INTERNATIONAL JOURNAL OF CURRENT RESEARCH IN CHEMISTRY AND PHARMACEUTICAL SCIENCES
(p-ISSN: 2348-5213: e-ISSN: 2348-5221)
www.ijcrcps.com
(A Peer Reviewed, Referred, Indexed and Open Access Journal)
DOI: 10.22192/ijcrcps Coden: IJCROO(USA) Volume 9, Issue 2 - 2022

Research Article

DOI: http://dx.doi.org/10.22192/ijcrcps.2022.09.02.003

Synthesis and Characterization of New Heterocyclic Azo dyes

Dr Alka Pradhan*, Pratibha Dwivedi**
*Department of Chemistry, Govt. Motilal Vigyan Mahavidyalaya, Bhopal, M.P. India-462008
**Department of Chemistry, Govt. Motilal Vigyan Mahavidyalaya, Bhopal, M.P. India-462008

Abstract

The synthesis of novel preparation of substituted ethyl (4-methoxyphenyl) methyl) carbamothioyl)diazenyl)-3-oxobutanoate heterocyclic coupling agent. To create a novel heterocyclic azo pigment, the coupling agent was subjected to an azo coupling reaction with an aromatic amine, After that, the pigment was tested for solubility, hiding power, and light fastness. The pigment's properties and structural features were determined using Fourier Transform Infrared Spectroscopy (FTIR), Ultraviolet/Visible (UV/Vis) Spectroscopy, and Nuclear Magnetic Resonance Spectroscopy (1 H-NMR, 13C-NMR).

Keywords: aromatic amines, Heterocyclic azo dyes, spectral analysis.

Introduction

Azo dyes are a versatile group of coloured organic compounds that have found widespread use in industry and analytical science. The chromogenic markers o, dihydroxy azodyes, which have been utilised as metal chelates for colouring protein fibres, are especially helpful in analytical chemistry.[1-5] Perkin identified the previous synthetic dyestuff, Mauveine, in the 1850s [6]. Dyes have become essential tools in a wide range of applications [7]. They are used as colourants in plastics, paper, ceramics, coatings, structures, textiles, and other high-tech applications [8-11]. Sensors, inks, tinting, adhesives, drinks, cosmetics, cuisine, polymers, optical data storage, leather, and wax biomedicine are just a few of the applications for dyes [12-18]. Colour fastness to sublimation, light, wash, perspiration and rubbing are common qualities of dyes used for textile dyeing. Dyes used in the textile industry are now considered mature. Nonetheless, it is a vibrant and challenging area that involves the ongoing manufacture of novel materials to meet the rapidly changing needs of global clients.[19-27]

Materials and Methods

All of the reagents and solvents used were of the best standard. Which were used without being purified further. The melting point was measured

in uncorrected open capillary tubes. In KBr pellets, IR spectra were obtained on an FT-IR-Alpha Bruker IR spectrometer from 4000 cm1 to 400 cm1. The 1H and 13C NMR spectra were obtained in DMSO-d6 at 500 MHz with tetramethylsilane as an internal standard using an AV500 High-Resolution Multinuclear F-NMR Spectrometer. A Jeol-8X 102 (FAB) Mass Spectrometer was used to record LC-MS spectra.

Synthesis series of novel heterocyclic azo dyes: To a magnetically stirred compound (half hours) and cooled solution of novel synthesized amines (0.01mol) added aqueous hydrochloric acid (1:1 80 ml) the content was stirred and then cooled (0-5^{0}C) on water bath further cold solution of sodium nitrite (12.9 g, 30 ml water) was added slowly and maintaining the freezing temp condition. This cold diazotized solution was added dropwise to the prepared cooled and stirred mixture of ethyl acetoacetate (11.83 ml) and sodium acetate (13.19g) dissolve in 10 ml of 50 % aqueous ethanol. The crude was filtered under vacuum, washed with water, and recrystallized from ethanol.

Synthesis Scheme

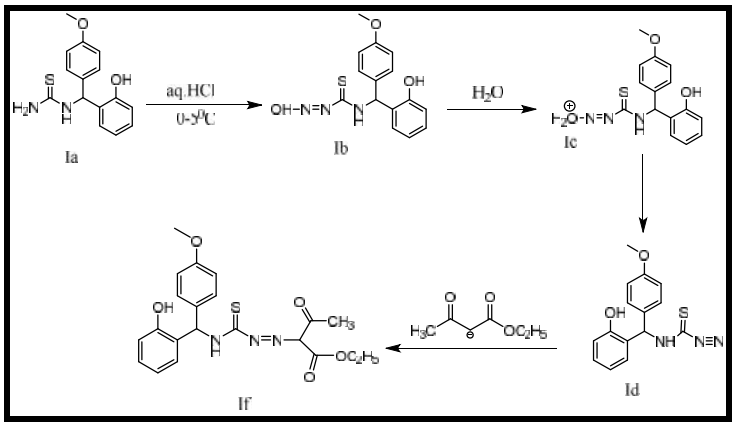

Table 1 Physical data of novel synthesized azo dyes series

Comp. Code	Molecular Weight	Melting Point ^{0}C	Molecular Formula	Yield (%)	Color
If	479.15	162	$C_{25}H_{35}N_3O_5S$	66	Yellow
IIf	474.12	160	$C_{21}H_{22}N_4O_7S$	62	Yellow
IIIf	445.13	135	$C_{21}H_{22}N_3O_6S$	58	Yellow
IVf	429.14	125	$C_{21}H_{23}N_3O_5S$	54	Yellow

