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An Ecofriendly Synthesis and Bioactivity Evaluation of New Bromo Schiff's Bases in Water under Stirring Method

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Abstract

An efficient and ecofriendly procedure for the synthesis of a series of bromosubstituted Schiff bases in aqueous medium under stirringmethod. Method was compared with the conventional method. The present work involvescondensation between4-bromobenzaldehyde with various aromatic amines under stirring method. The final products were generated in excellent yields,in short reaction time. The stirring method gives advantageslike very cheap, simple work up procedure, reduced wastes and 100% yields. The structures of synthesized compoundswere confirmed by IR, ^{1}H NMR, and Mass spectralanalysis and evaluated against antibacterial *E.coli* and antifungal *A.niger.*

Keywords: 4-bromo benzaldehyde, substituted aromatic amines, water, stirring, microbial activity.

1. Introduction

Schiff base are the compound containing azomethine group (-HC=N-). They are condensation products of ketones (or) aldehydes (aldehyde and ketones) with primary amines and were first reported by Hugo Schiff in 1864[1].Schiff bases are versatile C=N (imine) containing compounds possessing broad spectrum of biological activity and incorporation of metals in form of complexes showed some degree of antibacterial, antifungal , antitumor and anti-inflammatory activity[2]. Various methodology and routes have been developed for synthesis of Schiff bases. Schiff bases are the important intermediate for the synthesis of various bioactive compounds. Furthermore they are reported to show a variety of biological activities including Antifungal, Anticancer and Antitumor activities [3].Schiff bases are typically formed by the condensation of a primary amine and an aldehyde which involves the use of organic solvents such as methanol, tetrahydrofuran (THF), and 1,2-dichloroethane[4].Aromatic aldehydes especially with an effective conjugation system, form stable Schiff bases, where as those aliphatic aldehydes are unstable and readily polymerize. Schiff base ligands with aldehydes are formed more readily than with ketone (carbonyl carbon).Schiff bases have very flexible and different structures[5].

The Schiff bases constitute one of the most active class of the compound posses biological activities such as antitubercular[6], anticancer[7], plant growth inhibitors[8], insectisidal[9], CNS depressant[10],

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antibacterial[11-12].ArshiNaquvi et.al[13].It was thought worthwhile to be synthesizednewbromo Schiff's bases in water under stirring method.

2. Experimental

Melting points were determined in open capillary tube method and are uncorrected. The IR Spectra were recorded on Perkin-Elmer Spectrophotometer instrument using KBr disc. NMR Spectra were recorded on 200 MHz Spectrophotometerinstrument using $CDCl_3$ solvent and TMS as internal standard.Mass Spectra was taken with Shimadzu GCMS-QP2010 Ultra (semi chemical ionization) with 5-20evionization energy.

General procedure for synthesis of *N*-(4-bromophenyl)methylidene-4-nitroaniline (3a):

Equimolar quantity of 4-bromo benzaldehyde (0.47gm) (0.01M) (1a) and4-Nitro aniline (1.24gm) (0.01M) (2b)was dissolved in 30 ml water and 2to 3 ml methanol in a conical flask at room temperature. Reaction mixture content was kept for stirred at two hours. After two hours the reaction mixture content was poured on crushed ice, solid obtained was filtered & washed with cold water, dried and recrystalized from 95 %ethanol. The purity of compound was checked by TLC. All new synthesised of Schiff bases were confirmed by spectral analysis and antimicrobial activity listed in table 2.

Spectral analysis

3a)N-[(*E*)-(4-bromophenyl)methylidene]-4-nitroaniline:IR:3480,2924,1616,1482, 1230, 1560, ^{1}HNMR: 6.82 (Ar-H, dd),6.53 (Ar-H,dd), 6.39 (Ar-H, dd), 6.74 (Ar-H,dd), 8.60(1H, -CH=N). Mass (m/z): 305.12,298. 12,277.48,156.54, 54.84.

3b)N-[(*E*)-(4-bromophenyl)methylidene]-2-nitroaniline: IR:3482, 2920, 1616, 1482, 1232, 1556. ^{1}HNMR: 6.78 (Ar-H, dd),6.53 (Ar-H,dd), 6.39 (Ar-H, dd), 6.64 (Ar-H,dd), 8.580 (1H, -CH=N). Mass (m/z): 305.12, 272.12, 196.48, 156.54, 54.04.

3c)N-[(*E*)-(4-bromophenyl)methylidene]-3-nitroaniline: IR:3482,2924, 1616, 1482, 1282, 580,1555. ^{1}HNMR: 6.72 (Ar-H, dd),6.53 (Ar-H,dd), 6.66 (Ar-H, dd), 6.64 (Ar-H,dd), 8.60 (1H, -CH=N). Mass (m/z): 305.12,,272.12, 196.48, 156.54, 54.04.

