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The low cost, Fast and environment friendly method of formation of Schiff base of 4-nitroaniline and its Fe, Co, Cu and Sn complexes by using microwave irradiation

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Abstract:

The formation of new Schiff bases and their metal complexes is a great area of interest for many researchers. But by conventional method they are produced by using high quantity of chemicals, needs more time and work. In this regard microwave irradiation method is more active, fast, and environment friendly method of preparing Schiff bases and its metal complexes. Schiff base of 4-nitroaniline and its Fe, Co, Cu, and Sn complexes are made both by conventional and microwave irradiation method. In this work the aim is the comparison to identify that the microwave irradiation method reduces the time, lowers the quantity of waste, and also gives high yield at lesser amount of chemicals. Synthesis was confirmed by FTIR and antibacterial

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activity was carried out against E coli and Bacillus Sabtilis to depict the importance of work.

Key words: Schiff bases, 4-nitroaniline, microwave irradiation

INTRODUCTION

The name schiff base is for a class of compounds having azomethine group which is RHC=N-R. This class of compound was first described by Hugo Schiff (1864), that's the reason for which they are known Schiff base. According to nature they are basic [1, 2]. They are also known as imines and formed by the condensation process of primary amines with carbonyl group containing aldehyde or ketone. The Schiff bases of aromatic aldehydes or ketones are more stable than the Schiff bases formed by aliphatic aldehyde or ketone. The reason behind is the effective conjugation in the structure [3, 4, 5, 6, 7] on the other hand the Schiff base of aliphatic aldehyde or ketone are unstable and polymerizable. [8]. In present time the Schiff base metal complexes are very important as their wide application is observed in different fields e.g. In pharmaceutical chemistry and in toxicological studding. Their use as antibacterial, antifungal and antiviral drugs is also known. They are also used as anticancer drug [9] anti-tumor [10]. the conventional method for the formation of Schiff bases and their metal complexes is by refluxing the mixture of amines and aldehyde or ketone in organic solvent under azeotropic conditions in order to separate the water formed but this method needs solvent in high quantity and takes a lot of time for reflux while another method for the formation of Schiff base and its metal complex is microwave irradiation method. This method in most of cases needs lesser amount of solvent and in some cases no solvent is needed [10]. This also have advantage to reduce the

danger of having explosions due to solvent when reaction takes place in a close flask in microwave oven[11] which is also an environment friendly, economical (as solvent is used in less quantity or not used) and time saving method. So we tried and successful in the formation of a Schiff base by reacting 4-nitroaniline with salicylaldehyde and its Fe, Co, Cu and Sn metal complex by micro wave irradiation methods. This Schiff base was also made by us and this type is known earlier too [12] by general method but the method for these compounds is new and the Sn complex is new. The microwave irradiation method gives more yields and lesser time with lesser amount of solvent. The reaction is in Figure.1

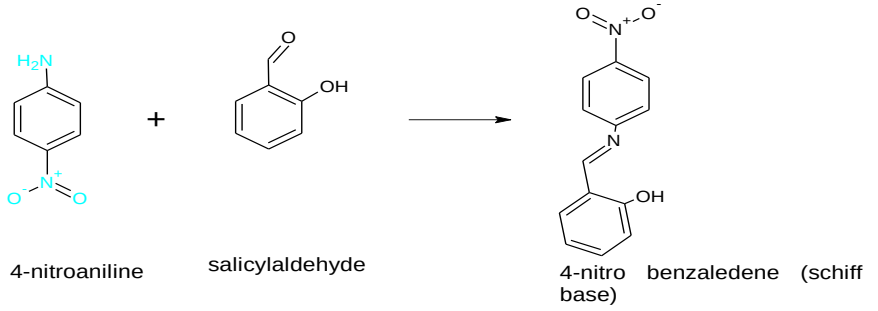


Figure 1: Scheme for Ligand synthesis.