Results and Discussion

ethyl-2-(((1-hydroxy-naphthalen-2-yl)(4-methoxyphenyl)methyl)carbamothioyl)diazenyl)-3-oxobutanoate (comp.code If) IR (KBr) cm-^{1}: 3441 (Ar-OH stretching), 3089 (NH stretching), 2946 (CH stretching), 1267(C=S), 1176(C-N stretching), 1445(N=N), 2972(CH_3), 1648(C=O) **^{1}HNMR (CDCl3) δ:** 8.18 (d,1H,NH), 9.68 (s,1H,OH), 6.11(s,1H,CH), 2.24(s,3H) ,1.21(t,3H),4.21(qt.2H), 7.08, 6.56,6.83,(m, 3H, Ar-H),7.21, 7.32, 6.89, 6.85 (m, 4H, Ar H), 3.81(s,3H CH_3) **m/z:** 479.15

ethyl-2-(((4-hydroxy-3-nitrophenyl)(4-methoxyphenyl)methyl)carbamothioyl)diazenyl)-3-oxobutanoate (comp.code IIf) IR (KBr) cm-^{1}: 3545 (Ar-OH stretching), 3115 (NH stretching), 2866 (CH stretching), 3155(NH2 stretching), 1290(C=S), 1166(C-N stretching), 1463(N=N), 2972(CH_3), 1670(C=O) **^{1}HNMR (CDCl3) δ:** 8.18 (d,1H,NH),9.65 (s,1H,OH),

5.11(s,1H,CH), 2.24(s,3H) 5.25(s, NH_2),1.21(t,3H),4.21(qt.2H), 6.08, 6.56,6.83,(m, 3H, Ar-H),7.21, 7.32, 6.89, 6.85 (m, 4H, Ar H), 3.81(s,3H CH_3) **^{13}CNMR (CDCl3) δ:** 164.5(C=O),124(Ar), 129(Ar), 52.6(CH), 25.2(CH_3), 55(CH_2) **m/z:** 474.12

ethyl-2-(((2,5-dihydroxy-phenyl)(4-methoxy phenyl)methyl)carbamothioyl)diazenyl)-3-oxobutanoate (comp.code IIIf) IR (KBr) cm^{-1}: 3555 (Ar-OH stretching), 3059 (NH stretching), 2850 (CH stretching), 1268 (C=S), 1156(C-N stretching), 1460(N=N), 2962(CH_3), 1650(C=O) **^{1}HNMR (CDCl3) δ:** 14.87 (d,1H,NH),9.29 (s,1H,OH), 5.15(s,1H,CH), 2.24(s,3H) ,1.21(t,3H),4.21(qt.2H), 6.65,6.56,6.83,(m, 3H, Ar-H),7.21, 7.32, 6.89, 6.85 (m, 4H, Ar H), 3.81(s,3H CH_3) **^{13}CNMR (CDCl3) δ:** 168.5(C=O),127(Ar), 129(Ar), 56.2(CH), 25.2(CH_3), 55(CH_2) **m/z:** 445.13

ethyl-2-(((2-hydroxy-phenyl)(4-methoxy phenyl)methyl)carbamothioyl)diazenyl)-3-oxobutanoate (comp.code IVf) IR (KBr) cm^{-1}: 3445 (Ar-OH stretching), 3109 (NH stretching), 2856 (CH stretching), 1274 (C=S), 1166(C-N stretching), 1465(N=N), 2972(CH_3), 1656(C=O) **^{1}HNMR (CDCl3) δ:** 14.67 (d,1H,NH),9.65 (s,1H,OH), 5.11(s,1H,CH), 2.24(s,3H) ,1.21(t,3H),4.21(qt.2H), 7.27, 7.08, 6.56,6.83,(m, 4H, Ar-H),7.21, 7.32, 6.89, 6.85 (m, 4H, Ar H), 3.81(s,3H CH_3) **^{13}CNMR (CDCl3) δ:** 170.5(C=O),128(Ar), 129(Ar), 57(CH), 25.2(CH_3), 55(CH_2) **m/z:** 429.14

Conclusion

In this study, we have successfully synthesized new series of heterocyclic azo dyes compounds. The proper analysis of the synthesis of new azo dyes derivatives has been systematically evaluated by FT-IR, NMR, UV-Visible, Mass spectrometry.

