maxima and solvatochromism effect were evaluated. The synthesized dyes applied on polyester fibres. Data showed that they were suitable for dyeing of polyester fibres, giving yellow to bluish red color. The dyes containing ester group revealed an alkali-clearable properties.

KEYWORDS

Dyeing, Alkali- Clearable, Polyester, Disperse dyes, Ester compounds

1. INTRODUCTION

Polyester fibres and its blendes with cotton extensively are used in textile industry. Due to being hydrophobic, the polyester fibers are dyed mostly with disperse dyes [1]. The disperse dyes possessing various chemical structure but more than 50% of these dyes having azo chromogen [2]. These easily synthesized dyes and having extended shades from greenish yellow to magneta is the reason for application of these dyes for coloring of polyester. These dyes are synthesized from intermediate materials which are cheap and easily available.

There are a few research reports where the azonaphthalimides are used as disperse dyes [3]-[5]. These dyes are able to dye hydrophobic fibers as polyamide, polyester and acetate fibers and give acceptable color fastness. For synthesizing of azonaphthalimides compound 4-amino-N-substituted-1,8-naphthalimides were used as diazo component and suitable aromatic amines as coupler were utilized [4]-[6]. 4-amino-N-alkyl-1,8-naphthalimides were diazotized and coupled with N,N-diethyl-m-toluidine and they produced a group of disperse dyes which were able to dye polyester and produce bluish-red shades with reasonable fastness on polyester fibres[6].

2-Naphthol, p-aminoacetophenone, N-ethyl-N-cyanoethyl-aniline and 3- methoxy-N,N-diethylaniline have been used as coupler for preparation of azo naphthalimide dyes.

These compound coupled with diazotized 4-amino –1,8-naphthalimide and produced new disperse dyes, which showed strong potential for dyeing of polyester fibers [6,7].

The spectrophotometeric properties of azo-1,8-naphthalimides have been studied and high color intensity and brightness were reported. In comparison to the similar azobenzene compound, naphthalimide compounds proved to have strong bathochromic effect [8].

To our knowledge, synthesis and dyeing properties of azo disperse dyes based on N-ester-1,8-naphthalimides were not investigated. In this study, a group of novel azo disperses dyes with different chemical structure show in Fig. 1 were synthesized and their properties investigated.

Spectrophotometeric data of each synthesized dyes in different solvent were evaluated and solvatochromism properties were emphasized. Also, the application of synthesized dyes on polyester fibres were carried on and fastness properties were studied.

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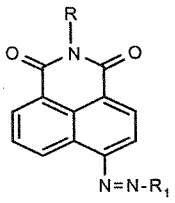
2. EXPERIMENTAL

Materials and apparatus

All materials for synthesis of dyes were obtained from Aldrich and Merck Company. These materials were used without further purification. ^{1}HNMR spectra were recorded on a Brucker AVANCE-300MHZ. Electronic spectra (in various solvents) were measured using a Cecil 9200 Spectrophotometer. FTIR Spectra (KBr) were recorded on a Nicholet 470/670/870 spectrophotometer. The melting point of dyes and intermediates were measured by DSC 2010 TA instrument. The reflectance spectrophotometer data of dyed fabrics were obtained by Gratig Macbeth 7000A spectrophotometer. Elemental analysis was carried out by CHNO Analyser Foss Herps.



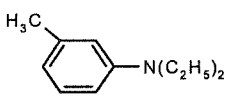
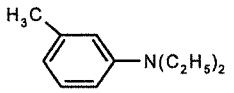
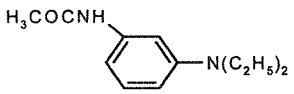
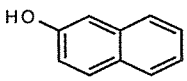
	R	R1
1	CH_3	
2	$CH_2COOC_2H_5$	
3	$CH_2COOC_2H_5$	
4	$CH_2COOC_2H_5$	

Figure. 1. Chemical structure of the synthesized dyesnthesized dyes.

Syntheses of intermediates

Synthesis of 5-nitroacenaphthene and 4-nitro-1,8-naphthalic anhydride were carried out according to Okazaki and Taniguchi methods [8].

Synthesis of 4- nitro – N- ethylglycinate -1,8- naphthalimide

A mixture of 4-Nitro-1,8–naphthalic anhydride (24.3 g), ethanol (500 ml) and ethyl glycinate (13.4 g) were added to round bottom flask (1000 ml) and was refluxed for 10 hrs. This mixture was added to water and filtered. The crude product was purified by recrystalization with ethanol.

