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#### AN ANALYTICAL STUDY OF SYNTHESIS AND CHARACTERIZATION OF SCHIFFS BASES BY USING MICROWAVE



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#### ABSTRACT

The schiffs bases have enormous biological importance. There are number of publications showing antimicrobial, antitumor, anticancer, and antifungal activity of Schiff bases. On the basis of previous finding, we did a microwave assisted and conventional synthesis of Schiff base. Researcher found that the microwave assisted method is more efficient and provide good yield of the products. Furthermore, the Schiff bases have been screened for their biological activity against



E.Coli and Basillus subtilis bacteria among all our Schiff bases 2-hydroxy benzaldehyde and paraanisidine shown a potent activity. The aim of this present research article is to screen simple and economic methods for synthesis of Schiff bases.

**KEYWORDS**: schiffs bases, conventional synthesis, 2-hydroxy benzaldehyde, para-anisidine.

#### **1.INTRODUCTION :**

Schiff bases are derived from the primary aromatic amine and carbonyl compounds by nucleophilic addition forming a hemiaminal, followed by a dehydration to generate an immine and they were first reported by Hugo Schiff in 1864, that contains a C=N with the nitrogen atom attached to an aryl or alkyl group, having general formula R-CH=N-R Where R is an aryl or alkyl group. Which may be variously substituted such compounds are known as azomethine and is particularly for binding via. The nitrogen atom lone pair especially when used in combination with one or more donor atoms to form poly dentate chelating ligands.

The Schiff bases are organic compounds they contain carbon-nitrogen double bound plays a vital role in the progresses of chemistry science. Schiff bases ligands have been used as fine chemicals and medicinal substrates. Schiff bases offer a versatile and flexible series of ligands capable to bind

with various metal ions to give complexes with suitable properties and biologically application since the publication of Schiff bases compounds have been structurally characterized and extensively investigated. Schiff base and there complex is have been extensively studied over past few decades of the various classes of Schiff bases which can be prepared by condensation of different type of amines and carbonyl compounds.

Schiff base synthesized is often carried out with acid catalyzed by refluxing the mixture of aromatic aldehyde and aryl amine in organic medium. However with the assistant of microwave irradiation, it was observed that the condensation reaction of aryl amine and various aldehydes could precede fast and exallent yield. The product could be purified by re-crystallization in an organic solvent. The yields of products were high.

The aim of this present research article is to screen simple and economic methods for synthesis of Schiff bases. Here in the microwave (mw) promoted condensation reaction of 2-hydroxy benzaldehyde with aryl amines displayed the convenient practicing way for forming a series of Schiff bases.

#### **2.LITERATURE SURVEY**

Schiff base are an important class of compounds, which have number of application such as preparative use, identification, detection and determination of aldehydes or ketones. The Schiff bases are organic compounds they contain carbon–nitrogen double bond plays significant role in organic synthesis. This can be achieving by the reaction of aldehydes and amines in acidic medium which leads to synthesis of Schiff bases (imines).

In recent years, several improved procedures have been reported for the synthesis of Schiff's base ligands using different catalyst and routes. In the present proposed, a brief review of literature on synthesis and biological active Schiff base by various workers is mentioned.

ManiKpuri et al [1] have reported the synthesis and antimicrobial activity of Schiff base. Mohamed et al [2] have synthesized microwave assisted some Schiff's base derived from sulphanilamide and aldehydes. The Schiff's base ligands were characterized by IR, NMR. The free ligands were also screened for antimicrobial activities again S.Aureus, E.coli and C.Albicans at the concentration level of by using Agur diffusion method. Roman et al [3] have studied the synthesis and characterization of to entry new Schiff's bases from ortho-hydroxyl aryl aldehydes and aryl amines. Schiff's base ligands were analyses by elemental analysis, IR, NMR and C-NMR.

New chelating ligands of mono and Tri Schiff's bases of thiozole derivative were prepared by Doss et al [4]. They reported mass, IR, H'NMR and antimicrobial activity by paper disc diffusion method. Gupta et al [5] have reported some transition metal complexes with Schiff bases obtained from 2-.Pyridyl carboxyaldehyde and ethylene diamine. Then their Cu (II), Co (II), Ni (II), Cd (II), Zn (II), and Hg (II) complexes have been prepared. The synthesized ligands and their metal complexes were also screened for their antibacterial activity against bacterial species E.coli, S.Bacillus Subtils and Salmonella typhi. Singh et al [6] have found that the synthesis and antimicrobial activity of Schiff's and N-Mannich bases of Isatin and its derivatives with 4-amino-N-carbomimidoyl benzene. The authors have also reported the IR, NMR elemental analysis and the antimicrobial activity of the synthesized compounds were evaluated by tube diffusion method. The synthesized compounds showed better antibacterial activity then the reference drugs. Herin and cynamon[7] have investigated the synthesis and antitubercular activity of Schiff base of the following type.

