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Eight Newazo Compounds Synthesis and Spectral and Antimicrobial Analysis

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Abstract: ***Eight new azo compounds containing o-methoxy phenolic moiety were synthesized and characterized by IR and ^{1}H-NMR spectroscopy. A clean preparation of azo compounds using o-methoxy phenol and eight different amines by azo coupling reaction was described. Further, the compounds were evaluated for antibacterial activity against Staphylococcus aureus, Escherichia coli, Pseudomonas aeruginosa and salmonella typhibythe disk diffusion method. The screening data revealed that the studied azo compounds exhibited antimicrobial activity against most of the tested species, presenting a particularinterest against Staphylococcus aureus and Escheria coli.***

Keywords: -o-methoxy phenol,amines, antibacterial activity, azo compounds

1. Introduction

Azo dyes are designed to resist chemical and microbial attacks and to be stable in the light and during washing. Azo dyes are the largest and the most versatile class of dyes. They possess intense bright colors, in particular orange, red and yellow. In addition, azo dyes exhibit a variety of interesting biological activities. Medical importance of these compounds is well known for their antibiotic, antifungal and anti-HIV properties. On the other hand, they bring a certain danger for health and environment because of cancer and mutagenicity. Azo dyes in purified form are mutagenic or carcinogenic, except for some azo dyes, leads to formation of aromatic amines and several aromatic amines are known mutagens and carcinogens to human beings.

They are also known to be involved in a number of biological OPEN ACCESS Molecules reactions such as inhibition of DNA, RNA and protein synthesis, carcinogenesis and nitrogen fixation. In a broader sense, the azo dyes constitute the largest diverse group of all the synthetic colorants.

Many azo-dyes, such as methyl red, methyl orange, and congo red, can be used as acid base indicators due to their ability to function as weak acids or bases. Color changes are caused by changes in extent of delocalization of electrons - More delocalization shifts the absorption max to longer wavelengths and makes the light absorbed redder, while less delocalization shifts the absorption max to shorter wavelengths. Another positive property of azo dyes is their antimicrobial activity. Photochemical reactions may be important as dyes are good absorbers of solar energy. However, little information is available on this. The present study is focused on the possibility of developing new eco-friendly azodyes with good coloristic and application properties, and exhibiting biological activity.

2. Materials and Method

Most of the chemicals used in this study were of analytical grade and were obtained fromAldrich Chemical Co.The IR spectra were recorded on Perkin-Elmer spectrum. The ^{1}H-NMR spectra were recorded as solution in DMSO−d6 using a BRUKERAVANCE DRX-400 spectrometer with tetramethylsilane (TMS) as internal standard.The crude products were recrystallized from ethanol.

GENERAL PROCEDURE

In a large test tube aromatic amines were mixed with 2.5 ml of concentrated HCl. Keep this solution at 0 °C – 5 °C using an ice-water bath. Add 2.5 ml (4N) sodium nitrite solution to the first test tube containing the aromatic amine solution. Use a Pasteurpipette to slowly add this suspension to the second test tube containing the HCl solution, with constant stirring using a glass rod. Make sure to maintain the temperature below 10°C. Keep the diazonium salt solution cold and covered to minimize evaporation of the solution. To this diazonium salt solution alkaline solution of m-methoxy phenol was added drop wise. This reaction mixture was stirred for 15-20 minutes maintaining the temperature 5-10^o C. The colored product obtained was filtered and washed with water and recrystallized by ethanol as solvent.

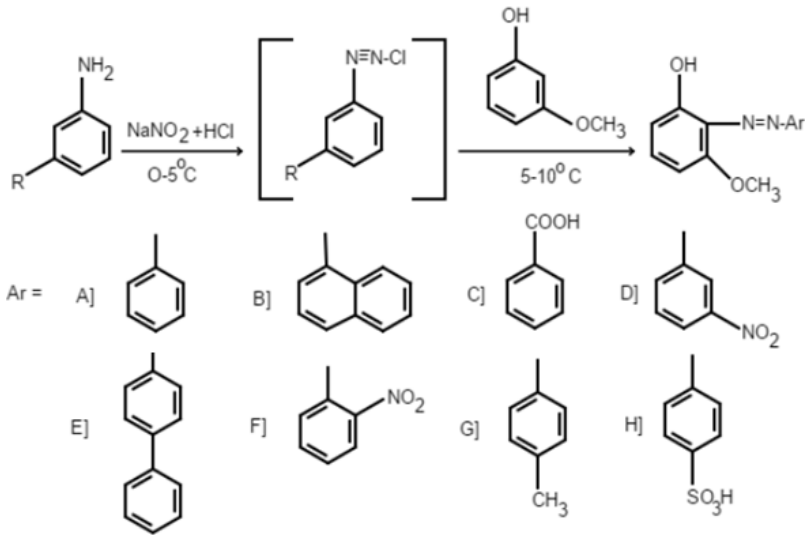

Figure 1: General reaction for synthesis of compounds IIIA-IIIH

3. Results and Conclusion

The results of screening antimicrobial activity are given in Table 1. The di-azocompounds IIIA-IIIH were subjected to antibacterial activity screening against two gram-positivebacteria (*Staphylococcus aureus* and *Salmonella typhi*) and two gram-negative bacteria (*Pseudomonas aeruginosa, Escherichia coli*)employing the disk diffusion technique.