Synthesis of 4- amino-N-ethylglycinate-1,8- naphthalimide

A mixture of 12 g 4-nitro-N-ethylglycinate-1,8-naphthalimide, 40.62 g stannous chloride, 180 ml ethanol and 45 ml hydrochloric acid were refluxed for 1 hrs. The mixture was poured into water and filtered. The crude product was purified by column chromatography (eluent: toluene/ acetone, 2/1).

Syntheses of dyes

Diazotization of 4-amino-N-ethylglycinate-1,8-naphthal-imide

4-Amino-N-ethylglycinate-1,8-naphthalimide (15.8 g) was added over 60 min at 10°C to nitrosyl sulphuric acid prepared from sodium nitrite (3.7 g) and concentrated sulphuric acid (23.2 ml), and diluted with glacial acetic acid (10 ml). Diazotisation was continued for a further 3 hrs at below 5°C. The clear diazonium salt obtained. For syntheses of dyes this solution was added slowly to various couplers. Diazotisation of 4-amino-N-methyl-1,8- naphthalimide was carried out [6,9].

Coupling

53 mmol of each coupling components dissolved in 101 ml glacial acetic acid. Then diazonium salt was added to this solution. The mixture was stirred for 1 hrs at below 10 °C. The pH was maintained between 4 and 5 by adding sodium acetate solution. Stirring was continued for 4 hrs at 10 °C. The dyes were filtered and washed with hot water. 2-Naphthol as coupler was dissolved in ethanol and diazonium salt was added to it. The crude dyes were purified by TLC method using chloroform as effluent.

Preparation of dye dispersions

Lyoprint EV as nonionic dispersing agent was used to make the dispersions. For this purpose, 1 g pure dye, 2 g dispersing agent and 30 ml water milled (ball – mill) for 20 hrs. The mixture was diluted with water to 100 ml and then filtered (5 μm).

Dyeing

Polyester fabrics (1 g) were pretreated with a nonionic detergent, (5 g/1) at 80 °C for 20 min prior to being used for the dyeing. The samples were dyed in a rotadyer apparatus (Nasaj sanat yazd) using acetic acid (pH = 4-5) and L.R:50:1. The build up properties of the synthesized dyes on polyester fabrics obtained by dyeing with dye dispersions 0.1, 0.3, 0.5, 0.7, 1, 1.5,2 and 4% (o.w.f). Dyeing was carried out by raising the dyebath temperature from 30 °C to 90 °C at 2 °C/min, followed 90 °C to 130 °C at 1°C /min, holding at this temperature for 60 min and cooling to 70 °C at 3 °C/min.

The dyed samples were rinsed with cold water and reduction cleared using sodium hydrosulfite (2 g/1), sodium hydroxide (1 g/1) and detergent (1g/1), at a liquor to goods ratio of 50:1 (Fig. 2).

Alkali-clearing of the dyed samples were carried out with

solution containing sodium hydroxide (2.5 g/l) and detergent (1 g/l) at 80 °C for 30 min.

$$50^{\circ}C \xrightarrow[\text{Reduction - clearing}]{20\ \text{min}}$$

$$80^{\circ}C \xrightarrow[\text{Alkali - clearing}]{30\ \text{min}}$$

Figure 2. Profile of reduction clearing and alkali-clearing.

The measurement of color fastness of dyed fabrics

The wash fastness of dyed fabrics was tested according to ISO 105C06/C2S. For this purpose, the dyed samples with undyed sample 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 testing light fastness, the samples and the wool standards were mounted on a frame partly covered on opaque sheet, leaving the other half exposed in xenone lamp (ISO 105/B02). The heat fastness property of the samples were done according to ISO 105/p01. The rubbing fastness of the dyed substrate was carried out according to standard method of ISOX12:1993 (E).

3. Results and Discussion

The most convenient starting material for the preparation of naphthalimide dyes is acenaphthene. This material is readily and cheaply available as a by-product of coal processes industry. In this study, acenaphttene was used for synthesis of azo disperse dyes based on N-ester-1,8-naphthalimide.

5-Nitroacenaphthene is the main product when acenaphthene reacts with nitric acid in acetic acid at room temperature. Yellow needles of 5-nitro-acenaphthene were obtained with m.p=101-102°C, when recrystallized from ethanol.

Oxidation of this product to 4-nitro-1,8-naphthalic anhydride was carried out using sodium dichromate as oxidizing agent and hot glacial acetic acid as the solvent. The crude product was recrystallized from nitric acid and m.p of the pure product was 229-230°C. The yield of 4-nitro-1,8-naphthalic anhydride was 55%.

