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Synthesis and Characterization of Monoazo Disperse Dyes Derived from Naphthalimide Derivatives

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ABSTRACT

New series of monoazo disperse dyes were synthesized from naphthalimide derivatives by coupling intermediate A [6-amino-2-(4-aminophenyl)-IH-benzo[de]isoquinoline-1,3(2H) dione] with different substituted anilines and naphthalenes in appropriate reaction condition. The synthesized intermediates and the dyes were purified by recrystallization method. The structure of the synthesized dyes were confirmed using FTIR, 1HNMR, MS and UV-visible spectroscopic techniques. Spectrophotometric investigations of the dyes in solvent of different polarities were measured to obtain absorption maxima, molar extintion coefficient and salvatochromic effects These dyes gives percentage yield of 72-85%, molecular weight of 435-467 g/mol and melting ing point of 76-208℃ with hues such as orange, yellow and purple respectively.

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INTRODUCTION

Disperse dyes are non-ionic colourants with sparing solubility in water which are able to retain better substantivity for hydrophobic fibres, such as polyester, nylon and acetate [1]. They are characterized by their nonpolar chemical structures, rendering them sparingly soluble in water [2]. This characteristic allows them to disperse readily in organic solvents or nonpolar media, facilitating their application in dyeing synthetic fibres. Additionally, disperse dyes possess molecular structures that enable them to penetrate hydrophobic fibres, forming strong bonds that result in excellent colour fastness and resistance to washing and light exposure. Disperse dyes find widespread application in dyeing synthetic fibres, particularly polyester, acetate, and nylon [3].

Polyester fibre is the most widely produced of all synthetic fibres and has a very compact and crystalline structure [4,5]. Disperse dyes are typically regarded as the best options for colouring polyesters because of their good to exceptional light and washing fastness capabilities [6,7]. They are used in various textile processes, including dyeing yarns, fabrics, and garments. With a broad spectrum of colour options, disperse dyes are extensively utilized in the fashion industry to produce vibrant and durable-coloured textiles. Moreover, they are employed in other sectors such as printing inks, plastics, and leather dyeing. The presence of the naphthalimide moieties in the dye structure improved the fastness properties of the dyes and bring about deep shade, brilliant hues of high bathachromic effects due to donor acceptor character of the amino and carbonyl groups present in the napthalimide moiety. This investigation aimed at synthesis, and characterization of monoazo disperse dyes derived from naphthalimide derivatives.

MATERIALS AND METHODS

All the chemicals used in the synthesis for this research were of commercial grade. The synthesized compounds were characterized by melting point, TLC, FTIR, ^{1}HNMR and GC.MS. Melting points were recorded on Gallenkamp C10127. Infra-red was recorded using Bruker-Tensor 27 Platinum ATR-FTIR Spectrophotometer,^{1}H NMR were carried out on

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Bruker Avance FT NMR Spectrometer for ^{1}H NMR determination using deuterated chloroform ($CDCl_3$) and dimethlsulphoxide (DMSO-d_6) as solvent. Chemical shifts were reported in parts per million(ppm) and the mass spectra were recorded using Agilent technologies 5975 C VL MSD spectrometer.

Synthesis of 5-Nitroacenapthene

Acenaphthene (15.4 g, 0.1 mol) was dissolved in hot glacial acetic acid (57.2 ml) and was cooled with vigorous stirring to separate crystalline acenaphthene as fine as possible. Concentrated nitric acid (9.84 g, 0.156mol) was added drop wise to the mixture for 1 hr at 22–27 °C and then further stirred for 1 hr at 25 °C. The solution was warmed gradually for 30 mins to 68 °C then cooled to form crystals that were filtered, washed with 1:1 acetic acid–water and water to give yellow needles [8].

Synthesis of 4-nitronaphthalic anhydride

5-Nitroacenaphthene (24.87 g, 0.125 mol) was dissolved in hot glacial acetic acid (248.75 ml), sodium dichromate (158.5 g) was added portion wise for 3 hr at 65–70 °C. The solution was warmed gradually to 98–100 °C for 30 minutes and further refluxed for 5 hr. The contents were wash out with hot water (0.6 L) and cooled to room temperature. The precipitate was filtered and washed with dilute HCl (10 %). In addition, the precipitate was boiled with 200 mL of 5 % Na_2CO_3 solution for 30 mins and filtered. The filtrate was acidified and the separated crystals dried at 120 °C for 4 hours to obtain 4-nitronaphthalene-1,8-anhydride, which was recrystallized from concentrated HNO_3 (d = 1.40 g /mL) to afford colourless needle crystals [8].

Synthesis of N-Amino- 4-nitronapthalic anhydride

A suspension of 4-nitronapthalic anhydride (0.02 mol, 4.86 g) and benzene -1, 4-diamine (0.03 mol, 5.55 g) was stirred under reflux for 7 hr in ethanol (102 mL). The cooled suspension was filtered out and recrystallized with ethanol to give yellow crystals.

