WORLD JOURNAL OF PHARMACEUTICAL RESEARCH
SJIF Impact Factor 7.523
Volume 6, Issue 3, 1654-1660. Research Article ISSN 2277– 7105

SYNTHESIS OF SOME NOVEL HETEROCYCLIC AZO DYES FOR ACRIDINE DERIVATIVES AND EVALUATION OF THEIR ANTIBACTERIAL ACTIVITIES

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Article Received on
18 Jan. 2017,

Revised on 08 Feb. 2017,
Accepted on 28 Feb. 2017

DOI: 10.20959/wjpr20173-8074

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ABSTRACT

The novel mordent and disperse heterocyclic dyes were prepared by coupling of various diazo solution of aromatic amines with N-[{4-[2-(5-(4-chlorophenyl)-3-(4-methoxyphenyl)-4,5-dihydro-pyrazol-1-yl)-2-oxoethoxy]phenyl}methylene]substitutedaniline. The resultant mordent and disperse heterocyclic dyes were characterized by elemental analyses, IR and NMR spectral studies. The UV-visible spectral data have also been discussed in terms of structural property relationship. All the disperse azo dyes were applied on polyester textile fibers. The percentage dye bath exhaustion and fixation on the polyester fibers have been found to be very good. Moderate to very good light fastness and washing fastness properties were indicated by the dyied fabrics. Structures of synthesized compounds were confirmed by physical and spectral analysis. The compounds are evaluated for their antimicrobial activities.

KEYWORDS: Schiff bases, heterocyclic dyes, UV absorber, antimicrobial-screening.

INTRODUCTION

Synthetic dyes are extensively used in textile, paper and printing industries as well as in dyehouses.[1] The coloring process discharge huge quantities of dye effluents, which pollute localterrestrial habitat, aquatic bodies as well as rivers. Synthetic dyes are not easily amenable formicrobial attack as they contain substitution such as azo, nitro and sulpho groups.[2,3] Sulphonated azo dyes represent a large class ofdyes used in textile industries. These dyes may form sulphonated aromatic amines which are recognized as possiblehuman carcinogens.[4] Both sulfonated and unsulfoated aromatic amines formed during the reduction

of sulfonatedazo dyes are an important group of environmental pollutants that can potentially pass through biological treatmentsystem.[5] Azo dyes are usually designed to resist biodegradation under aerobic conditions, the recalcitrance of thesecompounds being attributed to the presence of sulfonate groups and azo bonds[6], hence the development of nongenotoxic dyes and investment in research to find effective treatments for effluents and drinking water is required, inorder to avoid environmental and human exposure to these compounds and prevent the deleterious effects they canhave on human and aquatic organisms.[7] For many years, the azo comounds have beenthe main class of dyes used in various applications such as textile fibers dyeing, colouring of different materials andadvanced organic synthesis. The synthesis and dyeing properties of azo compounds are described in many papers.[8-13] They are synthetic compounds and account for more than 50% of all the dyes produced annually, showing largestspectrum of colors.[14-16]

MATERIALS AND METHODS

1. Experimental

Melting points were taken in open capillary tube and were uncorrected. IR spectra were recorded on I.R. Spectrophotometer of Buck scientific Model No. 500 and instrument used for NMR Spectroscopy was Bruker Advance II 400 and DMSO used as internal standard. Solvent used were $CDCl_3$ and DMSO. Purity of the compounds were checked by TLC on silica- G plates. All the compounds were tested for their antibacterial and antifungal activities by broth dilution method

2. MATERIALS AND METHODS

2.1 Preparation of *N*-[{4-[2-(5-(4-chlorophenyl)-3-(4-methoxyphenyl)-4,5-dihydro-pyrazol -1-yl)-2-oxoethoxy]phenyl}methylene] substituted aniline (1a-j)

A mixture of 2-(4-{[(2-chlorophenyl)imino]methyl}phenoxy)acetohydrazide (0.1M), ethanol (25ml) and 3-(4-chlorophenyl)-1-(4-methoxyphenyl)prop-2-en-1-one(0.1M) with piperidine (1ml) was refluxed for 16 hours. The resulting mixture was concentrated, cooled and poured into cold water containing 6 to 8 drops of HCl, when orange colored product separated. It was filtered, washed with water and crystallized from methanol-petroleum ether mixture.