The reaction of naphthalic anhydride or substituted naphthalic anhydride with ammonia or alkylamines gives naphthalimide derivatives. Therefore, for preparation of 4-nitro-N-ethylglycinate-1,8-naphthalimide, 4-nitro-1,8-naphthalic anhydride was reacted with ethyl glycinate in alcoholic media, and the end of reaction was examined by TLC on silica gel. This reaction was completed in 10 hrs which is relatively longer in comparison to the 4-nitro-1,8-naphthalic anhydride with methyl or ethylamines (about 7 hrs). These differences in the reaction time can be attributed to the higher activation of alkylamines to alkylglycinates in nucleophilic substitution reaction groups. In comparison with alkylglycinates, they are strongly electron donor groups. The yield of the pale yellow and needle crystals product was 92.28%.

Preparation of 4-amino-N-ethylglycinate-1,8-naphthalimide from 4-nitro-N-ethylglycinate-1,8-naphthalimide was carried out by reduction with stannous chloride and hydrochloric acid. This product with an amino group at the 4-position is highly fluorescent. The yield of reaction was 84.97%. The FTIR, ^{1}HNMR and Elemental Analysis data were showed in Table 1.

The diazotization of 4-amino-N-alkyl-1,8-naphtha-limides was carried out with nitrosyl sulphuric acid. Therefore, 4-amino-N-ethylglycinate-1,8- naphthal-imide was diazotized using sodium nitrite and sulphuric acid. The diazonium salts were coupled with 2-naphthol, N,N-diethyl-m-toluidine and 3-(N,N-diethylamino) acetanilide. The coupling reaction diazonium salt with 2-naphthal was carried out in alcoholic media, while other couplers were coupled in acidic media (pH= 4-5). All dyes were purified by preparative TLC and characterized by FTIR, ^{1}HNMR, DSC and Elemental Analysis (Table 2).

The correlation between the color and constitution is a highly interesting aspect of dyes development and commercialization. In this respect, the wavelength of maximum absorption, the molar extinction coefficient and absorption profile are important for dyes.

The spectrophotometric properties of the synthesized dyes measured in toluene, chloroform and DMF. Data showed that, the absorbance maximum of the dyes were 538.7 to 554 nm in chloroform. The corresponding molar extinction coefficients were about 21368 to 44080 l/mol cm. Dye 3 showed strong bathochromic effect, where $-NHCOCH_3$ group introduced in coupling component. The presence of electron donor group ($-NHCOCH_3$) in coupler causes strong bathochromic effect in azobenzen series [12]. Also, the presence of $-NHCOCH_3$ group at ortho-possition of azo linkage on coupling component caused bright hue. This brightness effect has been attributed to intramolecular hydrogen bonding between azo and acetamido groups [13]. This phenomenon was observed with dye 4 where, 2-naphthal was used as coupler. In this dye, intramolecular hydrogen bonding formed between azo and hydroxyl groups. The measurement of maximum absorption of dyes in toluene and DMF showed that Dyes 1 to 3 have positive solvatochromism, when polarity of solvent was changed from low to high. The lowest $\Delta\lambda_{max}$ pertain to dye 3 ($\Delta\lambda_{max}$ = 18.4 nm), where $-NHCOCH_3$ group introduced to N,N-diethylaniline. In other cases $\Delta\lambda_{max}$ were 25 nm. These results are confirmed by other researchers with heterocyclic compounds [13]. Dye 4 showed negative solvatochromism effect.

The maximum absorption of dye 1 and 2 showed that the

nature of imide residue is not very significant in the context of color change. Comparing the maximum absorption of these dyes with when imide residue R= C_2H_5, R= C_3H_7, R= C_4H_9 were not revealed considerable significant changes [4,6].

