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Synthesis, Characterization and antibacterial study of Bis- Schiff Bases using Natural Acid.

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Abstract: *Bis-Schiff bases are important compounds. They have an important role in the synthesis of metal complexes and other synthetic compounds. The synthesis, characterization and anti-bacterial activities of a series of bis-Schiff bases of o-phenylene diamine with different aromatic aldehydes were studied due to its medicinal utility. Three of the synthesized series of bis Schiff bases were endowed with excellent antibacterial properties. The present work is related to the study of synthetic bis-Schiff bases as possible anti-bacterial agents. In future we have planned to prepare metal complexes from this Schiff bases.*

Key Words: Aldehydes, o-phenylene diamine, anti-bacterial activities, future scope.

1. INTRODUCTION

Schiff bases are an important class of organic ligands formed by the condensation of primary amines with carbonyl compounds, which are widely used in pharmaceutical development, material science and coordination chemistry. (1,2) Schiff bases and bis-Schiff bases are of great interest due to their wide range of biological applications. These compounds are usually synthesised by the condensation of aldehydes and ketones wit primary amines, hydrazine. Attributed to the existence of two azomethine (-CH=N-) group. (3) These compounds have the general formula R1HC=N-N=CHR2.

The primary diamines or di-carbonyl compounds (aldehydes and ketones) are condensed with the corresponding core reagents to form two azomethine (-CH=N-) groups to give bis-Schiff bases. Current methods for increasing yields include conventional reflux, solvent-free grinding, ultrasound assistance and microwave irradiation. Yasemin UNVER et al. synthesised bis(benzo[b]thiophene-2-yl) alkyl methenamine derivatives by reaction of benzo[b]thiophene-2-carbaldehyde with diamines. (4)

All new compounds were characterised by different spectral analysis. These compounds were studied by using binary QSAR based models on therapeutic activity prediction of synthesised compounds. These showed high predicted activities in diseases such as bacterial, angina, allergy, depression and obesity. As a consequence of this theoretical study, they were then tested for their antimicrobial and antileishmanial activities. Some compound found most active in both diseases.

Bis-Schiff bases bearing two azomethine functions in the molecular framework have been found to be important scaffolds for rational ligand design. These compounds are symmetric or asymmetric depending on the nature of the attached substituents. The di-imines & bis-Schiff bases compounds have attracted special interest because of their wide range of pharmacological use. The reaction of primary amines with carbonyl compounds is usually refluxed. The reaction is reversible and the water formed in the reaction medium must be removed to prevent hydrolysis.

Water is often eliminated with the Dean-Stark apparatus. Moreover, the reaction was successfully carried out using dehydration agents such as sodium sulphate and molecular sieve. (5) Schiff bases have attained considerable values in medical and pharmaceutical fields due to their wide range of biological such as analgesic (6), anti-inflammatory (7, 8), anti-microbial

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(9, 10) anti-convulsant (11), anti-tubercular (12), anti-oxidant (13), anti- cancer (14, 15), and anthelmintic (16). The nitrogen atom of the azomethine C=N linkage is involved in the formation of hydrogen bond with the active sites of cellular components and interferes with normal cell function (17-20).

The paper of T. R. van Den Ancker, et. al., Green Chemistry, 2006, 8, 564-568 describes the two main benign approaches for the synthesis of pure bis-imine Schiff bases via the condensation of aliphatic diamines with aldehydes. Pure bis-imine Schiff bases are readily accessible in yield, when aliphatic diamine/aldehyde condensation reactions are carried out under solvent-free conditions or in poly (propylene glycol) (PPG) as a recyclable reaction medium, with negligible waste in this synthesis. (21)

Ifzan Arshed et. al. produced three novel bis-Schiff bases, specifically 1,10-(2,20-dibromo-[1,10-biphenyl]-4,40-diyl)bis(N-phenylmethanimine) (BNSB01), 1,10-(2,20-dibromo-[1,10-biphenyl]-4,40-diyl)bis(N-(4-bromophenyl)methenamines) (BNSB02), and 4,40 –(((2,20-dibromo-[1,10-biphenyl]-4,40-diyl)bis(methanylylidene) bis(azanylylidene)) diphenol (BNSB03), which were synthesized. The corrosion inhibition ability of these Schiff bases on mild steel samples in 0.5 M HCl was assessed using electrochemical methods and weight loss techniques. (22)