Synthesis of 4-amino -N- substituted 1, 8-Napthalimide

A mixture of N-Amino- 4-nitronapthalic anhydride (a) 18 mmol, 6.88 g) and stannous chloride (90 mmol, 20.25 g) in ethanol (81 mL) was refluxed for 1 hr with hydrochloric acid (10.1 mL) until the reaction was completed. The mixture was transferred into 100ml of water and the observed precipitate was filtered out. The crude product was purified by recrystallization from ethanol as yellow crystals.

Synthesis of dyes

Sodium nitrite (1.5 mmol, 0.104 g) was slowly added with constant stirring to cold concentrated sulphuric acid (1.1 mL) at room temperature such a rate that yellow fume was not evolved. The temperature of the reaction mixture was gradually increased to 65 °C using water bath until all the sodium nitrite was dissolved. The resulting solution was then cooled to 0-5 °C and a mixture of propionic acid and glacial acetic acid (10mL, 1.5:8.5 volume ratio) was added drop wise at 5 -20 °C. The Finely ground powder of 1.5 mmol 4-amino-N-subtituted-1,8-naphthalimide was added portion wise below 10 °C, and then the mixture was further stirred for 3 hr. The obtained clear diazonium salt solution was used immediately in coupling reactions.

The coupling components, 2-naphthol, 3-nitroaniline, 2,4- nitroanaline, were each dissolved with ethanol and few drops of acetic acid was added at 0-5 °C. The previously prepared diazonium solution was added over 30–40 minutes under vigorous stirring. The mixture was stirred further for 2 hours at 0-5 °C and the pH of the solution was adjusted to 4–5 using 10 % sodium acetate and stirred for 1 hr. The resulting product was then collected by filtration, washed with warm water and then cold water and dried. The crude product was purified by recrystallization from DMSO several times. Similar procedure was used for the coupling components N, N-dimethylaniline, N, N- diethylaniline but without ethanol.

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Percentage Yield

The percentage Yield is determined using the formula in equation 3.1

% Yield =

$$\frac{\pi Actual\ mass\ of\ product}{2Theoretical\ mass\ of\ product} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots 1$$

Melting point determination

The melting points were determined by the open capillary method and are expressed in degree centigrade (°C). Small amounts of each of the synthesized dyes were poured into the capillary tube, which was then carefully inserted into the Gallenkamp melting point apparatus. The melting point was recorded by closely monitoring the apparatus as it gradually heated the substance, until the precise melting point was reached.

SPECTROSCOPIC ANALYSIS

Determination of UV VISIBLE Analysis (λ max)

The wavelength of maximum absorption of each of the synthesized intermediates and dyes were determined in different solvents like dimethylformamide (DMF), chloroform, ethanol and ethanol plus a drop of hydrochloric acid. The spectra of the intermediates and dyes solutions were determined and recorded using UV- Visible spectra scanning (Agilent CARY 300 UV- Visible spectrophotometer).

Fourier Transform Infrared (FTIR) Analysis

About 1.5mg of finely ground synthesized samples was mixed with about 150mg of powdered potassium bromide (KBr). The finely ground mixtures were introduced in the beam of IR spectrometer and a blank KBr pellet was kept in the path of reference beam and run (Bruker-Tensor 27 Platinum ATR-FTIR Spectrophotometer.

Nuclear Magnetic Resonance (NMR) Analysis

About 20mg of the synthesized sample was dissolved in 0.6 cm3 of the chosen solvents (DMSO-de). The solution was filtered into 5mm NMR sample tube through a Pasteur pipette containing an oven dried glass wool plug. The tube was closed with a cap; the top was sealed with paraffin to reduce evaporation. The glass tube containing the sample to be analysed was held in a plastic spinner. A sample depth gauge was provided to control the depth of the sample tube in the spinner. This is to ensure that the sample was correctly aligned with the coils inside probe. The depth gauge has a graduated scale that set the top of the white plastic base between 18mm and 20mm below the center (0mm line) for 5mm probes. The depth gauge was removed before inserting the sample and spinner into the magnet (Bruker Avance FT NMR Spectrometer for ^{1}H NMR determination.

Mass Spectroscopy (MS) Analysis

A mass Spectroscopy (MS) technique was used to determine the molecular mass of the compounds using Agilent Technologies 5975C VL MSD (MS) machine.

RESULTS AND DISCUSSION

Table 1: Physical properties of intermediates

Compounds	Molecular formula	Molecular weight (g/mol)	Melting point (0C)	Yield (%)	Colour Observed
5-nitroacenaphthene	C12H9NO2	199	101-102	86.2	Yellow
4-nitronapthalic anhydride	C8H3NO5	193	231-232	70.1	Off white
6-amino-2-(4-aminophenyl)-IH-benzo[de]isoquinoline-1,3(2H) dione	C18H13N3O2	303	278-280	69.5	Yellow

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Table 2: Physical properties of the dyes

Dye No:	Molecular formula	Molecular weight (g/mol)	Melting point (°C)	Yield (%)	Colour Observed
DA$_1$	$C_{28}H_{18}N_4O_3$	458	193-195	80.2	Orange
DA$_2$	$C_{24}H_{17}N_7O_4$	467	76-78	70.3	Yellow
DA$_3$	$C_{18}H_{13}N_7O_4$	391	182-184	82.1	Yellow
DA$_4$	$C_{28}H_{19}N_5O_2$	457	201-202	85.4	Orange
DA$_5$	$C_{26}H_{21}N_5O_2$	435	206-208	72.5	Yellow