IR; 1-d(cm^{-1})**:**3010(=CH-), 2935 (-CH-), 1710(>C=O),1665 (C=N-), 1605(>C=C<), 1450(-CH_2-), 1390(-CH_3-), 1260 (C-N) 1225 (-N-N-), 1167 (-C-O-) 1105(-C-O-C).

^{1}H NMR (DMSO): 1-e: 2.5675, doudlet (2H) (CH_2-cyclic), 3.8489, singlet (3H) (-OCH_3-), 4.5719, singlate (2H)(-CH_2-),5.0906 triplet (1H) (-CH<) 8.5267, singlet (1H) (Ar-CH=N-), 6.6093-8.0824 multiplate (16H) (Ar-H).

2.2 Preparation of *N*-(substitutedphenyl)-1-{4-[2-{5-(4-chlorophenyl)-3-(4-methoxyphenyl)-4,5-dihydro-pyrazol-1-yl}-2-oxoethoxy]phenyl}-1-(phenyldiazenyl)methanimine(2a-2j)

A solution of aniline (0.01M)in glacial acetic acid (10ml) concentrated hydrochloric acid (3ml) was added at 0 to 5^{o}C. Then a solution of saturated $NaNO_2$ (1g in 5ml of water) was mixed to above solution. The diazonium salt solution thus prepared was added drop by drop to a solution of compound N-[{4-[2-{5-(4-chlorophenyl)-3-(4-methoxyphenyl)-4,5-dihydro-pyrazol-1-yl}-2-oxoethoxy]phenyl}methylene]substitutedaniline(0.01M),inmethanol (40 ml) with constant stirring at 0^{o}C temperature. The reaction mixture was kept at room temperature for a day and then poured into crushed ice. The resulting solid was washed with the water and obtained product was crystallized from absolute ethanol.

IR; 2-b(cm^{-1})**:**3015(=CH-), 2930 (-CH-stretch), 1715(>C=O),1655 (C=N-str),1600(N=N), 1610(>C=C<) aromatic,1440 (-CH_2-bend),1370(-CH_3-bend),1255 (C-N) 1225 (-N-N-), 1110(-C-O-C), 605(C-Cl).

^{1}H NMR (DMSO); 2-j: 2.5429,doudlet (2H) (CH_2-cyclic),3.8383 singlet (3H)(-OCH_3-), 4.8565, singlate (2H)(-CH_2-),4.9494 triplet (1H) (-CH<) 6.8707-8.4322multiplate (21H) (Ar-H).