Nair et al [8] have reported the synthesis and antibacterial activity of some Schiff base complexes. The Schiff bases showed greater activity than their metal complexes. GAO and Zheng [9]

have studied the synthesized of optically active Schiff base obtained from the condensation of 2hydroxy acetophenones and 1, 2-diamino cyclo hexane. Gudasi et al [10] have investigated the synthesis, characterization biological activity of dioxalranium (II) and thorium (II) metal complexes of Schiff bases obtained from 2-amino pyridine and acetophenones. A. Siri et al [11] the UV visible spectra of some Schiff bases obtained from 2-amino pyridine and 2-amino pyrazine have been suggested in acetonitrile and toluene. The compounds were in tautomeric equilibrium in polar and non-polar solvent.

#### **3. EXPERIMENTAL SECTION**

All the organic chemicals and solvents were obtained from commercial sources (SD fine, Loba) and used without further purification. Melting point were taken on a Viggo melting point apparatus and uncorrected. Purity of the yield was checked by TLC of the compound was performed on silica gel aluminum coated plate with pet ether and ethyl acetate as solvent. The absorbance maxima (wavelength) were recorded on shimadzu UV visible spectrophotometer, 1 H-NMR was recorded on 300MHz using DMSO. The title compounds were screened for their antibacterial and antifungal activity. A conventional household microwave oven operating at 900W was used for irradiation. All the products are known compounds and are characterized by comparing their physical and spectral characteristics with those reported in literature.

#### **4.GENERAL PROCEDURE FOR PREPARATION OF SCHIFF BASE**

In the present work Researcher have to develop one pot synthesis of Schiff base. An equimolar amine and aromatic aldehyde are dissolved with minimum amount of methanol then 2; 3 drops of concentrated sulphuric acid are added to the mixture. The mixture was irradiated inside Samsung microwave oven (100 watt) for about 2-3 min progress of reaction was monitored by TLC after completion of the reaction. Then it's cooled to the room temperature and the Schiff bases that separated are filtered with suction washed with a little methanol and recrystalized from hot ethanol.

#### **5.GENERAL REACTION BY USING MICROWAVE**



#### 5.1 Mechanism for Schiff base synthesis:-





Similarly researcher calculates the different Practical and Theoretical Yield of complex using the above method.

#### 6.2 Synthesis of Schiff Base by using Microwave



#### 6.3 Synthesis of Schiff Base by using Microwave



#### 6.4 Synthesis of Schiff Base by using Microwave



#### Table No.1 Physical characterization of Schiff base by using microwave

Entry	R <sub>1</sub>	R <sub>2</sub>	Time	Power	yield	m.p
	Aldehyde	Amine	(min)	(M.W)		(found)
1	4-NO <sub>2</sub>	4-OCH <sub>3</sub>	3 min	100	88 %	70
2	4-OCH <sub>3</sub>	4-OCH <sub>3</sub>	4 min	100	80.91 %	143
3	4-OH	4-OCH3	4 min	100	82.60	217
4	Н	2-ОН	4 min	100	90 %	48
5	2-ОН	4-OCH <sub>3</sub>	3 min	100	94.71 %	147
6	2-ОН	Н	4 min	100	93.22 %	48
7	2-ОН	4-CH <sub>3</sub>	3 min	100	94.04 %	104
8	2-ОН	4-C1	3 min	100	88.40 %	103
9	2-ОН	-NH <sub>2</sub>	3 min	100	90%	200

#### 7.0 SPECTRAL ANALYSIS OF SCHIFF'S BASE (COMPOUND NO. 1)





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#### Figure No.2 NMR Spectral analysis report of schiffs base of compound no.1

#### **8.RESULT AND DISCUSSION**

It is found that the condensation reaction of a carbonyl compounds and primary aryl amine leading to the formation of Schiff bases should be facile reaction due to the good electrophilic and nucleophilic characteristics properties of the carbonyl and amine groups. The present methodology we have in reported efficient way no form Schiff base, in which microwave irradiation plays an important role for condensation reaction of aldehydes and aryl amines.

The method herein showed that the condensation reaction between P-anisidine and salicylaldehyde, p-nitro benzaldehyde proceed smoothly, in which microwave irradiation of equimolecular or amount from P-anisidine and aryl benzaldehyde in methanol and in the presence of 2, 3 drops of sulfuric acid and curd filtrate for a very short period it gives excellent to good yield of the desired Schiff base. Recrystalization from ethanol gave the pure products as colored compounds.

#### **9.CONCLUSION**

In this present project we are reporting the most convenient way to synthesize the Schiff base or immines, in which microwave irradiation plays an important role for promoting condensation reaction of P-anisidine and substituted aryl aldehydes. In conclusion, in this work we investigate a simple efficient and fast method has been developed for the synthesis of Schiff bases under microwave irradiation method. The application of microwave irradiation in combinatorial chemistry becomes powerful tool in accelerating the pace of library synthesis. Domestic microwave ovens are very popularity used in organic synthesis because of its low cost and easily available, shorter reaction time and products are obtained in excellent yields. Furthermore this methodology also follows several principles of green chemistry.

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