Table 3: The FT-IR and ^{1}H NMR Spectra of the synthesized Intermediate and Dyes

Dyes	FT-IR (cm-^{1})	^{1}H NMR
I$_A$	3462 (N-H str,), 3071, 2974 (C-H str. Aromatic, Aliphatic), 1707 (-N-C=O-str.), 1587(N=N str.), 1353(C-N str.).	(400 MHz, DMSO) δ 9.84 (s, 1H), 8.77 (d, *J* = 8.7 Hz, 1H), 8.62 (dd, *J* = 14.9, 6.4Hz, 2H), 8.13 (t, *J* = 8.0 Hz, 1H), 7.49 – 7.36 (m, 3H), 7.36 – 7.22 (m, 4H), 4.09 (s, 1H).
DA$_1$	3427 (O-H str.), 3072, 2937(C-H str. Aromatic, Aliphatic), 1990, (-N-C=O-str.), 1581(N=N str.), 1349(C-N str.).	(400 MHz, DMSO) δ 9.84 (s, 1H), 8.77 (d, *J* = 8.7 Hz, 1H), 8.62 (dd, *J* = 14.9, 6.4Hz, 3H), 8.44- 7.91 (m, 2H), 7.75 – 7.17 (m, 8H), 7.17-6.53(m, 1H), 4.09 (s, 3H).
DA$_2$	3460(N-H str.), 3334, 1889(-N-C=O-str.), 1889, 1517(N=N str.), 1422 (NO_2 str.), 1337 (C-N str.).	(400 MHz, $CDCl_3$) δ 9.75 (s, 1H), 8.77 (d, *J* = 8.7 Hz, 4H), 7.5 – 7.0 (m, 6H), 5.3 – 5.0 (m, 2H), 2.25 (m, 3H), 1.25 (s, 1H).
DA$_3$	3440 (N-H str.), 3070, 2977, 2862 (C-H str. Aromatic, Aliphatic), 1860, (N-C=O str.), 1519(N=N str.), 1344(C-N str.)	(400 MHz, CDCl3) δ 9.76 (s, 1H), 8.90 (d, *J* = 8.7 Hz, 1H), 8.79 (d, *J* = 7.2 Hz, 1H), 8.74 (d, *J* = 8.0 Hz, 1H), 8.44 (d, *J* = 8.1 Hz, 1H), 8.03 (s, 1H), 7.82 (d, *J* = 8.2 Hz, 1H), 7.72 (t, *J* = 10.4 Hz, 2H), 7.56 (t, *J* = 6.5 Hz, 2H), 7.42 (t, *J* = 7.7 Hz, 1H), 7.36 (d, *J* = 8.2 Hz, 1H),7.10 (d, *J* = 9.1 Hz, 1H), 5.52 (s, 2H), 5.06 (s, 3H).
DA$_4$	3365(N-H str.), 3071(C-H str.), 1928 N-C=O-str.), 1519(N=N str.), 1344(C-N str.).	(400 MHz, DMSO) δ 9.0-8.60 (s, 1H), 8.90-8.0 (d, *J* = 4.0 Hz, 4H), 7.50 – 7.0 (m, 2H), 3.0 – 2.0(m, 5H), 1.5-0.5 (m, 7H).
DA$_5$	3447(N-H str.), 3034(.2C-H str.), 1848(-N-C=O-str.), 1508(N=N str.), 1331(C-N str.).	(400 MHz, CDCl3) δ 9.0-8.0 (m, 6H), 7.5.-7.0 (d, *J* = 8.6 Hz, 6H), 4.50-4-0 (d, *J* = 7.2 Hz, 2H), 3.50-3.0(3H), 2.70-2.30(5H).

Table 4: Mass Spectrometry of Synthesized Intermediates and the Dyes

Dye No	Empirical formula	Experimental values of m/z fragment	Corresponding positive charge fragment	Theoretical value
I$_A$	$C_{18}H_{13}N_3O_2$	55.1,81.1,109.1,135.0,163.0,263.3,303.4.	C_2NO^+, $C_4H_5N_2^+$, $C_6H_7NO^+$, $C_7H_7N_2O^+$, $C_9H_9NO_2^+$, $C_{16}H_{11}N_2O_2^+$, M	303.31
DA$_1$	$C_{28}H_{18}N_4O_3$	55.0, 97.0, 129.0, 207.0, 265.2 , 458.4.	C_2NO^+ ,$C_5H_7NO^+$, $C_6H_{10}NO_2^+$,	458.47

