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SYNTHESIS, SPECTRAL CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF SOME AZO DYES DERIVED FROM P-CRESOL.

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Abstract: A number of new azo dyes were synthesized by coupling of p-cresol with diazonium salts obtained from Aniline, o-Nitro aniline, p-Toluedine, α-Naphthylamine, Sulphanilic acid, m-Nitro aniline, Benzedine and Anthranilic acid. The structures of synthesized azo dyes have been confirmed by FT-IR and H^1 NMR spectral data. In addition, the antimicrobial activity of these dyes was examined by using disc diffusion method. The compounds analysed for its antibacterial action showed moderate to significant inhibitory effect at some specific concentrations against the tested pathogens.

Keywords: Azo dyes, p-cresol, antimicrobial activity.

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INTRODUCTION

Azo compounds in which two phenyl rings are separated with by an azo (-N=N-) bond are versatile Molecules and have received much attention in research areas both fundamental and application. The strong electronic absorption maximum can be made by ring substitution to fall anywhere from the ultraviolet to red visible regions, allowing chemical fine-tuning of color. This combined with the fact that these azo dyes are relatively strong and chemically stable, has prompted extensive study of dyes and colorants. Due to their ability to absorb visible light, and ease of synthesis, have been extensively used in the textile, fiber, leather, and paint, printing industries, biological-medical studies and advanced applications in organic synthesis form more than a century[1, 2]. Synthetic azo dyes produced in large quantities are generally considered as xenobiotic compounds which are very recalcitrant to biodegradation. Textile industry is a major consumer of these dyes. The industrial effluents containing azo dyes released to the eco system undergo reductive cleavage to form aromatic amines that have known mutagenic and/or carcinogenic properties. The usual synthesis of azo dyes involves diazotization of a primary aromatic amine, followed by coupling with nucleophiles.
Azo compounds are the most fundamental class of commercial dyes and are well colored that have been used as dyes and pigments[3, 4]. Azo compounds are known to be involved in a number of biological reactions such as inhibition of DNA, RNA and protein synthesis, carcinogenesis and nitrogen fixation[5, 6] also known for their use as antibacterial[7-12], antifungal anti neoplastics, antidiabetics, antiseptics, anticancer, anti-inflammatory and other useful chemotherapeutic agents[13-16].

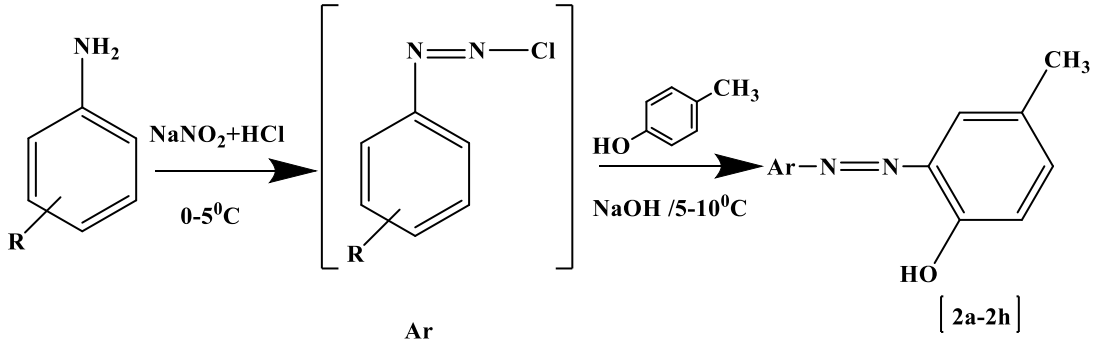
In the present research work p-cresol is coupled with diazonium salt of eight different aromatic amines VIZ: Aniline, o-Nitro aniline, p-Toluedine, α-Naphthylamine, Sulphanilic acid, m-Nitro aniline, Benzedine and Anthranilic acid.

Methods and Materials:

All the chemicals used in these experiments were of analytical grade. All the melting points were determined by open capillary method and are uncorrected. The products were confirmed by ^{1}H NMR (Burker avernce II 400 NMR Specrtometer) and IR technique (Shimatzu). The biological activity was evaluated against two kinds of bacteria gram positive and gram negative. The products were recrystallized by ethanol as solvent.

General procedure for synthesis of azo compounds.[17]
Substituted aromatic amines (0.01mole) were mixed with 2.5 ml conc. HCl and 2.5 ml (4N) cold solution of $NaNO_2$ was added with the stirring. The temperature of the reaction was maintained up to 0-5^0 C. Diazonium salt solution prepared above was added drop wise to the alkaline solution of p-cresol. The reaction mixture stirred for 10 – 20 minutes maintaining the temperature 5-10^0 C. The colored product so obtained is filtered washed with water and recrystallised from 80% ethanol. The general Scheme for the synthesis of azo dyes of p-cresol is shown in figure (I).

