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Synthesis and Characterization of Azo Compounds Containing O-Cresol and Beta-Naphthol Moieties and Study of Antimicrobial Activity

KEYWORDS: Azo compounds, Antimicrobial activity, IR, NMR.

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ABSTRACT *Noval azo dyes have been synthesized using substituted aromatic amines and phenolic compounds by using simple diazotization reaction. The resulting dyes were characterized by spectral techniques i.e, IR, H1-NMR spectroscopy. The newly synthesized azo compounds were screened for their antimicrobial activities against six different bacterial species and these dyes were found to show excellent antimicrobial activity.*

INTRODUCTION:
Azo compounds are the oldest and largest class of industrial synthetic organic dyes due to their versatile application in various fields[1-2].Dyes used before the nineteenth century were either of vegetables or animal origin and belonged to various chemical types such as flavoniods, anthraquinones and indigoids[3]. Azo dyes have been most widely used in dyeing textile fibres, biomedical studies, advanced application in organic synthesis and high technology areas like lasers, liquid crystalline displays, electro-optical devices and ink-jet printers[4]. There are about 3000 azo dyes currently in use all over the world.

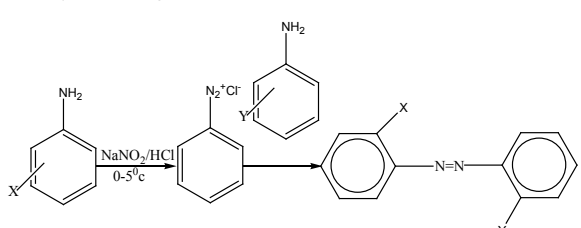
The great majority of them are mono azo compounds, which have the common structure unit of the azochromophore N=N linking two aromatic systems. The textile industry is the largest consumer of dye stuffs[5]. They have some variety of interesting biological activities[6]including antimicrobial activity, antibacterial[7] and pesticidal activities[8]. The azo dyes posses antiseptic and antiprotozoal properties and also promote wound healing.Azo compounds have received much attention due to their versatile use in many practical applications such as coloring, fiber, photoelectronic applications, printing systems, optical storage technology and in various analytical techniques[9].

Traditionally, azo dyes are the most important class of commercial dyes occupying more than half of the dye chemistry which contain phenols as intermediates[10-16].Azo dyes have been reported to be good antibacterial agents[17]. Our work is focused on the synthesis and characterization of azo dyes containing o-cresol, β-napthol moieties and study of antimicrobial activity. We have synthesized azo dyes using substituted aromatic amines and phenolic compounds such as o-cresol, beta naphthol and resorcinol following simple diazotisation method. The dyes synthesized were characterized by IR and NMR spectroscopy these azo dyes have shown antibacterial property.

EXPERIMENTAL PROCEDURE:
All the chemicals and solvents were obtained from Merck Company Ltd (AR-Grade). IR-spectra were recorded on a Perkin Elmer instrument. H^1-NMR spectra of the synthesized compounds were recorded on as Aspeet spectrometer using DMSO solvent and TMS as the internal standard using ethanol. The antimicrobial activity of azo dyes was analyzed in micro labs Vellore.

GENERAL PROCEDURE FOR SYNTHESIS OF AZO COMPOUNDS:
Substituted aromatic amine (15 mmol) is treated with 5ml of con HCl and 5ml cold solution of sodium nitrite was added with constant stirring. The temperature of the reaction was maintained upto 0-5^{0}C. Diazonium salt solution prepared was added drop wise to substituted phenolic compounds (15mmol). The reaction mixture stirred for 10-30 minutes maintaining the temperature 5-10^{0}C. The colored product obtained is filtered and washed with water. The product synthesized was recrystallized using ethanol.

RESULTS AND DISCUSSION
The azo compounds with various substituents were synthesized by simple diazotization reaction of various substituted aromatic amines followed by coupling reaction with substituted phenolic compounds (Scheme-1.Sulphanilicacid, m-nitroaniline and m-chloroaniline were diazotized and coupled with phenolic compounds such as o-cresol, beta-naphthol and resorcinol. The product azo dye obtained was recrystallised using ethanol. The substituted aromatic amines and phenolic compounds used for the synthesis of azo dyes were given in Table-1.