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Dye No	Empirical formula	Experimental values of m/z fragment	Corresponding positive charge fragment	Theoretical value
DA_2	$C_{24}H_{16}N_6O_4^+$	57.1, 95.0,127.0, 183.3,239.2, 411.4, 452.4	$C_{11}H_{15}N_2O_2^+$, $C_{16}H_{12}N_2O_2^+$, M $C_4H_{10}^+$, $C_5H_5NO^+$, $C_7H_{13}NO^+$, $C_{10}H_5N_2O_2^+$, $C_{14}H_{29}^+$, $C_{24}H_{17}N_3O_4^+$	452.42
DA_3	$C_{24}H_{17}N_7O_4$	57.1,97.1,127.2,383.4, 467.4	$C_{23}H_{16}N_3O_2^+$ $C_4H_{10}^+$, $C_5H_7NO^+$, $C_7H_{13}NO^+$,	467.44
DA_4	$C_{28}H_{25}N_5O_2$	55.1,97.1,207.0, 281.0,341.0, 463.2	$C_{23}H_{16}N_3O_3^+$,M C_2NO^+, $C_5H_7NO^+$, $C_{11}H_{15}N_2O_2^+$, $C_{17}H_{17}N_2O_2^+$,	463.53
DA5	$C_{26}H_{21}N_5O_2$	55.0,105.0,276.0,411.0,439.0	$C_{21}H_{15}N_3O_2^+$, C_2NO^+, $C_8H_9^+$, $C_{17}H_{17}N_2O_2^+$, $C_{25}H_{21}N_3O_3^+$,M	439.48

Acenaphthene **(1)** reacted with nitric acid in an acetic acid media at room temperature to give yellow crystals of 5-nitroacenaphthene after recrystallization from ethanol with a yield and melting point of 86 % and 101-102 °C respectively. 5-nitroacenaphthene **(2)** was oxidised using sodium dichromate and hot glacial acetic acid as the solvent under reflux condition of 5hr to give of white crystals of 4-naphthalic anhydride after recrystallization from nitric acid with a yield and melting point of 70.1 % and 231-232°C. 4-nitronapthalic anhydride**(3)** was reacted with 1,4- diamino benzene under reflux for 7 hr in ethanoic media and recrystallized with ethanol to give yellow crystals of 4-nitro-N-substituted-1, 8-naphthalimide. 4-nitro-N-substituted-1, 8-naphthalimide **(4)** was reacted with stannous chloride in hydrochloric acid as a reducing agent and ethanol as solvent to give yellow crystals of 4-amino -N- substituted 1, 8- Napthalimide. 6-amino-2-(4-aminophenyl)-1H-benzo [de] isoquinoline - 1,3(2H)-dione which is the intermediate with a yield and melting point of 69.1 % and 278-280 °C respectively.

The reaction sequence is shown in scheme 1. The structure of the intermediates were confirmed by FTIR, ^1H NMR, and mass spectroscopic techniques as shown in Table 3 and 4. The IR spectrum of the intermediate 6-amino-2-(4-aminophenyl)-1H-benzo[de]isoquinoline-1,3(2H)-dione showed absorption at 3462 which indicates aromatic N-H stretch, the signal at 2954 correspond to C-H stretch of aliphatic. The signal at 1707 is due to carbonyl (C=O) stretch. The signal at 1583 corresponds to N=N stretch, while the signal at 1353 correspond to (C-N) stretch. The ^1H-NMR spectrum, the peak at 9.84ppm (1H) correspond to protonated nitrogen atom in a heterocyclic ring, peaks at 8.77-7.22ppm (11H) correspond to aromatic protons. The peak at 4.09 ppm (1H) showed a downfield correspond to aliphatic protons. The Mass spectrometry analysis showed a molecular ion$[M^+]$ at m/z 303.4 which is in agreement with the formula mass 303.31$gmol^{-1}$.

The diazotization of 6-amino-2-(4-aminophenyl)-1H-benzo[de]isoquinoline-1,3(2H)-dione was carried out using nitrosyl sulphuric acid method and coupling with various electron-rich arenes produce the dyes shown in the reaction (Scheme 1). The structures of the dyes were characterized using FT-IR and^1H NMR, MS and UV spectroscopic methods. The results of the characterization are shown in Table 3 and 4. Figures 2–7 showed the FT-IR, ^1HNMR and Mass spectra of the dyes.

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Spectroscopic characteristics of the synthesized monoazo disperse dyes A$_1$-A$_5$ derived from naphthalimide derivatives

Spectra Analysis of dye DA$_1$

Intermediate A (I$_A$) was diazotized using nitrosyl sulphuric acid method and coupled with 2-napthol to give dye DA$_1$. The yield and melting point were 80.2% and 193-195 ^{0}C respectively. The IR spectrum showed absorption at 3427 which is due to OH stretch while the signal at 3072, is due to aromatic C-H stretch. The signal at 2937 is attributed to C-H stretch aliphatic while signal at 1990 indicates a carbonyl (C=O) stretch. The azo (-N=N-) was observed at 1581 while signal at 1349 indicates a C-H stretch. The ^{1}H-NMR spectrum revealed that peaks at 9.84-719ppm correspond to (7H) of aromatic protons, peaks at 7.75-6.53 correspond (9H) protons adjacent to the aromatic region. The peaks at 4.09 ppm correspond aliphatic protons. The MS (analysis showed a molecular ion [M$^+$] at m/z 458.4which is in agreement with the formula mass 458.47gmol^{-1}.