Figure I: General Scheme for the synthesis of azo dyes of p-cresol.



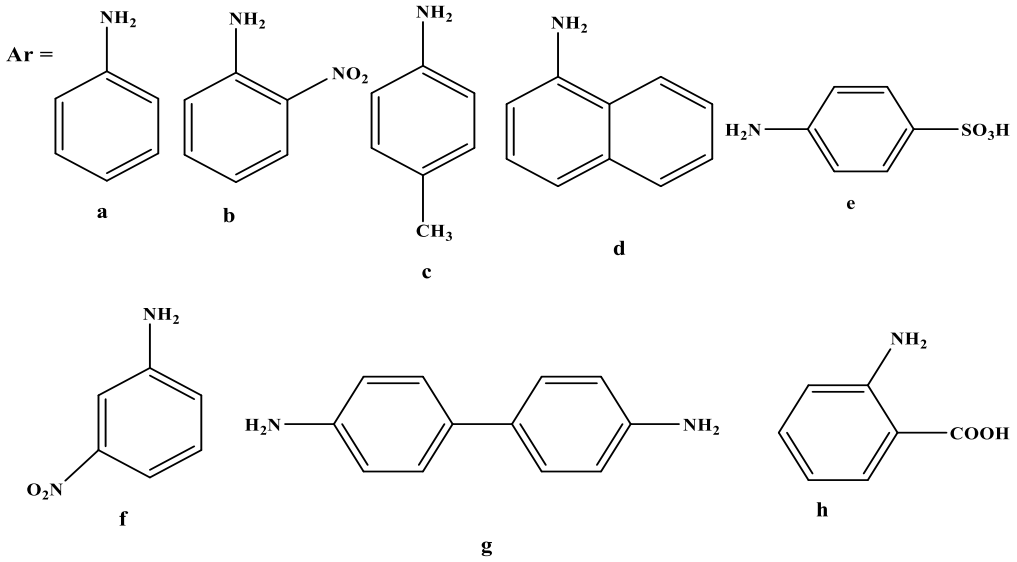


Table (I): The code, compound name, molecular formula, molecular weight, melting point and percentage yield of synthesized compounds of p-cresol.

Code	Structure	Molecular Formulae	Molecular Weight	Melting Point (^{0}C)	% Yield
2a	4-methyl-2-(phenyldiazenyl)phenol	$C_{13}H_{12}N_2O$	212	184	63
2b	4-methyl-2-((2-nitrophenyl)diazenyl)phenol	$C_{13}H_{11}N_3O_3$	257	179	58
2c	4-methyl-2-(*p*-tolyldiazenyl)phenol	$C_{14}H_{14}N_2O$	226	182	68
2d	4-methyl-2-(naphthalene-1-yldiazenyl)phenol	$C_{17}H_{14}N_2O$	262	186	67
2e	4-((2-hyderoxy-5-methylphenl)diazenyl)benzenesulphonic acid.	$C_{13}H_{12}N_2O_4S$	292	172	70
2f	4-methyl-2-((3-nitrophenyl)diazenyl)phenol	$C_{13}H_{11}N_3O_3$	257	181	65
2g	2-((4'-amino-[1,1'-biphenyl]-4-yl)diazenyl)-4-methylphenol	$C_{19}H_{17}N_3O$	303	174	72
2h	2-((2-hydroxy-5-methylphenyl)diazenyl)benzoic acid.	$C_{14}H_{12}N_2O_3$	256	163	60

Antimicrobial Activity:

The newly synthesized azo compounds 2a-2h were analyzed for their antimicrobial activity against four gram positive and gram negative bacteria viz. *Escherichia coli, Staphylococcus aureus, Pseudomanas aeroginosa and Salmonella typhi* by using agar well diffusion method[18]. These compounds were mixed in Ethanol to form the solution of concentration 1mg/ml. sterile disc were dipped in the solutions, dried it and placed on the nutrient agar medium spreaded with the bacteria. The plates were further incubated for 24 to 48 hours at 37^0 C and the diameter of zones of inhibition was measured in millimeter.

Result and Discussion:

The azo dyes synthesized were characterized by IR and NMR spectroscopic methods. IR and ^{1}H-NMR spectra showed the expected signals which correspond to various groups present in each compounds. The IR and ^{1}H-NMR spectral values for different synthesis dyes are shown in table II.