EXPERIMENTAL, MATERIALS AND METHODS

All chemicals and solvents used for this work are of analytical grades. Melting points of the synthesized compounds were determined by using open capillaries and were uncorrected. The reaction progress was monitored by using TLC of Merck silica gel coated aluminum plates and the solvent system used was Chloroform and n- hexane in 1:2. The spots are visualized under UV lamp and single spot indicates the completion of reaction. The IR spectra were recorded by FTIR of Quaid e Azam University Islamabad. The microwave used for microwave irradiation method was Haire model no

Formation of Schiff base and metal complex by using conventional method

4-nitro aniline and Salicylaldehyde were taken in equimolar amounts in methanol are refluxed for 6 hours. The orange colored powder product was 9 of Schiff base in chloroform and metal salt in methanol. The reaction is refluxed for 4 to 12 hours. The solid product was dried and recrystallized. [13]

Formation of Schiff base and metal complex by using microwave irradiation

The microwave irradiation condensation of 4-nitroaniline and salicylaldehyde and their metal complex were carried out by using domestic oven, Haire Model. 5mmol of salicylaldehyde and equimolar amount of 4-nitroaniline were mixed together in an Erlenmeyer flask (25 ml). The power was set at 200W. After 2min 30 sec the product was found and then recrystallized with ethanol. The metal complex was formed by taking equimolar amounts of both ligand and metal salt in Erlenmeyer flask in methanol (5ml) and choloroform (5ml). After 40 sec to 2min the metal complexes were produced and then they were recrystallized.

RESULTS AND DISCUSSION

The microwave irradiation method gives high yields and saves time as compare to conventional method. Comparison is given in table 1 and 2.

Table 1: conventional method

compound	Temperature ℃	time	Yield%
L	Ambient	6hrs	75
FeL2[(CH3COO)2]	Ambient	4 hrs	80
CoL2Cl2	Ambient	10 hrs	70
CuL2	Ambient	8 hrs	75
SnL2Cl2	Ambient	12hrs	65

Table 2: Microwave irradiation method

compound	power	Temperature ℃	time	Yield%
L	200W	80	30 Sec	95
FeL2[(CH3COO)2]	200W	80	50 sec	90
CoL2Cl2	200W	80	70 sec	89
CuL2	200W	80	40 sec	92
SnL2Cl2	200W	80	2 min	90

Physical properties of the compounds are in table 3

Table 3: physical properties

Compounds	Color	Melting point **(°C)**	Soluble In
L	orange	182-187	CH3Cl
FeL2[(CH3COO)2]CH3COO	Dark brown	145-148	DMSO
CoL2Cl2	Dark green	160-163	DmSO,Acetone
CuL2	maroon	154-157	DMSO
SnL2Cl2	Rust color	211-216	DMSO

To confirm the formation of Schiff base and its metal complex FTIR is used. According to which the carbonate peak of salicylaldehyde (1670-1820) Cm^{-1} and amine peak of 4-nitroaniline (3300-3500) Cm^{-1} were disappeared showing formation of Schiff base. In addition the appearance of peak at 1606Cm^{-1} shows the formation of azomethene group (-C=N). New peaks at Far IR regions and schiffting of azomethene peak to other frequencies indicates the formation of metal complexes. Same results were observed by other researchers using FTIR [14, 15, 16].

Table 4: IR stretching frequencies

Compound	V C=N	V M-O	V M-N	V M-Cl	Phenyl ring
L	1606				1026 & 738
Fe	1633	679	547		1003 &754
Co	1608	680	515	315	1020 & 755
Cu	1603	689	526		1033 & 745
Sn	1638	628	548	308	1029 & 755