Spectra Analysis of dye DA$_2$

Intermediate A (I$_A$) was diazotized using nitrosyl sulphuric acid method and coupled with 3-nitroaniline to give dye DA$_2$. The yield and melting point were 70.3% and 76-78^{0}C respectively. The IR spectrum showed absorption at 3460 which is due to N-H stretch while the signal at 3336 is due C-H stretch. while signal at 1889 indicates a carbonyl C=O stretch. The signal is 1517 and 1337 represents -N=N- and C-N stretch respectively. The ^{1}H-NMR spectrum showed the peaks at 9.75-8.77correspond to (5H) in the aromatic region, the peaks at 7.50-7.0ppm correspond to the (6H) at the aromatic region and also peaks at 5.30-5.0ppm correspond to the (2H), adjacent to double bond. Peaks at 2.25-1-24 correspond to (4H) of a methylene proton. The MS analysis showed a molecular ion [M$^+$] at m/z 452.4 which is in agreement with the formula mass 452.42gmol^{-1}.

Spectra Analysis of dye DA$_3$

Intermediate A (I$_A$) was diazotized using nitrosyl sulphuric acid method and coupled with 2,4-nitroaniline to give dye DA$_3$. The yield and melting point were 82.1% and 182-184^{0}C respectively. The IR spectrum showed absorption at 3406 which is due to N-H stretch while the signal at 3331and 2980 are due to aromatic and aliphatic C-H stretch. While signal at 1931 indicates a carbonyl N-C=O stretch. The signal at 1581 and 1300 represents -N=N- and C-N stretch respectively. The MS analysis showed a molecular ion [M$^+$] at m/z 467.4 which is in agreement with the formula mass 467.44 gmol^{-1}.

Spectra Analysis of dye DA$_4$

Intermediate A (I$_A$) was diazotized using nitrosyl sulphuric acid method and coupled with Diethylaniline to give dye DA$_4$. The yield and melting point were 85.4. % and 201-202^{0}C respectively. The IR spectrum showed absorption at 3365 which is due to N-H stretch while signals at 3071 due to C-H stretch. While signal at 1928 indicates a carbonyl N-C=O stretch. The signal at 1514 and 1355 represents -N=N- and C-N stretch respectively. The MS analysis showed a molecular ion [M$^+$] at m/z 463.2 which is in agreement with the formula mass 463.53 gmol^{-1}.

Spectra Analysis of dye DA$_5$

Intermediate A (I$_A$) was diazotized using nitrosyl sulphuric acid method and coupled with Dimethylaniline to give dye DA$_5$. The yield and melting point were 72.5. % and 206-208^{0}C respectively. The IR spectrum showed absorption at 3447 which is due to N-H stretch while signals at 3034 due to C-H stretch. While signal at 1944 indicates a carbonyl N-C=O stretch. The signal at 1509 and 1331 represents -N=N- and C-N stretch respectively. The ^{1}H-NMR spectrum showed peaks at 9.0-8.0ppm correspond to (6H) protons in aromatic region, peaks at 7.5-6.50 correspond to (11H) in aromatic region whereas peaks at 4.10ppm correspond to (7H) in aliphatic region. The MS analysis showed a molecular ion [M$^+$] at m/z 439.6 which is in agreement with the formula mass 439.38gmol^{-1}.

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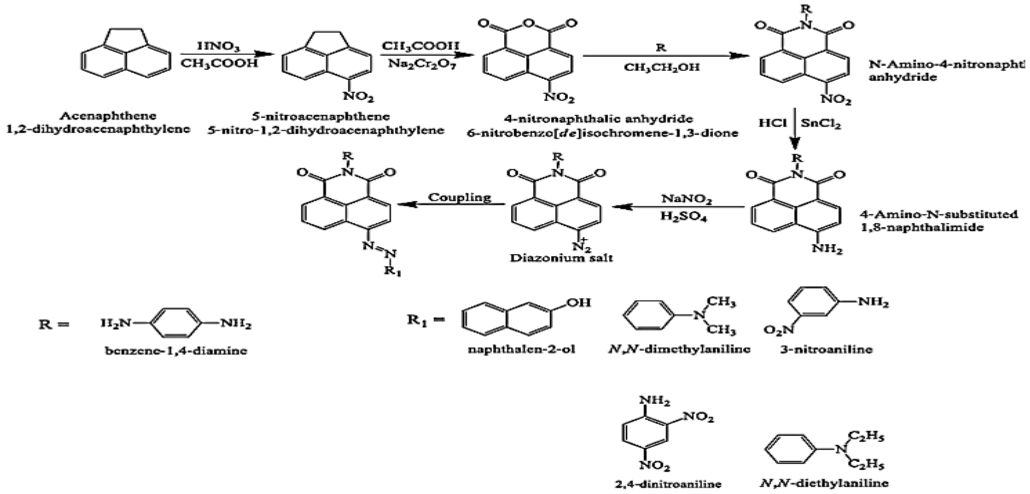