Table (II): FTIR AND ^{1}H NMR data of azo compounds of p-cresol.

Compound	Spectra	Spectroscopic Data
SDB 2a	IR (KBr. cm^{-1})	3468 (Phenolic –OH stretch), 1591 (C=C Aromatic), 1496 (N=N), 1278 (C-N Stretch), 3022 (C-H of CH_3).
	NMR (δ ppm)	3.18 (s 3H of $–CH_3$), 6.74 (s 1H of –OH), 6.99-7.75 (m 8H of Ar-H).
SDB 2b	IR (KBr. cm^{-1})	3510 (phenolic –OH stretch), 1583 (C=C Aromatic), 1501 (N=N), 1346 (C-N Stretch), 1246 (NO_2), 3026 (C-H of CH_3).
	NMR (δ ppm)	3.35 (s 3H of $–CH_3$), 11.13 (s 1H of –OH), 6.95-8.18 (m 8H of Ar-H).
SDB 2c	IR (KBr. cm^{-1})	3196 (phenolic –OH stretch), 1595(C=C Aromatic), 1496 (N=N), 1296 (C-N Stretch), 2918 (C-H of CH_3).
	NMR (δ ppm)	2.32 (s 3H of $–CH_3$), 2.40 (s 3H of $–CH_3$), 11.76 (s 1H of –OH), 6.89-8.16 (m 7H of Ar-H).
SDB 2d	IR (KBr. cm^{-1})	3375 (phenolic –OH stretch), 1593 (C=C Aromatic), 1494 (N=N), 1278 (C-N Stretch), 3055 (C-H of CH_3).
	NMR (δ ppm)	2.34 (s 3H of $–CH_3$), 11.09 (s 1H of –OH), 6.99-8.71 (m 10H of Ar-H).
SDB 2e	IR (KBr. cm^{-1})	3369 (Phenolic –OH stretch), 1591 (C=C Aromatic), 1496 (N=N), 1278 (C-N Stretch), 2916 (C-H of CH_3).
	NMR (δ ppm)	2.33 (s 3H of $–CH_3$), 11.70 (s 1H of –OH), 3.62 (s 1H of $–SO_3H$), 6.65-7.97 (m 7H of Ar-H).
SDB 2f	IR (KBr. cm^{-1})	3099 (phenolic –OH stretch), 1583 (C=C Aromatic), 1531 (N=N), 1253 (C-N Stretch), 1350 (NO_2), 2924 (C-H of CH_3).
	NMR (δ ppm)	2.07 (s 3H of $–CH_3$), 6.80 (s 1H of –OH), 6.82-8.52 (m 7H of Ar-H).
SDB 2g	IR (KBr. cm^{-1})	3365 (phenolic –OH stretch), 3030 (N-H Stretch), 1600 (C=C Aromatic), 1494 (N=N), 1247 (C-N Stretch), 2918 (C-H of CH_3).
	NMR (δ ppm)	2.27 (s 3H of $–CH_3$), 5.29 (s 1H of –OH), 3.36 (s 2H of $–NH_2$), 6.63-8.17 (m 11H of Ar-H).
SDB 2h	IR (KBr. cm^{-1})	3018 (phenolic –OH streach), 1593 (C=C Aromatic), 1496 (N=N), 1278 (C-N Streach), 1701 (C=O, Streach of COOH), 2914 (C-H of CH_3).
	NMR (δ ppm)	2.31 (s 3H of $–CH_3$), 7.5 (s 1H of –OH), 6.63-8.17 (m 7H of Ar-H), 8.26 (s 1H of –COOH).

Antimicrobial Activity:

A total eight azo compounds of p-cresol have been synthesized, recrystaliesed and used separately for its study of antimicrobial activity against four gram positive and gram negative bacteria viz. *Escherichia coli, Staphylococcus aureus, Pseudomanas aeroginosa and Salmonella typhi*. The data of antimicrobial activity of these newly synthesized azo dyes of p-cresol 2a-2h against four pathogens are presented in the tables 1-4.

Antibacterial properties of the synthesized azo compounds of p-cresol viz 2a – 2h [Zone of inhibition (mm)]

Table (1): Effect of azo compounds of p-cresol viz. 2a – 2h on the growth response of *Escherichia coli*.