References

1 Fabian J, Hartman H. Light absorption of organic colorants. New York: Springer±Verlag, 2008

2 Venkataraman K. The chemistry of synthetic dyes vols I± IV. London: Academic Press, 2005.

3 Zollinger H. Azo and diazo chemistry. New York: Interscience Publishers, 2011.

4 Gordon PF, Gregory PF. Organic chemistry in colour. New York: Springer±Verlag, 2017.

5 Pati S. The chemistry of the azo and azoxy groups. Part I. John Wiley, 2005

6 Towns A., Developments in azo disperse dyes derived from heterocyclic diazo components. Dyes and Pigments. (2009).

7 Hunger K., Industrial dyes: chemistry, properties, applications: John Wiley & Sons, (2007).

8 Abou-Yousef H., Khattab T.A., Youssef Y.A., Al-Balakocy N., Kamel S., Novel cellulose-based halochromic test strips for naked-eye detection of alkaline vapors and analytes. Talanta; (2017).

9 Rather L.J., Akhter S., Hassan Q.P., Mohammad F., Chemistry of plant dyes: Applications and environmental implications of dyeing processes. Current Environmental Engineering; (2017).

10 Khattab T.A., Rehan M., Aly S.A., Hamouda T., Haggag K.M., Klapötke T.M., Fabrication of PAN-TCF-hydrazone nanofibers by solution blowing spinning technique: Naked-eye colorimetric sensor. Journal of Environmental Chemical Engineering; (2017).

11 Murphree S.S., Heterocyclic dyes: Preparation, properties, and applications. Progress In Heterocyclic Chemistry: Elsevier; (2011).

12 Saini A., Christenson C., Khattab T., Wang R., Twieg R., Singer K., Threshold response using modulated continuous wave illumination for multilayer 3D optical data storage. Journal of Applied Physics; (2017)

13 Gregory P., Industrial applications of phthalocyanines. Journal of Porphyrins and Phthalocyanines; (2000).

14 Khattab T.A., Gaffer H.E., Synthesis and application of novel tricyanofuran hydrazone dyes as sensors for detection of microbes. Coloration Technology; (2016).

15 Luo S., Zhang E., Su Y., Cheng T., Shi C., A review of NIR dyes in cancer targeting and imaging. Biomaterials; (2011).
16 Khattab T.A., Tiu B.D.B., Adas S., Bunge S.D., Advincula R.C., pH triggered smart organogel from DCDHF-Hydrazone molecular switch. Dyes and Pigments; (2016).
17 Zollinger H., Color chemistry: syntheses, properties, and applications of organic dyes and pigments: John Wiley & Sons, (2003).
18 Abdelmoez S., El Azeem A., Rehab A., Nada A.A, Khattab T.A., Electrospun PDA-CA Nanofibers toward Hydrophobic Coatings. Zeitschrift für anorganische und allgemeine Chemie; (2016).
19 Centeno S.A., Buisan V.L., Ropret P., Raman study of synthetic organic pigments and dyes in early lithographic inks (1890–1920). Journal of Raman Spectroscopy; (2006).
20 Nowak M.T., Chen Q., Fluorescent inks for writing instruments using fluorescent dyes and white pigments. Google Patents; (2003).
21 Khattab T.A., Tiu B.D.B., Adas S., Bunge S.D., Advincula R.C., Solvatochromic, thermochromic and pH-sensory DCDHF-hydrazone molecular switch: response to alkaline analytes. RSC Advances; (2016).
22 Khattab T.A., Abdelmoez S., Klapötke T.M., Electrospun Nanofibers from a Tricyanofuran- Based Molecular Switch for Colorimetric Recognition of Ammonia Gas. Chemistry-A European Journal; (2016).
23 Khattab T.A., Novel solvatochromic and halochromic sulfahydrazone molecular switch. Journal of Molecular Structure; (2018).
24 Khattab T.A., Rehan M., Hamouda T., Smart textile framework: Photochromic and fluorescent cellulosic fabric printed by strontium aluminate pigment. Carbohydrate polymers; (2018).
25 Khattab T., Haggag K.M., Synthesis and spectral properties of symmetrical and asymmetrical 3-cyano-1, 5-diarylformazan dyestuffs for dyeing polyester fabrics. Egyptian Journal of Chemistry; (2017).
26 Khattab T.A., Haggag K.M., Elnagdi M.H., Abdelrahman A.A., Abdelmoez Aly S., Microwave- Assisted Synthesis of Arylazoaminopyrazoles as Disperse Dyes for Textile Printing. Zeitschrift für anorganische und allgemeine Chemie; (2016).
27 Khattab T.A., Gaffer H.E., Aly S.A., Klapötke T.M., Synthesis, Solvatochromism, Antibacterial Activity and Dyeing Performance of Tricyanofuran-Hydrazone Analogues. Chemistry Select; (2016).

Access this Article in Online

Website: www.ijcrcps.com

Subject: Chemistry

Quick Response Code

DOI: 10.22192/ijcrcps.2022.09.02.003

How to cite this article: Alka Pradhan, Pratibha Dwivedi. (2022). Synthesis and Characterization of New Heterocyclic Azo dyes. Int. J. Curr. Res. Chem. Pharm. Sci. 9(2): 16-19.
DOI: http://dx.doi.org/10.22192/ijcrcps.2022.09.02.003