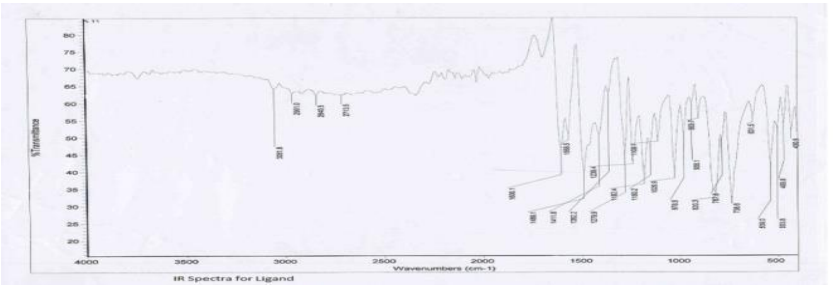


Figure 2: IR Spectra for Ligand

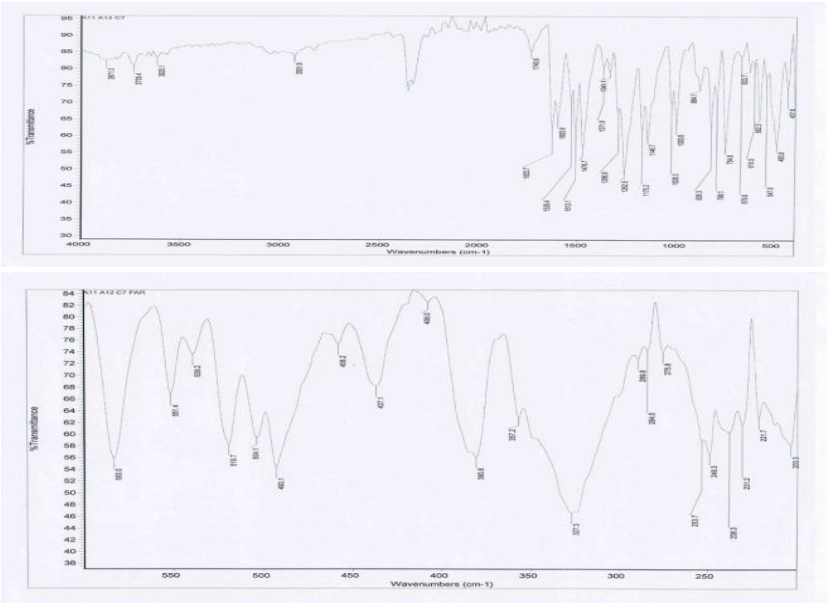
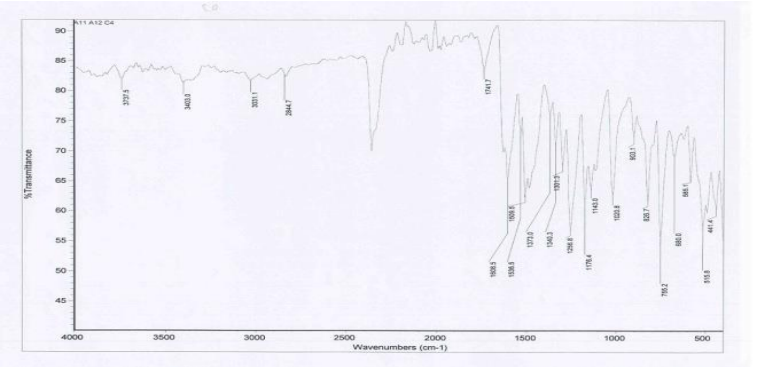


