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SYNTHESIS AND ANTIBACTERIAL ACTIVITIES OF BIS-SCHIFF BASES VIA GRINDSTONE TECHNIQUE USING FRESH LIME JUICE

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ABSTRACT

Highly efficient and practical approach for Chemistry of Schiff base plays an important role as an intermediate in the biologically important transamination reaction. Transamination reaction is a chemical process in which an amino group is transferred from one molecule to another. In living system, the amino group of an amino acid is transferred to carbonyl group of another molecule. The Schiff bases have been reported to possess higher degrees of antimicrobial activities such as anti-bacterial, anti-fungal, anti-tuberculosis, anti-cancer etc.

Literature survey reveals the various Schiff bases have been used in utility of potent biological activity. The wide application and diverse potential activities of Schiff bases promoted us to synthesize some Bis-Schiff bases and to show their antibacterial activity by green synthetic route.

KEYWORDS: Benzaldehyde, Bis-Schiff bases, Grindstone techniques, ethylene diamine.

INTRODUCTION

Aromatic primary amines undergo reaction with aldehydes and ketones to form benzyldine compounds which are commonly known as Schiff bases. Schiff base is an intermediate in the biologically important transamination reaction.

The Schiff bases constitute one of the most active classes of the compounds possessing diversified biological applications. It has been reported to possess higher degree of antibacterial activities.

Literature survey reveals that various Schiff bases have attracted considerable attention as they have wide range of bioactivity. Such activity is greatly influenced by different substituents and ring systems.

Recently Schiff bases have been synthesized by reaction of aromatic aldehydes or ketones and amines using water as solvent¹. The synthesis of Schiff bases under solvent free condition have been reported by Rajeshwar Rao and Movan².

An environmentally benign synthesis method has received considerable attention. Tania and his coworkers³ have reported synthesis of bis- amine Schiff bases under solvent free condition and also in poly propylene glycol (PPG) as a recyclable reaction medium. Nagvi *et. al.*⁴ have been synthesized Schiff bases using (A) Water based synthesis, (B) Microwave irradiation synthesis, (C) Grinding stone synthesis.

Laulloo *et. al.*⁵ synthesized bis Schiff bases under solvent free conditions. Schiff bases are used as amino protective groups in organic synthesis⁶.

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Based on above literature survey we decided to synthesize some reported Schiff bases by reactive ethylene diamine with highly substituted aromatic aldehydes using grind stone technique with few drops of fresh lime juices

EXPERIMENTAL SECTION

General procedure melting points were recorded in liquid paraffin bath using open capillaries and are uncorrected. ^1H NMR spectra were recorded on Bruker spectosin DPX-300 MHz in DMSO-d_6 , Chemical shift δ -values are reported in PPM downfield from TMS, which was used as internal standard. Mass spectra were recorded on JEOL, JMS D-300 instrument fitted with a JMS 2000 data system at 70ev. Thin Layer Chromatography (TLC) was carried out to monitor the reaction using silica gel G as stationary phase.

Experimental procedure 1, 2 ethylene diamine and substituted benzaldehyde were taken together in 1:2 molar proportions in mortar and few drops of fresh lime juices were added to it and reaction mixture was grinded in mortar for about 4 to 8 minutes. When colour of the reaction mixture changes to pale yellow to dark yellow on completion of reaction. Reaction was monitored on the TLC. After completion of reaction it is filtered using some quantity of water then purified by using ethanol as a solvent. Its physical and analytical data is given in table no. 1