Scheme 1: Synthesis of intermediates and the dyes

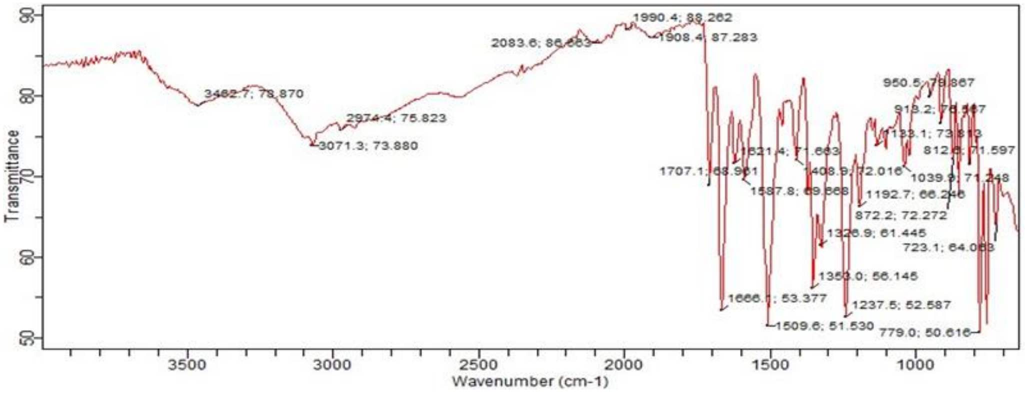

Figure 1: FTIR of Intermediate A

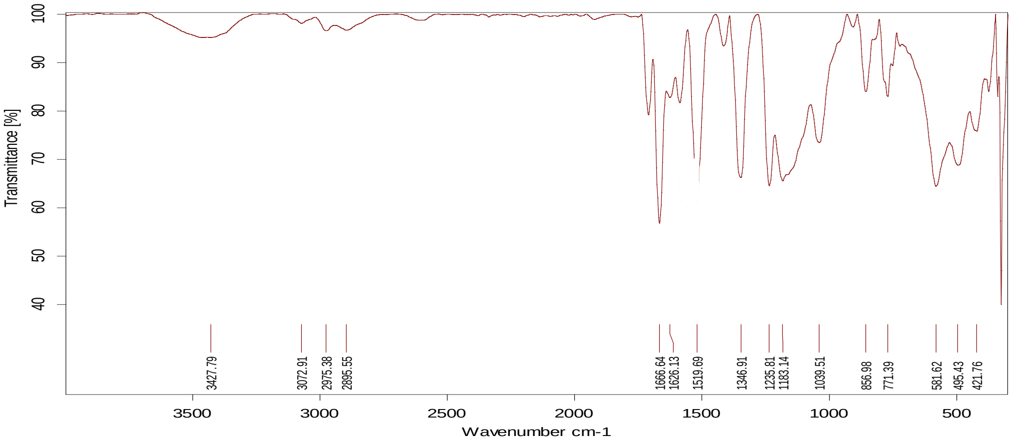

Figure 2: FTIR of dye A1

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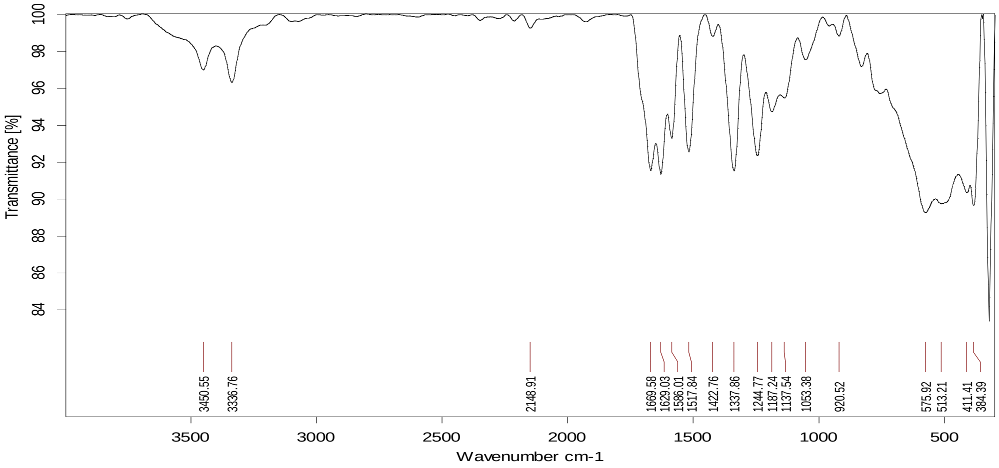