Conc.(mg/ml)	2a	2b	2c	2d	2e	2f	2g	2h
0.5	NI	NI	I(10)	NI	NI	NI	I(10)	NI
1.0	I(10)	NI	I(10)	NI	NI	I(10)	NI	I(10)
1.5	NI	I(11)	NI	I(10)	NI	NI	I(10)	I(10)

2.0	I(12)	NI	NI	NI	NI	NI	I(10)	I(11)
2.5	I(10)	NI	I(10)	NI	NI	NI	NI	I(11)
3.0	I(10)	NI	I(11)	NI	I(11)	NI	NI	NI

I = Inhibition, values of inhibition are given in parenthesis, NI = No inhibition

Table (2): Effect of azo compounds of p-cresol viz. 2a – 2h on the growth response of *Salmonella typhi*.

Conc.(mg/ml)	2a	2b	2c	2d	2e	2f	2g	2h
0.5	I(11)	I10	I(10)	I(10)	I(10)	I(10)	I(10)	I(10)
1.0	I(10)	I(11)	I(10)	I(10)	I(12)	I(13)	I(10)	I(10)
1.5	I(11)	I(12)	I(10)	I(10)	I(11)	I(15)	I(10)	I(10)
2.0	I(11)	I(11)	I(10)	I(10)	I(16)	I(14)	I(10)	I(10)
2.5	I(10)	I(10)	I(11)	I(11)	I(12)	I(12)	I(10)	I(17)
3.0	I(13)	I(12)	I(12)	I(12)	I(12)	I(12)	I(14)	NI

I = Inhibition, values of inhibition are given in parenthesis, NI = No inhibition

Table (3): Effect of azo compounds of p-cresol viz. 2a – 2h on the growth response of *Pseudomonas aeroginosa*.

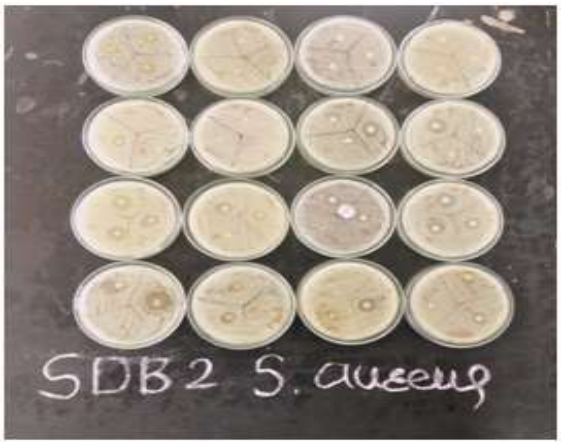
Conc.(mg/ml)	2a	2b	2c	2d	2e	2f	2g	2h
0.5	I(100	I(11)	I(10)	NI	I(10)	NI	I(10)	NI
1.0	I(10)	I(10)	I(10)	I(10)	I(10)	NI	I(10)	NI
1.5	I(10)	I(13)	I910)	I(12)	I(10)	NI	I(10)	NI
2.0	I(10)	I(10)	I(10)	I(10)	I(10)	I(10)	I(10)	I(10)
2.5	I(10)	I(10)	NI	I(10)	I(10)	NI	NI	I(10)
3.0	I(10)	I(10)	I(10)	I(10)	I(10)	I(10)	I(10)	NI

I = Inhibition, values of inhibition are given in parenthesis, NI = No inhibition

Table (4): Effect of azo compounds of p-cresol viz. 2a – 2h on the growth response of *Staphylococcus aureus*.

Conc.(mg/ml)	2a	2b	2c	2d	2e	2f	2g	2h
0.5	I(10)	I(10)	I(10)	I(10)	I(10)	I(13)	I(10)	I(10)
1.0	I(10)	NI	I(10)	I(10)	NI	I(10)	I(12)	I(10)
1.5	NI	NI	I(10)	I(19)	NI	I(11)	I(14)	I(10)
2.0	I(10)	I(10)	NI	I(10)	NI	I(10)	I(12)	I(11)
2.5	I(10)	NI	I(10)	I(10)	NI	NI	I(10)	I(10)
3.0	I(10)	NI	I(10)	NI	NI	I(15)	NI	NI

I = Inhibition, values of inhibition are given in parenthesis, NI = No inhibition



CONCLUSION

All the eight novel azo compounds 2a–2h containing p-cresol moiety were successfully synthesized in excellent yield and their structures are confirmed using elemental analysis, FTIR, 1HNMR & MASS spectroscopy. The results on antimicrobial activity reveal that all the eight newly synthesized compounds viz 2a–2h found to have outstanding antibacterial effect against *E.Coli*, *S. aureus, Pseudomonas aeroginosa,* and *Salmonella typhi* nearly at all the concentrations analysed. The results revealed, the broad spectrum potential of all the compounds in inhibiting the growth of human pathogens, and this finding enlighten the possible help in drug discovery.

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