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Synthesis & Characterization Of Azo Compounds Containing Disubstituted Phenolic Moities

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Abstract

Dyes are organic compounds which gives colour to a substrate. Azo dyes are the largest group of colorants used in industry. Due to their ability to absorb visible light & ease of synthesis dyes have wider industrial application and use such as textiles, fiber, leather, print and painting, also in agriculture, cosmetics & in laboratories. In this research eight azo compounds are prepared by diazo coupling. The diazotizonium salt is formed when $NaNO_2$ & concentrated HCl is treated with substituted aromatic amines viz. Aniline, Anthranilic acid, α-Naphathalamine, Benzidene, o-Nitroaniline, p-nitroaniline, sulphanilic acid and p-toulidine. The formation of novel dye took place with a sufficient yield with subsequent coupling of 2,4-dimethylphenol. The structures of prepared dyes are confirmed by Fourier transform Infrared & proton nuclear magnetic resonance spectroscopy. Perkin Elmer spectrum used for IR spectra & Bruker Avance 400MHz NMR spectrometer for NMR spectra. The IR spectra showed 3000-3500cm^{-1} streching for hydroxyl group & 1405-1550cm^{-1} streching for –N=N- bond and mass of azo compound found to be m/z 304(M+).

Keywords* : *Azo Dyes, diazo-coupling, 2,4-dimethylphenol

1. Introduction

Most of the azo dye are colourful itself due to presence of -N=N- azo group, the aromatic azo groups are highly coloured & are often used as dyes.[1] Azo dyes are the largest group of colourant with the variety of colours, so that it is largely used in textile, dyeing & paper printing.[2] In chemical industry aromatic azo compounds is used as main component for food additives, indicators, therapeutic agents, in dyes & pigments.[3] Azo dyes, shows wide application in many fields like dyeing textile fibres, biomedical studies, advanced applications in organic synthesis & high technology areas like lasers, liquid crystalline displays, electro-optical devices & ink-jet printers.[4-5] Some azo dye possess medicinal properties like antiseptic, antiprotzoal, antifungal, antibacterial and dyes antipyretic properties.[6-9]

Azo dyes are the largest & most important class of organic dyestuffs. These are the compounds consisting of a -N=N- linkage connected to phenyl ring[10]. In this compounds, -N=N- bond acts as Chromophore. These compounds are derived by coupling Ar-N=N- salt with C_6H_5OH or amino group. The process of conversion of primary aromatic amino compounds into a diazonium salt is known as diazotization.[11] The process is carried out in presence of acid & at a very low temperature. Aromatic azo groups are formed by a coupled reaction between a Ar-N=N- salt & a coupling agent.[12]

2. Objectives:

1. To synthesis azo dyes from 2,4-dimethylphenol from substituted phenyl amines .
2. To study the spectral investigation of azo dyes synthesized from 2,4-dimethylphenol from aromatic amines .

3. Materials & Method:

Materials :

The reagents utilized in this research are of synthetic grade. The structural composition of prepared dyes identified through H^1-NMR and IR spectra. IR spectra found out with the help of Perkin Elmer spectrum FTIR instrument, the H^1-NMR spectra estimate by Brucer Avance 400 MHz NMR

spectrometer. The crude products were recrystallize by using ethanol as solvent.

General procedure for synthesis of azo dyes containing 2,4-dimethylphenol moities :

Method: Aromatic amine like o-nitroaniline (0.01mole) was mixed with concentrated HCl (2.5 ml). The resultant suspension crushed into ice & 2.5 ml (4N) cold $NaNO_2$ was mixed with constant stirring. The temperature of reaction was maintained upto 0-5^{0}C, diazotization was carried out over 15-20 mints. The diazonium salt prepared above is mixed dropwise to the Disubstituted Phenol by maintain the P^H above 7. Stired the reaction mixture & maintained the 5-10^{0}C. The coloured products obtained is filtered & dried & recrystallized by using ethanol. The aromatic amines used in the present study are Aniline, Anthranilic acid, o-Nitroaniline, m-Nitroaniline, p-Toulidine, Sulphanilic acid, α-Naphthalamine & Benzedine.

4.Result :

COMPOUND 1a:
4-Hydroxy-3-Methyl-5-[(Benzenyl-1-diaze] toluene, ($C_{14}H_{14}N_2O$)
Molecular weight-226, Colour- Brown, Yield - 64%, IR (cm^{-1}): 3476 (Phenolic -OH) , 1411 (νCH3), 1502 (ν-N=N-), 1243(νC-N) 2922(aromatic νC-H), 1600(νC=C),
H^{1}NMR(∂ in ppm) : 2.5109(s H), 12.4838 (m H of phenolic OH), 7.7469 (s, 1H Ar-H), 7.5333 (d 1H Ar H), 7,2422 (d 1H Ar H) 6.6820 (s 3H of CH_3)

COMPOUND 1b:
4-Hydroxy-3-Methyl[2nitro phenyl) diaze] toluene, ($C_{14}H_{13}N_3O_3$)
Molecular weight- 271, Colour- Dark Red, Yield - 92 %, IR (cm^{-1}) : 3416(νOH), 1432(νCH3), 1585 (ν-N=N-), 1243 (νC-N), aromatic(νC-H)2922, 1600 (νC=C), 1504 (νNO_2)
H^{1}NMR(∂ in ppm) : 11.3198 (s 1H of phenolic OH) , 8.1439 (d 1H of Ar H), 8.0573 (d 1H of Ar H), 7.8862(t 1H Ar H), 7.7627 (t 1H Ar H)