For investigation of dyeing properties of the synthesis dyes on polyester fabrics a dispersion of dyes in a dispersing agent were prepared. These dispersions were used for dyeing of polyester fibres by HT method. Figure 3 shows the build up property of dyes on polyester fabrics. The color strength of dyed fabric reached saturation at the dye concentration ~1% owf. The dyed fabrics with all dyes (except 3) showed good build up and levelness. Data showed that also dye 3 has a strong bathochromic effect in comparison to dye 1-2, but with low exhaustion rate on polyester. In order to determine the rate of dyeing of synthesized colorants on polyester fibres, dyeing with 1.2 % owf of the dye was carried out at different times of dyeing. The obtained results indicated that dyes 1 and 3 have highest and lowest rate of dyeing respectively. Further elaboration on curves in Fig. 4 revealed that the dyes 2 and 4 representing similar dyeing rate. The measurement of color of dyed fabrics by reflectance spectrophotometer showed that the strongly electron donor substitution in coupling component have significant effect towards change in color of the samples. The presence of $-NHCOCH_3$ in dye 3 was caused displacement of color to violet, whilst the dyed fabric with dyes 1 and 2 are red to relatively bluish red. In comparing to the other dyes, the color of dyed fabric with dye 4 was relatively pure red.

The measurement of wash fastness of dyed polyester fabrics showed the color fastness of all the samples were very good (4-5), when they were reduction cleared. But alkali-cleared samples showed wash fastness from poor to very good. Dyes 2 and 4 present very good wash fastness, which can be attributed to the presence of the ester groups. These ester groups were converted and hydrolyzed to a water soluble carboxylate group by alkali-hydrolysis, which easily washed-off. The results indicated that the presence of ester group in dyes 2 and 4 did not lower the washing fastness of samples. This finding has been reported by other heterocyclic compound [13]. Hydrolysis of dyes containing ester group in alkaline media provides replacement of reduction clearing process, in other words, by these replacement drastically BOD of wastewater and other environmental pollution is prevented. The advantage of replacement of reduction clearing-method by alkali-clearing process will be the prevention of producing toxical and carcinogenic amine compounds [15]-[17]. It seems in absence of ester group the alkali-clearable property of dyes is negligible. The synthesized dyes showed light fastness of weak to medium and heat fastness of weak to high. The rubbing fastness of dyed substrate of showed that the highest rubbing fastness belongs to dye 4 (5) and the lowest to dye 3. Alkali-clearing and reduction clearing of the dyed substrate resulted in the same rubbing fastness for dye 4. In the case of dye 1, there is profound difference in rubbing fastness of dyed substrate. This effect could be attributed to ester groups presents on dye 1.

Table 1: FTIR, ^{1}HNMR and Elemental Analysis data of intermediates.

Intermediates	FTIR, ^{1}HNMR and Elemental Analysis data
4-nitro-N-ethylglycinate-1,8-naphthalimide	^{1}H NMR ($CDCl_3$): δ 1.30266-1.35023(3H, CH_3) 4.24031-4.31165(2H, CH_2CH_3) 4.95409(2H, NCH_2) 7.98208-8.03568(1H, 6-H) 8.40209-8.42872(1H, 3-H) 8.69880-8.76669(2H, 2-H, 5-H) 8.83807-8.87027(1H, 7-H); FTIR (KBr): ν 3118.31Cm^{-1}(C-H str. Ar.), 2987.36cm^{-1}(C-H str. Aliphatic), 1742.85Cm^{-1}(C=O str. ester), 1709.30Cm^{-1} (C=O str. Carbonyl), 1673.47Cm^{-1}(C=O str. Carbonyl), 1595.44Cm^{-1}(C=C str. Ar.), 1523.20Cm^{-1}(NO_2 str.), 1382.28Cm^{-1}(NO_2 str.); Anal. Calcd for C_{16} H_{12} N_2 O_6: C, 68.78%; H, 4.19%; N, 9.27%; Found: C, 68.9%; H, 4.3%; N, 9.5%.
4-amino-N-ethylglycinate-1,8-naphthalimide	^{1}H NMR (CD_3COCD_3): δ 1.23790-1.28519(3H, CH_3) 4.15878-4.22975(2H, CH_2CH_3) 4.83966(2H, NCH_2), 6.88810(2H, NH_2), 6.99072-7.01846(1H, 3-H), 7.68011-7.73233(1H, 6-H), 8.28968-8.31744(1H, 2-H), 8.51144-8.53565(1H, 5-H), 8.61004-8.63814(1H, 7-H); FTIR (KBr): ν 3367.89, 3156.39Cm^{-1}(N-H str. Primary amine), 2924.08Cm^{-1}(C-H str. Aliphatic), 1730.04Cm^{-1}(C=O str. ester), 1686.12Cm^{-1}, 1643.14Cm^{-1}(C=0 str. carbonyl), 1577.56Cm^{-1}(C=C Ar.); Anal. Calcd for C_{16} H_{14} N_2 O_4: C, 64.43%; H, 4.7%; N, 9.39%; Found: C, 65%; H, 4.9%; N, 9.3%.