Figure A. 7: FTIR of dye A_2

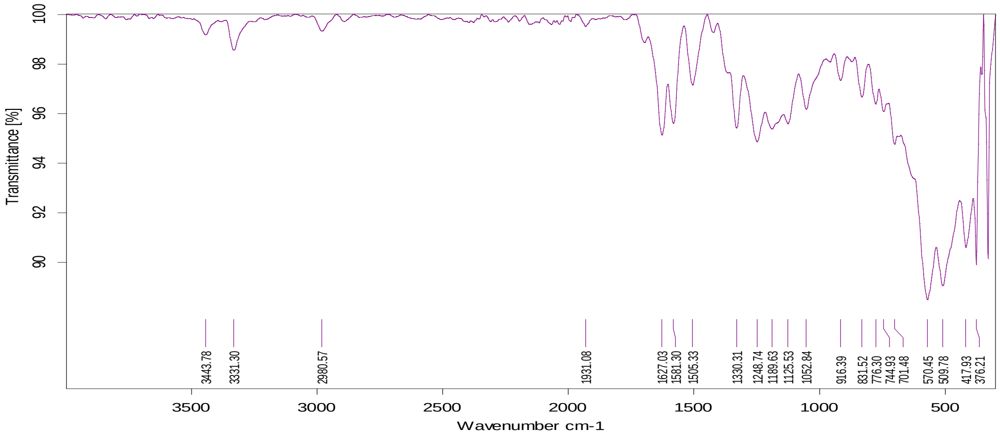

Figure 3: FTIR of dye A_3

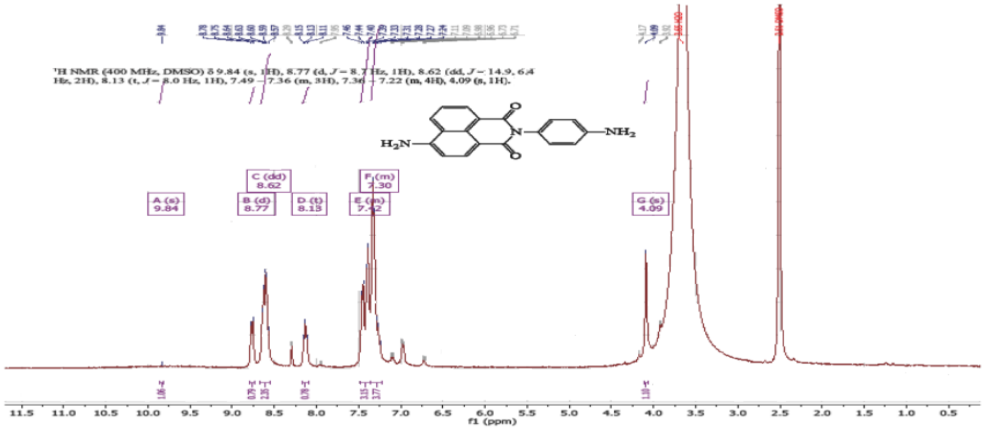

Figure 4: ¹H NMR of Intermediate A

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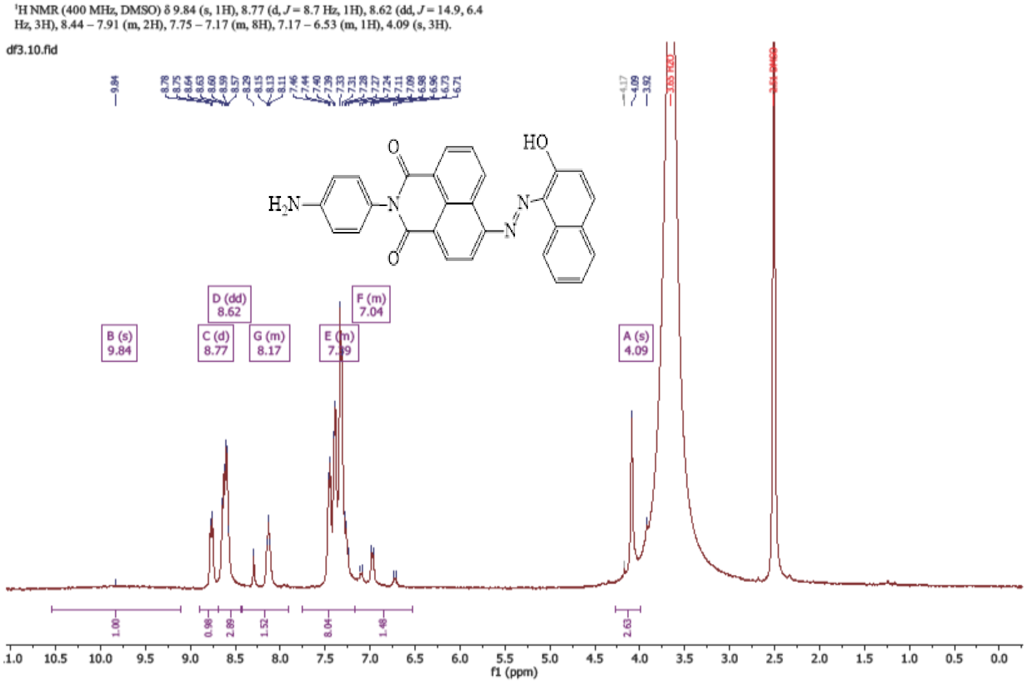

^{1}H NMR (400 MHz, DMSO) δ 9.84 (s, 1H), 8.77 (d, J = 8.7 Hz, 1H), 8.62 (dd, J = 14.9, 6.4 Hz, 3H), 8.44 – 7.91 (m, 2H), 7.75 – 7.17 (m, 8H), 7.17 – 6.53 (m, 1H), 4.09 (s, 3H).