3d) N-[(*E*)-(4-bromophenyl) methylidene] -4-methylaniline: IR : 3482, 2924, 1616, 1482, 1282,580. ^{1}HNMR:6.82 (Ar-H, dd),6.43 (Ar-H,dd), 6.39 (Ar-H, dd), 6.74 (Ar-H,dd), 8.62 (1H, -CH=N). Mass (m/z): 274.15,267.12, 189.48, 154.54, 44.04.

Scheme

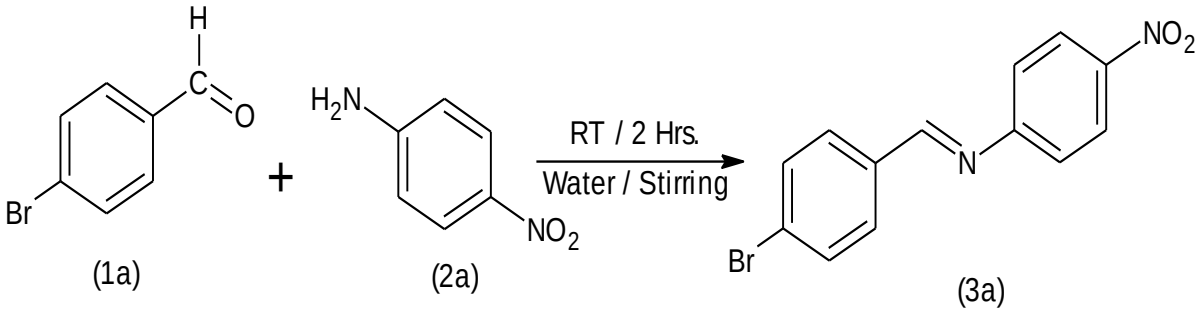

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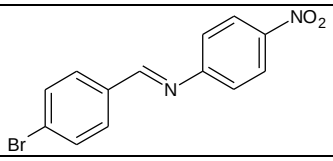
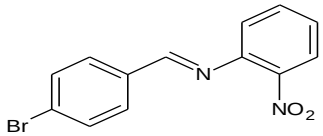
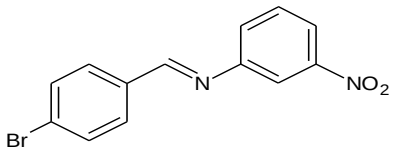
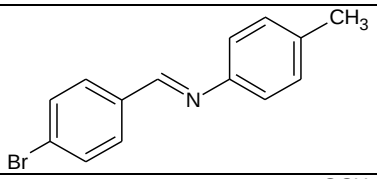
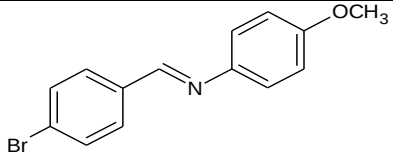
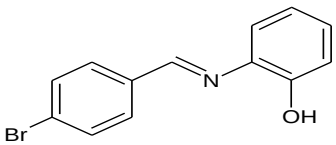
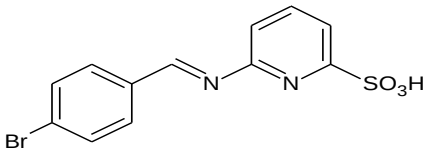
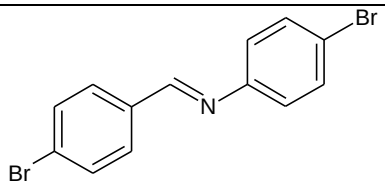
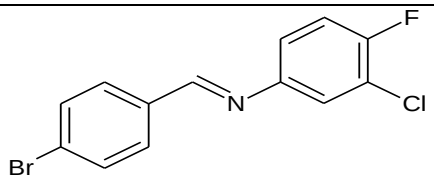
Table No.1:Physical data of synthesised Schiff's Bases

Sr.No.	Structure	M.P.(^{0}c)	Yield (%)
3a		126	92.34
3b		114	89.56
3c		130	87.32
3d		142	89.21
3e		210	90.21
3f		116	83.20
3g		112	78.32
3h		120	93.25
3i		136	94.35

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3e) N-[(*E*)-(4-bromophenyl) methylidene]-4-methoxyaniline: IR : 3482, 2924,1616, 1482, 1282,580,1225. ^{1}HNMR: 6.78 (Ar-H, dd),6.53 (Ar-H,dd), 6.39 (Ar-H, dd), 6.74 (Ar-H,dd), 8.50 (1H, -CH=N). Mass (m/z): 290.15, 270.12, 175.48, 160.54, 44.04.

3f) 2-{[(*E*)-(4-bromophenyl) methylidene] amino}phenol: IR : 3482,2924,1616, 1482, 1282,580. ^{1}HNMR: 6.82 (Ar-H, dd),6.53 (Ar-H,dd), 6.39 (Ar-H, dd), 6.74 (Ar-H,dd), 8.58 (1H, -CH=N). Mass (m/z): 267.12,267.12, 189.48,154.54 ,44.04.