Table: Antimicrobial properties of the synthesized azo compounds zone of inhibition (mm)(1) E. coli. (2) S. aureus (3) Salmonella typhi (4) Pseudomonas Aeruginosa

Compounds	1	2	3	4
IIIA	8	11	9	-
IIIB	13	10	10	9
IIIC	6	8	11	6
IIID	14	13	11	15
IIIE	9	10	8	10
IIIF	12	10	9	-
IIIG	9	8	10	7
IIIH	11	12	11	10

4. Results

Spectral data for synthesized compounds

IIIA] 3-methoxy-2-[(E)-2-phenyldiazen-1-yl]phenol
^{1}HNMR-7.8(s,1H,OH), 3.8(m,3H,OCH_3), 7.5(s,2H,Ar-H), 6.6(s,1H,Ar-H), 7.3(s,2H,Ar-H), 6.5(s,1H,Ar-H), 7.02(s,1H,Ar-H)IR-3003(OH), 1444(N=N), 1166(C-O,OCH_3), 1498(C=C), 2939(C-H,Ar), 2877(C-H,OCH_3)

IIIB] 3-methoxy-2-[(E)-2-(naphthalene-1-yl)diazen-1-yl]phenol
^{1}HNMR-9.9(s,1H,OH), 3.9(m,3H,OCH_3), 6.6(s,1H,Ar-H), 7.5(s,1H,Ar-H), 7.7(s,1H,Ar-H), 7.4(s,1H,Ar-H), 7.8(s,1H,Ar-H), 7.6(m,1H,Ar-H) IR-3331(OH), 1454(N=N), 1149(C-O,OCH_3), 3037(C-H,Ar), 1573(C=C,Ar), 2887(C-H,OCH_3)

IIIC] 4-[(E)-2-(2-hydroxy-6-methoxyphenyl)diazen-1-yl]benzoic acid
^{1}HNMR-8.4(d,1H,OH), 3.8(s,3H,OCH_3), 8.1(m,2H,Ar-H), 7.5(s,2H,Ar-H), 6.5(m,1H,Ar-H), 6.4(s,1H,Ar-H), 7.4(s,1H,Ar-H) IR-3001(OH), 1504(N=N), 1165(C-O,CH_3), 2875(C-H,OCH_3), 1562(C=C,Ar), 2943(C-H,Ar), 1693(C=O,COOH)

IIID] 3-methoxy-2-[(E)-2-(3-nitrophenyl)diazen-1-yl]phenol
^{1}HNMR-10.1(s,1H,OH), 3.8(m,3H.OCH_3), 7.4(s,1H,Ar-H), 6.5(d,1H,Ar-H), 8.4(d,1H,Ar-H), 8.1(s,1H,Ar-H), 7.4(s,1H,Ar-H), 7.3(m,1H,Ar-H) IR-3223(OH), 1454(N=N), 2887(C-H,OCH_3), 3088(C-H,Ar), 1562(C=C,Ar), 1112(C-O,OCH_3), 1409(N-O,NO_2)

IIIE] 2-[(E)-2{[1,1'-biphenyl]-4-yl}diazen-1-yl]-3-methoxyphenol
^{1}HNMR-9.8(s,1H,OH), 3.8(m,3H,OCH_3), 6.5(s,1H,Ar-H), 7.4(s,1H,Ar-H), 7.5(d,2H,Ar-H), 7.6(m,2H,Ar-H), 7.7(t,2H,Ar-H) IR-3001(OH), 1492(N=N), 2885(C-H,OCH_3), 2939(C-H,Ar), 1543(C=C,Ar), 1149(C-O,OCH_3)

IIIF]3-methoxy-2-[(E)-2-(2 nitrophenyl)diazen-1-yl]phenol
^{1}HNMR-8.2(s,1H,OH), 3.8(m,3H,OCH_3), 7.5(s,1H,Ar-H), 6.4(s,1H,Ar-H), 8.03(s,1H,Ar-H), 7.4(d,2H,Ar-H) IR-3489(OH), 3076(C-H,Ar), 1502(C=C,Ar), 2895(C-H,OCH_3), 1037(C-O,OCH_3),1454(N=N), 1562(NO_2)

IIIG] 3-methoxy-2-[(E)-2-(4-methylphenyl)diazen-1-yl]phenol
^{1}HNMR-8.7(s,1H,OH), 3.9(s,3H,OCH_3), 6.9(s,1H,Ar-H), 7.3(s,1H,Ar-H), 6.4(d,1H,Ar-H), 7.2(d,2H,Ar-H), 7.1(d,2H,Ar-H), 2.1(m,3H,CH_3) IR-3028(OH), 2943(C-H,Ar), 1614(C=C,Ar), 2885(C-H,OCH_3), 1008(C-O,OCH_3), 1494(N=N), 1379(C-H,CH_3)

IIIH] 4-[(E)-2-(2-hydroxy-6-methoxyphenyl)diazen-1-yl]benzene-1-sulfonic acid
^{1}HNMR-7.9(s,1H,OH), 3.9(s,3H,OCH_3), 6.4(s,1H,Ar-H), 7.6(d,1H.Ar-H), 7.4(d,2H,Ar-H),7.8(m,2H,Ar-H) IR-3165(OH), 3045(C-H,Ar), 2893(C-H,OCH_3),

1531(C=C,Ar), 1124(C-O,OCH_3), 1454(N=N), 686(S-O), 1311(S=O,SO_3H)

5. Conclusion

The azo dyes were successfully synthesized by standard methods of diazotization and azo-coupling. The results on the elemental analysis and spectral studies of each dye were consistent and hence confirmed the predicted structure. The present study of the prepared acid azo dyes showed wide range of shades. They showed good dyeing performance.

6. Acknowledgment

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