An uncomplicated and effective method for creating symmetrical bis-Schiff bases has been detailed by Abbas Shockravi, Mahdieh Sadeghpour, and Abolfazl Olyaei. (23) They utilized a condensation reaction of symmetrical primary bis-amine from 5,5'-methylene bis(2-aminothiazole) with various aromatic aldehyde derivatives without solvents at high temperatures. The benefits of these reactions included an easy reaction process, brief reaction times, straightforward workup, catalyst-free conditions, and high-purity products with good to excellent yields.

Wael A. A. Arafa and Raafat M. Shaker created a new series of bis-Schiff bases utilizing aromatic aldehydes and diamines (trans-1,4-diaminocyclohexane, p-xylylene diamine, and ethylenediamine dihydrochloride) without the use of a catalyst. (24) The yields and reaction times required for completion of reactions using conventional heating, microwave (MW), and ultrasound (US) irradiation are analysed. The eco-friendly Sonochemical waves, with electron withdrawing and donating groups present, provide the desired products in high yields and brief time frames. Elemental analysis confirmed the structures of the products. It showed that certain derivatives exhibit more robust intramolecular hydrogen bonding compared to others.

Gladiola Tantaru, V Dorneanu, and Maria Stan created bis-Schiff bases through the condensation of ethyl-o-hydroxybenzene with ethylene diamine and 1-ethyl-salicylidene bis ethylene diamine. These Schiff base compounds exhibit a strong ability to complex with Mn (II) ions, leading to the formation of brown complexes. (25) Gurjaspreet Singh et al. effectively synthesized a symmetric Bis Schiff Base compound, which was comprehensively characterized using multiple spectroscopic methods. (26) The increased sensitivity of Sn (II) assessed via UV-visible spectroscopy. The enhanced sensitivity of DSB to Sn (II) was clear, as the limit of detection (LOD) was determined to be 82. 31nM.

II. MATERIALS AND METHODS

(a) General Procedure:

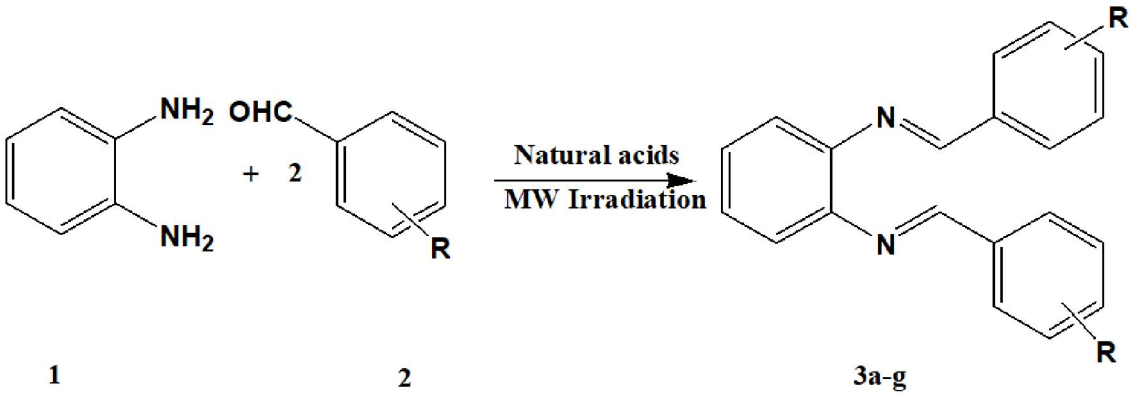
In this study, analytical grade chemicals, reagents, and solvents were used. All of which were purchased from Sima Aldric. The Thin layer chromatographic methods were employed to monitor the formation of the product, using silica gel aluminium plates. The spots were visualized using a UV spectrophotometer. The melting points of the compounds were determined and corrected. Other characterization were carried out using FTIR, 1H-NMR and mass spectroscopic techniques.