Scheme-1
TABLE-1 PREPARATIVE DETAILS OF AZO DYE

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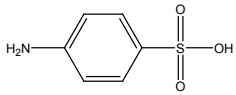
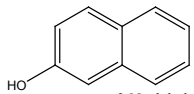
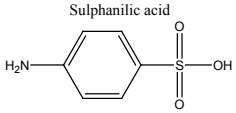
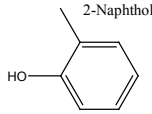
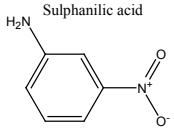
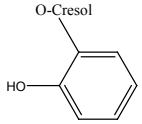
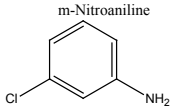
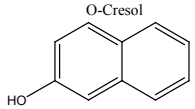
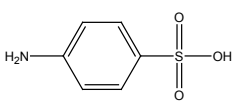
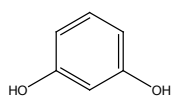
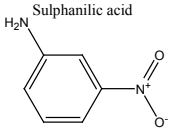
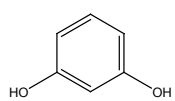
S.NO	Dyes prepared	Aromatic amines	Phenolic compound
1.	A	 Sulphanilic acid	 2-Naphthol
2.	B	 Sulphanilic acid	 O-Cresol
3.	C	 m-Nitroaniline	 O-Cresol
4.	D	 m-Chloroaniline	 2-Naphthol
5.	E	 Sulphanilic acid	 Resorcinol
6.	F	 m-Nitroaniline	 Resorcinol

The azo dyes synthesized were characterized by IR and NMR spectroscopic methods. IR and H^1-NMR spectra showedthe expected signals which correspond to various groups present in each compounds. The IR absorption bands were determined in KBr disk. The IR absorption band of azo group N=N is located at 1400-1600cm^{-1}. The O-H stretching frequency is expected at 3400-3600$cm^{-1}$and the sulphonyl stretching frequency range is 1160-1180cm^{-1}. The C=C (aromatic) stretching frequency at 1500-1700cm^{-1} and the C-H of CH_3 frequency is at 2900-3200cm^{-1} . The IR spectra of the azo dyes synthesized showed the characteristic N=N stretching band at 1500 cm^{-1}, sulphonyl stretching band at 1163cm^{-1} and O-H stretching band at 3400cm^{-1}. The results of IR spectral studies are summarized in Table-2.

TABLE-2 RESULTS OF IR SPECTROSCOPIC STUDY

COM-POUND	OH Cm^{-1}	N=N Cm^{-1}	SO_3H Cm^{-1}	C=C Cm^{-1}	C-H of CH_3 Cm^{-1}
A	3470.77	1500.78	1163.97	1586.34	-
B	3467.33	1466.13	1172.71	1593.98	2956.56
C	3422.79	1500	-	1550	2951.98
D	3432.15	1402.45	-	1626.89	-
E	3492.69	1453.36	1163.97	1586.37	-
F	3472.56	1500	-	1560	-

The H^1NMR spectra of the newly synthesized dyes showed the expected signal which corresponds to various groups present in each compound.

NMR SPECTRA DETAILS OF THE AZO DYES ARE GIVEN BELOW

A. 2.5(s 3H of CH_3), 6.9(m,1H of Ar-H), 7.0(m,1H of Ar-H), 7.2(m,1H 0f Ar-H), 7.7(s,1H of Ar-OH), 7.8(m,2H of Ar-H), 8.0(m,2H of Ar-H), 9.9(m,1H of SO_3H)

B. 2.47(s,3H of CH_3), 6.95(m,1H of Ar-H), 7.2(d,1H of Ar-H), 7.6(m,1H of Ar-H), 7.7(s,1H of Ar-OH),7.75(s,2H of Ar-H), 7.8(s,2H of Ar-H), 10.3(m,1H of SO_3H)

C. 2.3(s,3 H of C-H3), 6.5(m,1H of Ar-H), 7.0 (m,1 H of Ar-H) 7.5 (m,1H of Ar-H) 7.7 (m,1H of Ar-H),7.9 (m,1H of Ar-H), 8.2 (m,1H of Ar-H)), 8.5 (m,1H of Ar-H)), 8.7 (m,1H of Ar-OH).