Reaction Scheme

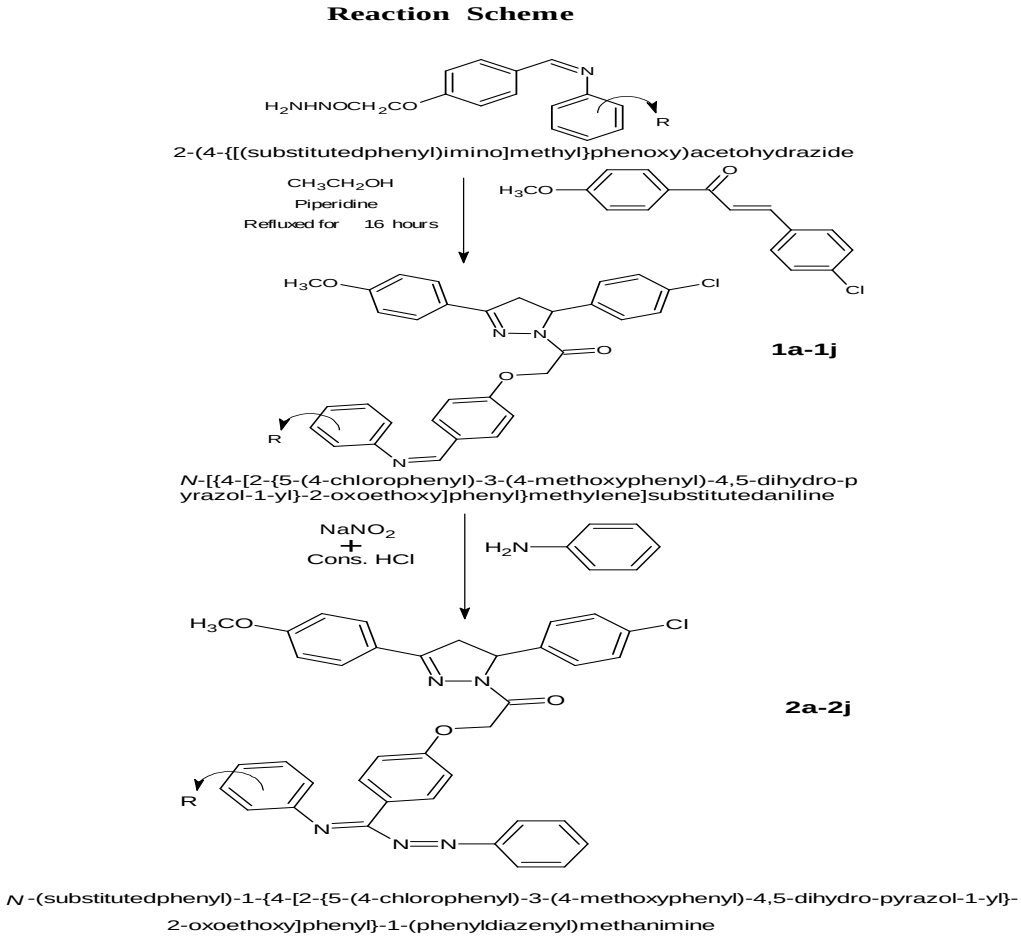

Table 1: *N*-(substitutedphenyl)-1-{4-[2-{5-(4-chlorophenyl)-3-(4-methoxyphenyl)-4,5-dihydro-pyrazol-1-yl}-2-oxoethoxy]phenyl}-1-phenyldiazenyl)methanimine

Sr. No.	Sample No.	R	Molecular Formula	Molecular Weight	Melting Point C	Yield	% C		% H		% N	
							Found	Required	Found	Required	Found	Required
1	2a	1-PHENYL	$C_{37}H_{30}ClN_5O_3$	628.12	98	73	70.71	70.75	4.79	4.81	1.12	11.15
2	2b	1-AMINO	$C_{41}H_{32}ClN_5O_3$	678.18	95	66	72.59	72.61	4.74	4.76	10.3	10.33
3	2c	4-CH_3	$C_{38}H_{32}ClN_5O_3$	642.15	130	70	71.04	71.08	5	5.02	10.88	10.91
4	2d	3-CH_3	$C_{38}H_{32}ClN_5O_3$	642.15	108	69	71.08	71.08	4.99	5.02	10.87	10.91
5	2e	2-NO_2	$C_{37}H_{29}ClN_6O_5$	673.12	114	68	65.99	66.02	4.3	4.34	12.45	12.49
6	2f	3-NO_2	$C_{37}H_{29}ClN_6O_5$	673.12	123	64	65.98	66.02	4.31	4.34	12.47	12.49
7	2g	4-NO_2	$C_{37}H_{29}ClN_6O_5$	673.12	117	71	66	66.02	4.32	4.34	12.46	12.49
8	2h	2-Cl	$C_{37}H_{29}Cl_2N_5O_3$	662.56	104	69	67.04	67.07	4.39	4.41	10.55	10.57
9	2i	3-Cl	$C_{37}H_{29}Cl_2N_5O_3$	662.56	113	72	67.05	67.07	4.38	4.41	10.54	10.57
10	2j	4-Cl	$C_{37}H_{29}Cl_2N_5O_3$	662.56	101	65	67.03	67.07	4.37	4.41	10.53	10.57

Table 2: Antimicrobial activity of *N*-(substitutedphenyl)-1-{4-[2-{5-(4-chlorophenyl)-3-(4-methoxyphenyl)-4,5-dihydro-pyrazol-1-yl}-2-oxoethoxy]phenyl}-1-(phenyldiazenyl) methanimine