Table 2: FTIR, ^{1}HNMR and Elemental Analysis data of dyes.

Dyes	FTIR, ^{1}HNMR and Elemental Analysis data of dyes

1	^{1}H NMR (CDCl3): 1.27-1.29 (6H, $N(CH_2CH_3)_2$), 2.76 (3H, CH_3), 3.48-3.52(4H, $N(CH_2CH_3)_2$), 3.58 (3H, CH3), 6.60-6.65(2H, 3´-H, 5´-H), 7.76--7.79(1H, 6-H), 7.86-7.93(2H, 6´-H, 5-H), 8.56-8.63 (2H, 2-H, 7-H), 9.32-9.35(1H, 3-H.); FTIR (KBr): ν 2972.25 Cm^{-1}(C-H str. Ar.), 2932.22Cm^{-1} (C-H str. Aliphatic), 1695.59, 1659.37 Cm^{-1}(C=O str. carbonyl), 1588.21Cm^{-1}(N=N str.); Anal. Calcd for $C_{24}H_{24}N_4O_2$: C, 72%; H, 6%; N, 14%; Found: C, 71.5%; H, 5.8%; N, 13.8%; m p=195.70°C
2	^{1}H NMR (CDCl3): 1.27-1.34(9H, CH_2CH_3), 3.50-3.57(4H, $N(CH_2CH_3)_2$), 4.24-4.31(2H, $COOCH_2CH_3$), 4.98(2H, N-CH_2), 6.79-6.82(2H, 3-H, 5-H), 7.83-7.89(1H, 6-H), 7.97-8.06(3H, 2-H, 6-H, 5-H), 8.66-8.70(2H, 2-H, 7-H), 9.28-9.31(1H, 3-H); FTIR (KBr): ν 2960.77(C-H str. Ar.), 2924.95Cm^{-1} (C-H str. Aliphatic), 1748.4Cm^{-1}(C=O str.ester), 1698.61Cm^{-1}, 1662.05Cm^{-1}(C=O str. Carbonyl), 1596.4 Cm^{-1}(N=N str.); Anal. Calcd for $C_{26}H_{26}N_4O_4$: C, 68.12%; H, 5.68%; N, 12.2%; Found: C, 68.1%; H, 5.6%; N, 11.5%; mp=201.27°C
3	^{1}H NMR (CDCl3): 1.27-1.34(9H, CH_3), 1.61 (1H,$NHCOCH_3$, D_2O exchangeable), 2.35(3H, $NHCOCH_3$), 3.52-3.59(4H,N $(CH_2CH_3)_2$, 4.24-4.31(2H, $COOCH_2CH_3$), 4.98(2H, N-CH_2), 6.54-6.56(1H, 5-H), 7.82-7.89(2H, 6-H, 5-H),7.93-7.96(1H, 6-H), 8.18(1H, 3-H), 8.65-8.70(2H, 2-H, 7-H), 9.12-9.15(1H, 3-H); FTIR (KBr): ν 2976.64 Cm^{-1} (C-H str. Ar.), 2930.82 Cm^{-1} (C-H str. Aliphatic), 1747.05 Cm^{-1} (C=O str. ester), 1697.99, 1658.53 Cm^{-1} (C=O str. Carbonyl), 1588.77 Cm^{-1} (N=N str.); Anal. Calcd for $C_{28}H_{29}N_5O_5$: C, 65.24%; H, 5.63%; N, 13.59% Found: C, 65.1%; H, 5.8%; N, 13.4%; mp=276.11°C
4	^{1}H NMR (CDCl3): 1.31-1.36 (3H, CH_3), 4.25-4.32 (2H, $COOCH_2CH_3$), 4.90 (2H, NCH_2), 6.62-6.65 (1H, 3´-H, 5-H), 7.40-7.49 (3H, 6´-H,7´-H,8´-H), 7.62-7.65 (1H, 5´-H), 7.74-7.79 (1H,6-H), 8.17-8.2 (1H,5-H), 8.25-8.31(1H, 7-H), 8.39-8.42 (1H,4´-H), 8.55-8.57 (2H, 2-H,3-H),17.04 (1H,OH); FTIR (KBr): ν 3441.68 Cm^{-1} (OH str.), 2924.45 Cm^{-1}(C-H str. Ar.), 1748.83 Cm^{-1}(C=O str. ester), 1698.86, 1622.40 Cm^{-1} (C=O str. carbonyl), 1588.89 Cm^{-1} (N=N str.); Anal. Calcd for $C_{26}H_{19}N_3O_5$: C, 68.87%; H, 4.19%; N, 9.27%; Found: C, 68.9%; H, 4.3%; N, 9.5%; mp=237.94°C

Table 3: The spectrophotometric properties of the synthesized dyes.