(b) Experimental Procedure:

1:2 molar ratio of o-phenylene diamine and aromatic substituted aldehyde exposed to domestic microwave at 180 watts for 30 to 180 seconds utilizing natural acid as a catalyst. Reaction conditions were observed using TLC. Crystals of yellow to greenish yellow hue were created upon cooling the reaction. All the compounds were recrystalized in 50% alcohol. The melting points of these substances were located within a spectrum of bis-Schiff bases.

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Schematic Scheme:

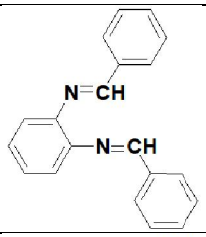
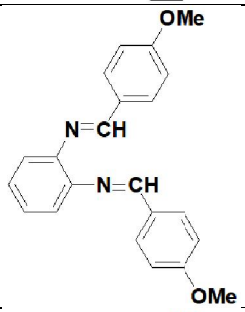
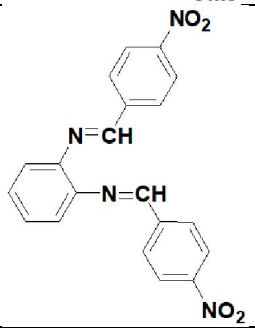


1 2 3a-g

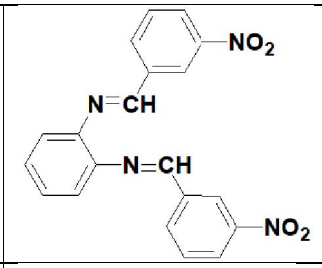
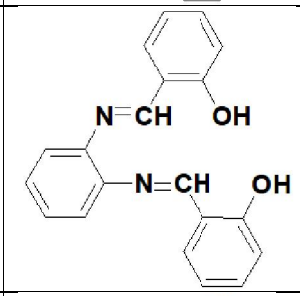
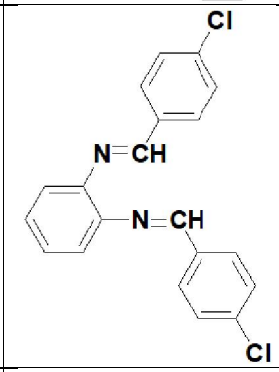
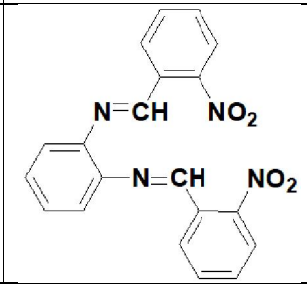
Synthesis of Bis-Schiff Bases from o-phenylene diamine & aromatic aldehyde

III. RESULT AND DISCUSSION

Table No.1 Physical data for newer Bis-Schiff bases: -

Entry	Structure	Time in second	Percentage Yield	Melting point (oC)
3a		4	65.45	160
3b		1	78.73	221
3c		3	80.38	261

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3d		4	90.33	201
3e		2.5	80.30	185
3f		1	66.71	271
3g		3	75.41	185

(c) Spectral Analysis: -

(1) (3c) Molecular formula: C20H14N4O4; M.P. 261 Colour: Reddish; Elemental: C-64.17; H-3.70; N-15.20; O-18.32; FTIR: C-H Ar Str. = 3100-3000 cm-3; C=C Str. 1600-1490 cm-3 ; C=N Str. 1290- 1100 cm-3; 1H-NMR: 7.4, 7.99, 8.30, 8.40 (N=CH); Mol. Wt.: 374.35

(2) (3d) Molecular formula: C20H14N4O4; M.P. 201 Colour: Magenta; Elemental: C-63.17; H-3.72; N-15.25; O-18.34; FTIR: C-H Ar Str. = 3120-3010 cm-3; C=C Str. 1650-1500 cm-3 ; C=N Str. 1300- 1150 cm-3; 1H-NMR: 7.36, 6.90, 7.00, 8. 29 (N=CH); Mol. Wt.: 374.0

