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Journal of Chemical and Pharmaceutical Research

J. Chem. Pharm. Res., 2010, 2(2): 300-303

ISSN No: 0975-7384

An environmentally benign organic solvent free approach for synthesis of new Schiff bases and evaluation of antibacterial activity

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Abstract

Eight new heterocyclic moiety containing Schiff bases have been synthesized by the condensation of aromatic amines with substituted benzaldehyde under organic solvent free condition efficiently in the presence of water. The Schiff bases were obtained in good yields and were easily isolated by filtration. Their structures were confirmed by IR, ^{1}HNMR and elemental analysis. Most of the Schiff bases have showed potent antibacterial activity.

Key Words: Schiff bases, Synthesis, Organic solvent free, Antibacterial activity.

Introduction

The Schiff bases constitute one of the most active class of the compounds possessing diversified biological activity such as antitubercular [1],anticancer [2], antibacterial [3-10], antifungal [10], analgesic [11], CNS depressant [11], anti-inflammatory [12], anticonvulsant [13], insecticidal [14], plant growth inhibitors [15]. Schiff bases are used as starting material for the synthesis of various bioactive heterocyclic compounds like 4-thiazolidinones, 2-azetidinones, benzoxazines and formazans. One of the important role of Schiff base is an intermediate in the biologically important transamination reaction. Schiff bases are used as protective agent in natural rubber [16]. Schiff bases are used as amino protective group in organic synthesis. Dabholkar and More [17] have synthesized Schiff bases under microwave irradiation. Recently Schiff bases [18,19] have been synthesized by condensing carbonyl compounds and amines in water suspension medium. These wide application and diverse potential biological activities of Schiff bases prompted us to synthesisze new Schiff bases containing heterocyclic moiety and to as certain their microbial activity.

Materials and Methods

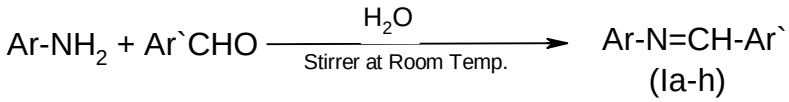
Experimental Section

All melting points were taken in open capillaries and are uncorrected. FT-IR spectra were recorded on perkin-Elmer-157 spectrophotometer instrument using KBr discs. ^{1}HNMR were taken on a Bruker WN-400 FTMHz NMR instrument using DMSO/$CdCl_3$ solvent and TMS as a internal standard.

Typical procedure for preparation of Schiff bases

A mixture of aldehydes and aromatic amines (0.01 mol) were taken in mortar. Added to it acetic acid (0.25 ml), water (5 ml.) and stirred at room temperature for 30-45 min. Reaction was monitored on T.L.C. After completion of reaction, water (25 ml) added.
Separated solid was filtered, washed with water and crystallized from ethyl alcohol. Physical and analytical data is given in Table-1.

Scheme - 1.



Antibacterial Activity:

Synthesized Schiff bases were evaluated for their antibacterial activity against plant pathogen *Xanthomonas citri (Xc), Ervinia carotovara (Ec)* and animal pathogen *Escherichia coli (E.coli)* and *Bacillus subtilis (Bs)*. An activity was studied using disc diffusion method [20] by measuring diamenter of zone of inhibition in mm. The compounds were dissolved in 5% aqueous DMF at the concentration of 150 ppm and discs were soaked and incubated at 27^{o}C for 24 hr. Ampicillin 150 ppm was used as a standard antibiotics for comparision. All the compounds tested showed good inhibitory action but compounds Ia, Ic and Ih. showed slightly more inhibitory action than standard (Table-1).

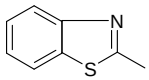
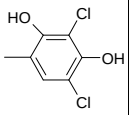
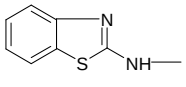
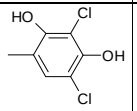
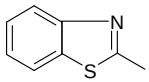
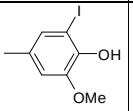
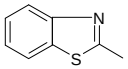
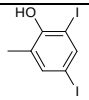
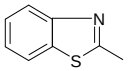
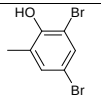
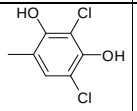
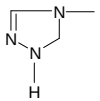
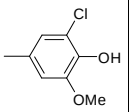
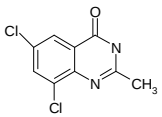
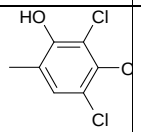
Results and Discussion