D. 6.5(m,1H of Ar-H), 7.1(m,1H of Ar-H), 7.2(t,1H of Ar-H), 7.4(t,1H of Ar-H), 7.7(d,1H of Ar-H), 7.8(t,2H of Ar-H), 9.8(S,1H of Ar-OH)

E. 7.8(m,2H of Ar-H), 8.0(m,2H of Ar-H), 9.8(m,1H of SO_3H), 6.29(m.1H of Ar-H), 7.92(m,1H of Ar-H),7.29(s,1H of Ar-H), 8.9(m,1H of Ar-OH)

F. 7.9 (m,1H of Ar-H), 8.2 (m,1H of Ar-H)), 8.5 (m,1H of Ar-H)), 8.7 (m,1H of Ar-OH).7.29(m.1H of Ar-H), 7.92(s,1H of Ar-H),7.29(m,1H of Ar-H), 8.9(m,1H of Ar-OH)

STUDY OF ANTIMICROBIAL ACTIVITY OF AZO DYES

The azo compounds synthesized were tested against antimicrobial activity. The three azo dyes were further used individually to analyze its antimicrobial activity against six human pathogens namely Escherichia coli, Bacillus cereus, Bacillus substilis, Staphylococcus aureus, Pseudomona aeruginosa and Salmonella typhi. Antimicrobial analysis of the azo dyes A, B and C were done using standard agar-well diffusion methods. Antimicrobial activity was evaluated by measuring the diameters of the zone of inhibition in mm against the test microorganisms. The antibacterial activity of the synthesized azo dyes was compared with ciproflaxin.

TABLE-3 RESULTS OF ANTIMICROBIAL STUDY OF AZO DYES

S.NO	Name of the organism	Control (mm)	A (mm)	B (mm)	C (mm)	Anti-biotic disc (mm)
1.	Escherichia coli	-	10	14	19	18
2.	Bacillus cereus	-	10	17	21	13
3.	Bacillus substilis	-	11	17	20	18
4.	Staphylococcus aureus	-	15	18	20	25
5.	Pseudomonas aeruginosa	-	12	14	18	14
6.	Salmonella typhi	-	13	16	18	26

Antimicrobial analysis was followed using standard agar well diffusion method to study the antimicrobial activity of compounds. Each bacterial isolate was suspended in Brain Heart Infusion (BHI) broth and diluted to approximately 10^5 colony forming unit (CFU) per mL. They were flood-inoculated onto the surface of BHI agar and then dried. Five-millimeter diameter wells were cut from the agar us-

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ing a sterile cork-borer and 30 µL (5µg compound in 500 µL DMSO) of the sample solution were poured into the wells. The plates were incubated for 18 h at 37°C for bacteria. Antimicrobial activity was evaluated by measuring the diameter of the zone of inhibition in mm against the test microorganisms. DMSO was used as solvent control. Ciprofloxacin was used as reference antibacterial agent. The tests were carried out in triplicates.

As we compared with ciproflaxacin the sample shows the zone of inhibition they are all antimicrobial active. The azo compounds synthesized showed excellent antimicrobial activity and among these three samples the azo dye C showed highest antimicrobial activity.

CONCLUSION

The azo compounds with various substituents have been synthesized by simple diazotization reaction of various substituted aromatic amines followed by coupling reaction with substituted phenolic compounds. Suphanilic acid, nitro-aniline and chloro-aniline were diazotized and coupled with o-cresol, beta-naphthol and resorcinol. The synthesized azo compounds have been characterized by IR and NMR spectroscopic methods. Antimicrobial analysis of synthesized dyes was carried out using standard agar-well diffusion method. The antimicrobial activity of the azo dyes was screened against six microorganisms viz Escherichia coli, Bacillus cereus, Bacillus substilis, Staphylococcus aureus, Pseudomonas aeruginosa and Salmonella typhi. Antimicrobial activity was evaluated by measuring the diameter of the zone of inhibition in mm against test microorganism. The azo compounds synthesized showed excellent antimicrobial activity as compared with ciproflaxin and among the three samples the azo dye C showed the highest antimicrobial activity.

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