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(3) (3e) Molecular formula: C20H16N2O2; M.P. 201 Colour: Yellow; Elemental: C-63.17; H-3.72; N-15.25; O-18.34; FTIR: C-H Ar Str. = 3110-3000 cm-3; C=C Str. 1620-1500 cm-3; C=N Str. 1290- 1130 cm-3; 3600-3300 (-OH) 1H-NMR: 7.3, 7.95, 8.30, 8.38 (N=CH); 5 .00 (-OH) Mol. Wt.: 316.35
(4) (3f) Molecular formula: C20H14Cl2N2O2; M.P. 271 Colour: Yellow; Elemental: C-70.17; H-4.12; Cl-20.02; N-4.15; FTIR: C-H Ar Str. = 3110-3000 cm-3; C=C Str. 1620-1500 cm-3 ; C=N Str. 1290- 1130 cm-3; 1H-NMR: 7.31, 7.30, 7.60, 8.39 (N=CH); Mol. Wt.: 352.0
(5) (3g) Molecular formula: C20H14N4O4; M.P. 201 Colour: Magenta; Elemental: C-63.17; H-3.72; N-15.25; O-18.34; FTIR: C-H Ar Str. = 3110-3000 cm-3; C=C Str. 1620-1500 cm-3 ; C=N Str. 1290- 1130 cm-3; 1H-NMR: 7.3, 7.95, 8.30, 8.38 (N=CH); Mol. Wt.: 374.0

(d) Anti-bacterial Activities:

The Bis-Schiff bases are widely utilized in numerous industrial applications. It acts as a precursor for creating more compounds. Especially from bis-Schiff bases, metal complexes are formed. This was clear when human pathogens were considered for study. These substances have shown promise as viable options for creating more effective antibacterial effects. As a result, all synthesized substances are tested against one gram-negative bacterium, such as Escherichia coli, and one gram-positive bacterium, like Staphylococcus aureus, using the cup plate agar diffusion method. The results of this study are shown in table number 2.

Table No. 2 Antibacterial Activities of Bis-Schiff Bases

Entry	Zone of inhibition in MM			
	Escherichia coli		Staphylococcus aureus	
Bacterial isolates Concentrations (mg/ml)	25	50	25	50
3a	7.0	7.5	11	10
3b	8.5	7.0	9.5	12
3c	11.0	12.5	14.5	12.5
3d	7.5	9.5	10.2	9.8
3e	7.8	10.0	12.4	13.5
3f	7.1	6.6	11.6	12.5
3g	6.0	8.2	10.5	9.7
Amoxicillin (20 mcg)	14		18	

IV. DISCUSSION

When recent bis-Schiff bases were contrasted with those in the literature, the cited bis-Schiff bases were prepared using traditional methods. These methods were labour-intensive, necessitate electricity for heating, and the yields produced were relatively much lower than our approach. Derivatives of Bis-Schiff Bases produced using environmentally friendly chemistry techniques. A set of Bis-Schiff Bases derivatives was produced by reacting o-phenylenediamine with various aromatic aldehydes, yielding good results. The generated compounds underwent purification via recrystallization and were analyzed with melting point and spectroscopy methods, confirming their identity as Bis-Schiff Bases.

The methodology applied in this research paper was more effective, environmentally friendly, and quicker. All the newly formed compounds emerged within the 1-4 minutes. All the new compounds demonstrate antibacterial properties. Table No. 2 shows that entry no. 3c and no. 3e exhibit higher activity against gram -ve and gram +ve bacteria.

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V. CONCLUSION

A straightforward, efficient, affordable, and eco-friendly approach was created for making Bis-Schiff Bases derivatives by following green chemistry methods. A group of Bis-Schiff Bases derivatives were made by reacting o-phenylenediamine with different aromatic aldehydes, and they were obtained in good yields. The produced compounds were purified through recrystallization and checked using melting point and spectroscopy techniques, which confirmed their structure as Bis-Schiff Bases. The compound Bis-Schiff Bases with entry no. 3c & 3e showed good microbiological activity and met the standards set by the Indian Pharmacopoeia (IP 2018). The method used is easy to follow, inexpensive, safe for the environment, and can be used for more research in medicine and drug development.

VI. ACKNOWLEDGEMENT

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