In this communication, we have prepared eight new Schiff bases under solvent free condition. Halogenosubstituted hydroxy benzaldehydes and heterocyclic amines were taken in a morter. Added to it traces of acetic acid and water to wait the reaction mixture. Reaction mixture was grinded for 30-45 min. Reaction was monitered on T.L.C. After completion of reaction, water was added and stirred. Separated solid was filtered, washed with water and crystallized from ethyl alconol. Structures of the Schiff bases were confirmed by IR, ^{1}HNMR and elemental analysis. This procedure eliminates the use of organic solvent, completes within 30-45 min. and isolation of the product is simple (Table-1).

Conclusion

Procedure of synthesis of Schiff bases eliminates the use of organic solvent. Reaction completes within 30-45 minutes. Isolation of the product is simple. All the compounds are more or less active to tested bacteria. Compounds Ia, Ic and Ih. found slightly more inhibitory to bacteria than standard.

Table 1 Analytical data, elemental analysis, spectral data and antibacterial activity of compounds (Ia-h)

Entry	Ar	Ar^1	M.P. (^oC)	Yield (%)	Crystal appearance	Time for completion of reaction (min)	Elemental analysis Found (Calculated)		Spectral analysis			Antimicrobial activity			
									IR (cm^{-1})		^{1}HNMR (δ)	Zone of inhibition (in mm) After 12 hour.			
							X= (I,Cl,Br)	N	C=N	C=C Aromatic		*Bs*	*E. coli*	*Ec.*	*Xc*
Ia			192	86	Colorless	40	20.40 (20.88)	9.88 (8.26)	1612	1595,1490	8.48 (s,1H,=CH), 7.45-8.01(m, 5H, ArH). 11.50 (s, 2H,OH).	23	25	29	36
Ib			239	73	Pale pink	40	19.00 (20.00)	11.50 (11.83)	3350 (NH) 1620	1605,1580	3.45 (s, 1H, NH) 8.45 (s,1H,=CH) 7.35-8.35 (s,5H, Ar-H)	18	14	21	18
Ic			188	66	Pale yellow	40	30.45 (30.90)	6.45 (6.81)	1628	1615,1602	3.95 (s,3H,OCH_3) 8.40 (s,1H=CH), 12.05 (s,1H,OH), 7.45-8.26 (m,6H, Ar-H)	27	23	27	34
Id			192	90	Yellow	35	49.85 (50.09)	5.21 (5.52)	1622	1612,1500	8.52 (s,1H, =CH), 11.85 (s,1H, OH), 7.68-8.30 (m,6H, Ar-H)	19	21	20	33
Ie			187	92	Yellow	46	38.60 (38.74)	7.01 (6.77)	1630	1618,1605	8.45 (s,1H,=CH), 12.35 (s,1H,OH), 7.65-8.40 (m,6H, Ar-H)	13	12	16	18
If			252	65	Pale yellow	30	26.12 (25.81)	20.05 (20.36)	3340 (N-H) 1626	1615,1593	4.01 (s,2H,CH_2) 8.35 (s,1H,=CH), 12.90 (s,1H, OH), 7.45 (s, 1H, 2Ar-H), 7.34 (s, 1H, 6Ar-H)	14	15	13	16
Ig			250	81	Colorless	30	13.80 (14.11)	21.62 (21.96)	3135 (NH) 1625	1590,1582	4.25 (s,1H,NH), 3.85 (s,2H,CH_2) 4.05 (s, 3H,OCH_3), 8.55 (s, 1H, = CH), 7.35, (s1H,=2Ar-H), 7.52 (s, 1H,6Ar-H), 7.62 (s, 1H,Ar-H)	16	14	15	22
Ih			285	68	Colorless	35	29.21 (28.91)	11.16 (11.35)	1615	1608,1586	2.55 (s, 3H, CH_3), 8.40 (s, 1H, =CH), 12.85 (s, 1H,OH), 7.21-8.60 (m, 4H, Ar-H).	24	19	26	32
	Ampicillin											25	22	--	--
	Streptomycin											--	--	25	33

Acknowledgements
The authors are thankful to principal, Yeshwant Mahavidyalaya, Nanded for providing laboratory facilities and the Director of IICT, Hyderabad for providing spectral analysis.

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