SR. NO.	COMP. NO.	R	ANTIBACTERIAL ACTIVITY Minimal Inhibition Concentration(µg/ml)				ANTIFUNGAL ACTIVITY Minimal Inhibition Concentration (µg/ml)		
			Gram negative bacteria		Gram positive bacteria		Fungus		
			E.COLI	P.AERUGINOSA	S.AUREUS	S.PYOGENUS	C.ALBICANS	A.NIGER	A.CLAVATUS
			MTCC 443	MTCC 1688	MTCC 96	MTCC 442	MTCC 227	MTCC 282	MTCC 1323
1	2a	1-Phenyl	250	150	150	200	500	600	700
2	2b	1- Napthyl	100	125	125	150	700	700	500
3	2c	-4-CH_3	175	200	175	175	600	500	800
4	2d	-3-CH_3	250	200	125	250	1000	500	1000
5	2e	-2-NO_2	150	175	250	100	900	900	700
6	2f	-3-NO_2	175	150	175	250	>1000	700	>1000
7	2g	-4-NO_2	150	100	150	150	800	900	900
8	2h	-2-Cl	125	175	150	175	900	800	700
9	2i	-3-Cl	250	200	175	200	500	>1000	500
10	2j	-4-Cl	125	100	250	150	600	700	800

3. RESULTS AND DISCUSSION

Antimicrobial activity

The MICs of synthesized compounds were carried out by broth micro dilution method as described by Ratan (2000). The invitro antimicrobial activity of test compounds were assessed against 24 hr cultures of several selected bacteria and fungi. The bacteria used were *E. coli*, *S.aureus*, *P. aeruginosa*, and *S. pyogenus*; the fungi used were *C. albicans*, *A. niger*, *and A.clavatus.* The antimicrobial activity was performed by broth dilution method in DMSO. Gentamycin, Ampicilin, Chloramphenicol, Ciprofloxacin, Norfloxacin, Nystatin and Greseofulvin were used as standard for the evaluation of antibacterial and antifungal activities respectively. The activity was reported byMinimal Inhibition Concentration. The results are summarized in Table-2.Biological screening result of*N*-(substitutedphenyl)-1-{4-[2-{5-(4-chlorophenyl)-3-(4-methoxyphenyl)-4,5-dihydro-pyrazol-1-yl}-2-oxoethoxy]phenyl}-1(phenyldiazenyl) methanimine based derivatives shows that compound (**2b,2h**) have shown betteractivity against E. coli, S. aureus, while rest of all compound possessed good activity againstS. aureus in the range of 125-250 µg/ml. Compounds with substitution 4-chloro (**2e**and **2j)**, showngood antibacterial activity against S. pyogenus, while rest of all derivatives possessed good activityagainst S. pyogenus in the range of 100-250 µg/ml. Compound(**2f)** and (**2i)** is found to besignificant antifungal activist against C. albicans, while rest of all derivatives are poor against A.niger and A.clavatus.

4. CONCLUSION

The Main focus of this research work was to synthesize, characterize and evaluate antimicrobial activities of the newly synthesized Chalcone derivatives, structures of synthesized compounds were confirmed and characterized with the help of analytical data's such as IR and 1H-NMR. In summary, we have described the synthesis and antimicrobial activity of novel*N*-(substitutedphenyl)-1-{4-[2-{5-(4-chlorophenyl)-3-(4-methoxyphenyl)-4,5-dihydro-pyrazol-1-yl}-2-oxoethoxy]phenyl}-1(phenyldiazenyl) methanimine. MIC values revealed that amongst newly synthesized compound having 4-chlorophenyl type linkage has shown good activity against the bacterial strains. Rest of all compounds exhibit moderate improvement in activity against some of the pathogenic strains.

ACKNOWLEDGEMENTS

The authors are thankful to the KKSJ Maninagar Science Colledge, Ahmedabad for providing research Facilities. One of the authors Pinka A. Patel is thankful to UGC. Ganeshkhind, pune. for Teacher Research Fellowship.

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