Figure 5: ^{1}H NMR of dye A_1

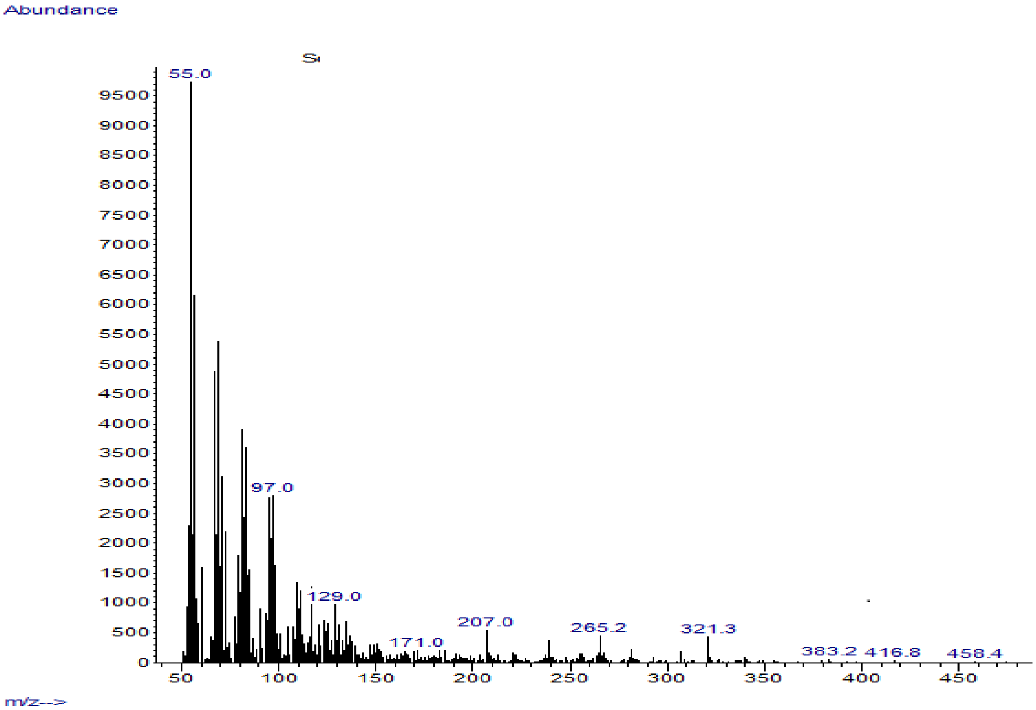

Figure 6: GCMS of dye A_1

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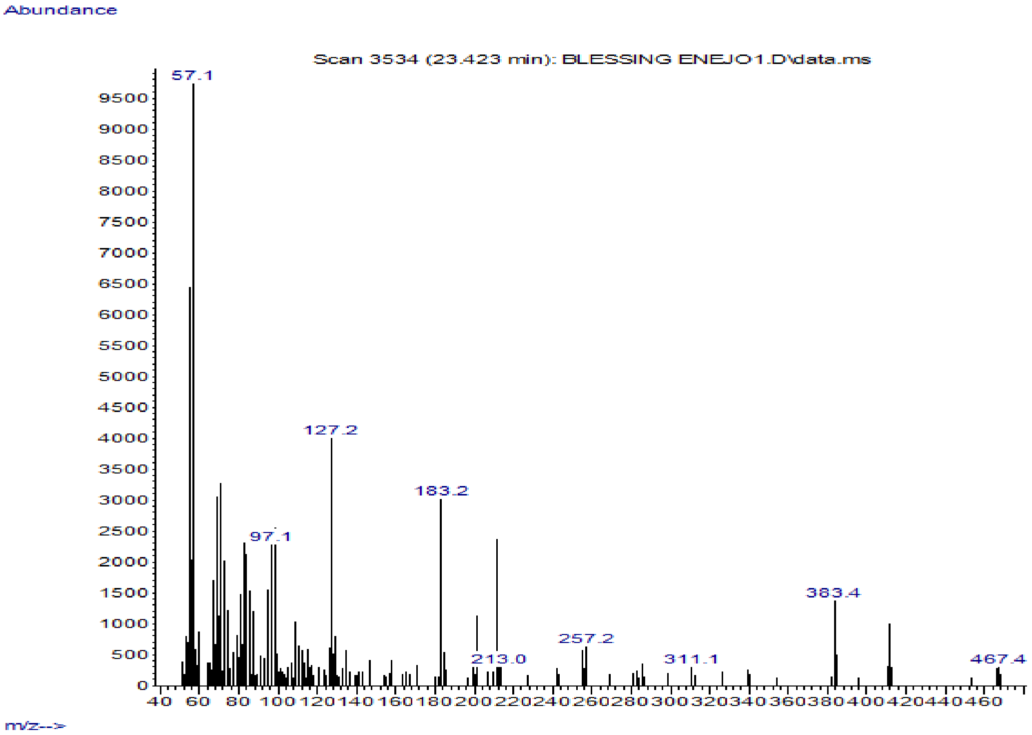

Figure 7: GCMS of dye A3

CONCLUSION

In this research, an efficient and simple protocol for the syntheses of monoazo disperse dyes derived from naphthalimide was developed in good yields, starting with the following reaction; nitration, oxidation, amination and reduction to synthesized the intermediate A[6-amino-2-(4-aminophenyl)-IH-benzo[de]isoquinoline-1,3(2H) dione]. The structures of the intermediates and the dyes were confirmed using FTIR, ^{1}HNMR, Mass and UV-visible spectroscopic techniques.
Data obtained by various analytical techniques proved the synthesis of the claimed disperse dyestuffs. It was found that by changing the coupling components, the bathochromic and hypsochromic properties of the dyes varied, which could be related to the structure of the replaced groups.

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