Schematic Work

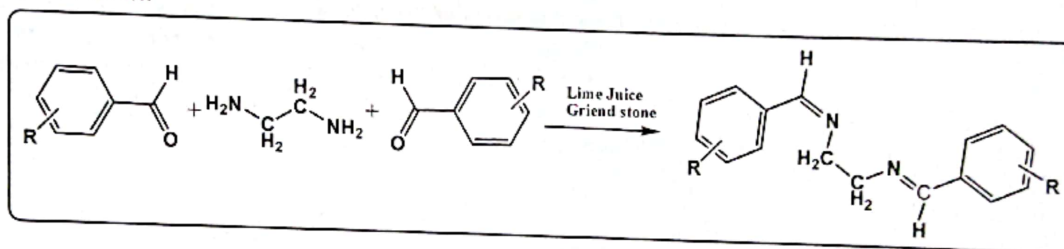


Fig. 1

RESULT AND DISCUSSION

Bis-Schiff bases **3a-f** were obtained in moderate to good yields from condensation reaction between ethylene diamine and highly substituted aromatic aldehyde given by Fig. 1. Ethoxy group containing aldehyde gave excellent yield (Table no. 1). All synthesized compounds were found active against human pathogens *E. coli* and *A. Niger*. Compound **3b** found more active than standard streptomycin against *E. coli* (Table 2).

Table no. 1 Physical & analytical data

Entry	R	R ₁	R ₂	R ₃	X	M.P. (oC)	Yield %
3a	H	I	H	H	H	222	75
3b	H	I	OH	OEt	H	142	85
3c	OH	Br	H	Cl	CH ₃	248	72
3d	OH	H	H	H	H	186	70
3e	OH	I	H	I	H	220	68
3f	OH	H	H	CH ₃	CH ₃	200	80

SPECTRAL ANALYSIS

1. **Compound 3a: Molecular Formula** $C_{16}H_{14}I_2N_2$; IR (V Max) Cm^{-1} 2940, 1760, 1836, 1570, 1400, 1150.; 1NMR ($CDCl_3$) δ 3.92 (S-4H- CH_2-CH_2), δ 7.08-7.7 (M, 4H, -Ar-H), δ 8.10 (S, 2H=CH); MS (m/z) 487 (m+)
2. **Compound 3c: Molecular Formula** $C_{18}H_{16}Br_2Cl_2N_2O_2$; IR (V Max) Cm^{-1} 3010, 2940, 1790, 1800, 1550, 1440, 1151.45.; 1NMR ($CDCl_3$) δ 3.95 (S-4H- CH_2-CH_2), δ 2.4 (S-3H- CH_3) δ 7.8 (S, 2H, -Ar-H), δ 8.10 (S, 2H=CH), δ 5.2 (S, 2H, OH).; MS (m/z) 522 (m+).
3. **Compound 3e: Molecular Formula** $C_{16}H_{12}I_4N_2O_2$; IR (V Max) Cm^{-1} 3020, 2910, 1750, 1831, 1580, 1434, 1150 86; 1NMR ($CDCl_3$) δ 3.95 (S-4H- CH_2-CH_2), δ 7.70-8 (M, 4H, -Ar-H), δ 8.50 (S, 2H=CH) δ 14.50 (S, 2H, OH); MS (m/z) 771 (m+)

Antibacterial Activity

The antibacterial activity checked by human pathogens like *E. coli* and *A. Niger* by using the cup plate agar diffusion method. This was employed to determine the antibacterial activity of the synthesized bis Schiff bases7.

Table 2. Antibacterial Activity of Schiff bases.

Entry	Zone of inhibition in MM	
	<i>E. coli</i>	<i>A. Niger</i>
1a	09	-
1b	25	25
1c	20	18
1d	19	18
1e	17	14
1f	22	16
Streptomycin	24	-

CONCLUSION:

From all above experimental data, analytical and antibacterial activity shows that an environmentally benign synthesis method has been received considerable attention. Grindstone chemistry was used for the preparation of Bis-Schiff bases from 1, 2- ethylene diamine and different substituted benzaldehyde by using fresh lime juice. This synthesis method provides several advantages such as simple workup procedure, Short reaction time, energy free and excellent yield grindstone technique is superior as compared to other known methods of synthesis of Schiff bases and Bis- Schiff bases. No acetic acid was used as a solvent to complete reaction. Few drops of fresh lime juice were sufficient to undergo reaction.

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