3g) 6-{[(*E*)-(4-bromophenyl) methylidene]amino}pyridine-2-sulfonic acid: IR : 3482,2924,1616, 1482, 1282,580. ^{1}HNMR: 6.78 (Ar-H, dd),6.63 (Ar-H,dd), 6.39 (Ar-H, dd), 6.64 (Ar-H,dd), 8.60 (1H, CH=N). Mass (m/z): 314.17,277.12,195.48,124.54, 54.04.

3h) 4-bromo-*N*-[(*E*)- (4-bromophenyl) methylidene]aniline: IR : 3482,2924,1616, 1482, 1282,580. ^{1}HNMR: 6.82 (Ar-H, dd),6.53 (Ar-H,dd), 6.39 (Ar-H, dd), 6.74 (Ar-H,dd), 8.5 (1H, -CH=N). Mass (m/z): 339.02,277.12,199.48,144.54, 24.04.

3i) N-[(*E*)-(4-bromophenyl) methylidene]-3-chloro-4-fluoroaniline: IR : 3482,2924,1616, 1482, 1282,580,660. ^{1}HNMR: 6.80 (Ar-H, dd),6.63 (Ar-H,dd), 6.40 (Ar-H, dd), 6.64 (Ar-H,dd), 8.60 (1H, -CH=N). Mass (m/z): 312.56,257.12,179.48,124.54, 44.04

Antimicrobial activity

Escherichia coli and fungal strains *Aspergillus Niger* were chosen based on their clinical and pharmacological importance. The bacterial strains obtained from ShriShivaji College, Department of microbiology, Parbhani were used for evaluating antimicrobial activity. The bacterial and fungal stock cultures were incubated for 24 hours at 37°C on nutrient agar (NA) and potato dextrose agar (PDA) medium respectively, following refrigeration storage at 4°C. The bacterial strains were grown in MacConkey's agar plates at 37°C (the bacteria were grown in the MacConkey's brothat 37°C and maintained on MacConkey's agar slants at 4°C) at 28°C. The stock cultures were maintained at 4°C.

Determination of zone of inhibition method

In vitro antibacterial and antifungal activities were examined for Schiff base. Antibacterial and antifungal activities Schiff base for pathogenic bacteria and fungus was investigated by the agar disc diffusion method. Antimicrobial activity testing was carried out by using agar cup method .Schiff base dissolved in sterile distilled water, and stored at 4°C. For the determination of zone of inhibition, pure bacterial culture and fungal strain was taken as a standard antibiotic for comparison of the results. Schiff base was screened for its antibacterial and antifungal activity against the *Escherichia coli*, and the fungi, *Aspergillusniger*. The sets of five dilutions (20, and 30 mg/ml) of schiff base and standard drugs were prepared in double-distilled water using MacConkey's agar tubes. MacConkey's agar sterile plates were seeded with indicator bacterial strains (10^{8} cfu) and allowed to stay at 37°C for 3 hours. Control experiments were carried out under similar condition by used tetracycline for antibacterial activity and griseofulvin for antifungal activity as standard drugs. The zones of growth inhibition around the disc/well were measured after 18 to 24 hours at incubation 37°C for bacteria and 48 to 96 hours for fungi at 28°C. The sensitivities of the microorganism species to Schiff base was determined by measured the sizes of inhibitory zones (including the diameter of disc/ well) on the agar surface around the disc werelabelledand valuesrecorded in table no .2<8 mm were considered as not active against microorganisms.

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Table 2: Zone of inhibition (mm) of Schiff bases

Compounds	Antibacterial *Escherichia coli*	Antifungal *Aspergillus niger*
3a	11	13
3b	_	12
3c	08	_
3d	12	14
3e	09	12
3f	12	_
3g	10	09
3h	12	13
3i	13	14
Control	00	00
Tetracycline	20	00
Griseofulvin	00	20

3. Result &Discussion

A series of Bromo Schiff bases (3a –3i) were synthesized from 4-bromobenzaldehyde with corresponding substituted aromatic amines under stirring method using wateras solvent. They were fully characterized and evaluated for their antibacterial and antifungal activity.Bromo Schiff bases exhibited moderate activity (3d, 3f, 3h, 3i) antibacterial activity against *E. Coli*&antifungal activity (3a,3b,3h,3i).The 3i shows antifungal activity substituted Bromo Schiff bases have shown higher antifungal activity against *Aspergillusniger* .It can be concluded that these Bromo Schiff bases are used as ligand and sources for various metal complexes is importance of inorganic chemistry.They also certainly great promise to good active leads in medicinal chemistry.

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Jeedemetla, Hyderabad for providing spectral analysis.

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