Dye	$\Delta\lambda_{max}$	DMF λ_{Max}	Toluene λ_{Max}	DMF $\Delta\lambda_{1/2}$	Chloroform ϵ	λ_{Max}
1	22.7	549.9	523.2	123	40833	528.7
2	30.5	554	523.5	125	33196	528.5
3	18.2	562.7	544.5	114	49080	554.6
4	*-2.4*	*527*	*524.6*	*96*	*21368*	*525.2*

Table 4: Reflectance spectrophotometeric of dyed fabrics.

Dye	omf (%)	L^*	a^*	b^*
1	0.3	25.557	32.365	-15.006
	2	16.768	17.568	-4.018
2	0.3	40.909	33.793	-25.188
	2	21.575	26.817	-14.945
3	0.3	43.727	27.03	-30.686
	2	38.01	25.98	-23.282
4	0.3	41.235	56.948	8.59
	2	36.579	55.606	12.973

Table 5: Color fastness of dyed fabrics.

Dye	R.F				H.F	W.F				L.F
	W	D	W	D		AC		RC		
	AC		RC			C	N	C	N	
1	2-3	4	5	5	2	3-4	5	4-5	5	4-5
2	4	4-5	4	4-5	3	5	5	5	5	4
3	2	2-3	2	2-3	5	3	4-5	5	5	3-4
4	5	5	5	5	5	5	5	5	5	2-3

L.F = Light fastness, W.F = Wash fastness, H.F = Heat fastness, D = Dry,

W = Wet, R.F. = Rubbing fastness, RC= Reduction cleared, Ac= Alkali cleared, C= cotton, N= Nylon

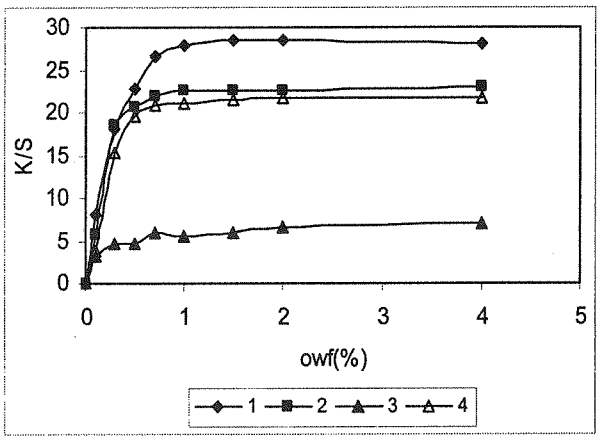


Figure 3. Build up property of dyes.

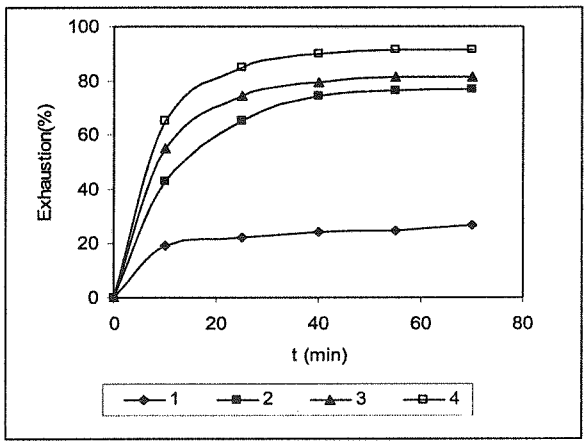


Figure 4. Rate of dyeing of dyes on polyester.

4. CONCLUSION

A series of monoazo disperse dyes based on N-ester-1,8-naphtalimid were synthesized from acenaphthene. In this respect, nitration, oxidation, imidation, reduction, diazotization and coupling reactions were performed. The synthesized dyes were characterized with analytical instruments method and applied on polyester fabrics.

The synthesized dyes are new organic dyes for polyester fabrics, on which they exhibit relatively good dyeing properties, good build up, and excellent wash fastness. The synthesized dyes offered red to bluish red on polyester.

Some of dyes have alkali-clearable properties in alkali media. Therefore, using these dyes for coloring of the polyester can eliminate consumption of sodium hydrosulphite, which is the main source of pollution in textile wastewater.

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