COMPOUND 1c:
4-Hydroxy-3-Methyl-5-[(4-methyl phenyl)diaze]toluene, ($C_{15}H_{16}N_2O$)
Molecular weight- 270, Colour-Orange, Yield - 78 %,
IR (cm^{-1}) : 3414(νOH), 1424(-νCH_3), 1500 (νN=N), 1268(νC-N), 2915 (νC-H aromatic), 1600 (νC= C)
H^{1}NMR(∂ in ppm) : 11.6633 (s 1H of OH), 7.9091, 7.8894, 7.3837 (d 4H of unsy CH), 7.4729, 7.4033, 7.1452(s H of CH3)

COMPOUND 1d:
4-Hydroxy-3-Methyl-5-[(Naphthalene-1-)diazeyl]toluene, ($C_{18}H_{16}N_2O$)
Molecular weight- 276, Colour- Chochlate, Yield - 80 %,
IR (cm^{-1}) : 3413(νOH), 1479(νCH_3), -1507(ν-N=N-), 1271(ν-C-N-), 1612 (νC=C), 2915 (νC - H aromatic)
H^{1}NMR(∂ in ppm) : 11.0580 (s Phenolic-OH), 7.4797(s H of CH_3), 7.2327(s H of CH_3), 7.4797(s H of CH_3), 8.8441(t 1H of unsy CH), 7.8837(t 1H of unsy CH), 8.4933,8.4749, 8.3757, 8.3551 (d1H of CH)

COMPOUND 1e: O
4-Hydroxy-3-Methyl-5-[(4-nitrophenyl) diazeyl] toluene, ($C_{14}H_{13}N_3$)
Molecular weight- 383, Colour-Orange Red, Yield - 82 %,
IR (cm^{-1}) : 3413 (νOH), 1406 (νCH_3), 1523 (νN=N), 1280(νC-N), 1611(νC=C), 1523 (νNO2), 2915 (νC-H aromatic)
H^{1}NMR(∂ in ppm) : 11.4294 (s H of OH), 8.7023(s 1H of SO_3H), 8.6814 (d H of CH), 8.1529,8.1113(d H of CH), 7.6553, 7.1964(s H of CH_3)

COMPOUND 1f:
4-Hydroxy-3-Methyl-5-[(Benzidiene) diazeyl]toluene, ($C_{20}H_{19}N_3O$)
Molecular weight- 317, Colour-**W**hite, Yield - 45 %,
IR (cm^{-1}) : 1447 (νCH3), 3412 (νOHphenolic), 2931 (νC-Haromatic), 1677 (νC=C), 1263 (νC-N), 1204,(νN=N) 1593, -νNH2 3379
H^{1}NMR(∂ in ppm) : 8.0205) , 8.0022(s of OH), 7.5737, 7.3212 (t H of CH), 7.4419(s H CH_3), 3.3859(s H)

COMPOUND 1g:
4-Hydroxy-3-Methyl-5-[(4-sulphonyl diazeyl]toluene, ($C_{14}H_{14}N_2O_4S$)
, Molecular weight- 306, Colour-**Red**, Yield - 96 %,
IR (cm^{-1}) : 3411 (νOH), 1418(νCH_3), 1579(νN=N), 1269(νC-N), 1220, 1621 (νC=C), 2971(νC-Haromatic), 1130, 1188(νSO_3H)
H^{1}NMR(∂ in ppm) : 11.5954(s of OH), 7.6321 7.3994 (t H of CH), 7.7419(s H CH_3), 4.3859(s H)

COMPOUND 1h :
4-Hydroxy-3-Methyl-5-[(4-benzoic acid)diazeyl]toluene, ($C_{15}H_{14}N_2O_3$)
Molecular weight- 278, Colour - Bargandy, Yield - 72 %,
IR (cm^{-1}) : 3413 (ν OH), 1440((ν CH_3), 1590 (ν N=N), 1295 (ν C-N), 1606(ν C=C), 2914(ν C-H), 1668(ν COOH)
H^{1}NMR(∂ in ppm) : 11.1681(s H of OH), 7.8062(s 1H of COOH), 7.6765,7.6637(t H CH), 7.0924,7.4805(s H of CH_3)

5. Discussions :

We report here the synthesis of some azo dyes from different eight aromatic amines by reacting with HCl & cold $NaNO_2$ solution by coupling with 2,4-dimethyl phenol to give corresponding azo dyes (as mentioned above in the table) with a good yield. The IR spectra of synthesized compounds showed the absorption bands at 3400-3500 cm^{-1} & 1500-1590 cm^{-1} for phenolic OH & azo group respectively. Also the absorption band for C=C at 1600-1605cm^{-1} , aromatic C-H is at 2915 cm^{-1}, NO_2 is at 1523 cm^{-1}. Furthermore the ^{1}H-NMR spectrum –OH proton is at ∂ 8.0811- 11.500 ppm. Mass of the new compound i.e. 1-g is found to be m/z 304.

6. Conclusion:

In this study, a series of eight novel azo dye were synthesized & characterized by FT-IR, H-NMR & MASS. All of the dyes showed a variety in colours & yield is sufficient. It can be concluded that eight azo dyes were successfully synthesized from 2,4-dimethylphenol by diazo coupling reaction.

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