Figure 3: IR Spectra for Fe. Complex



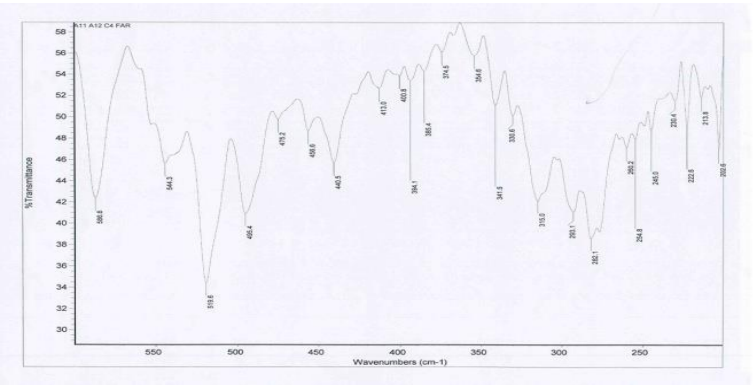


Figure 4: IR Spectra for Co. complex

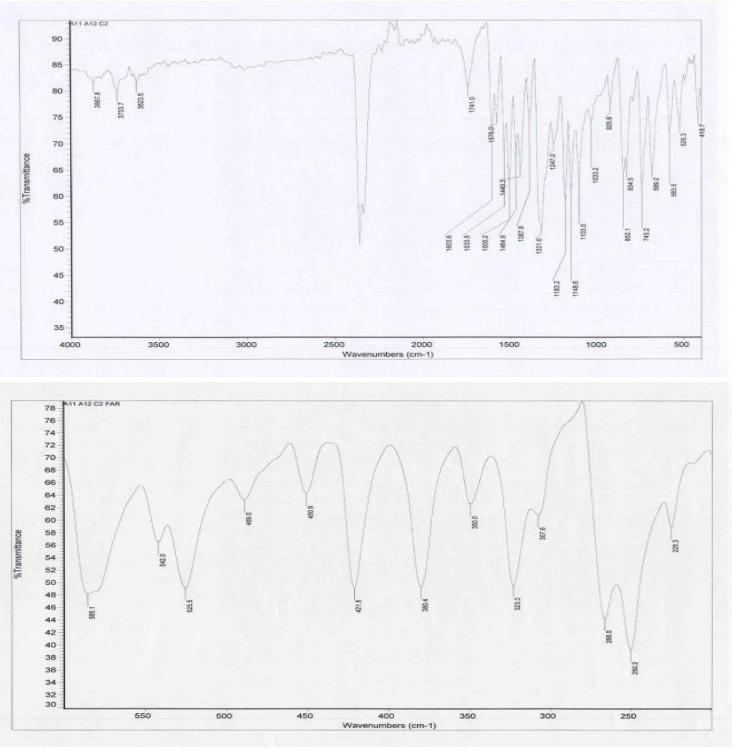


Figure 5: IR Spectra for Cu. Complex

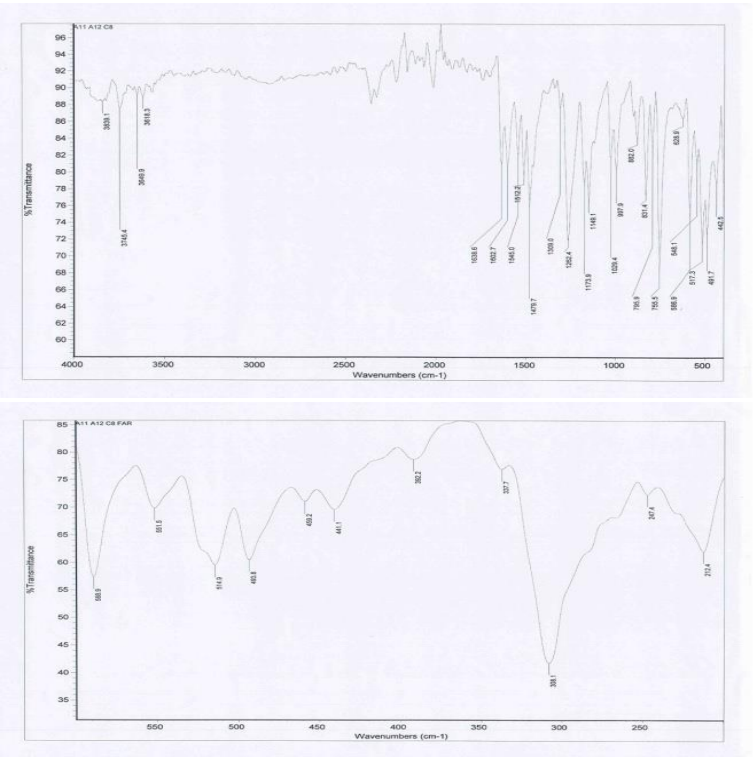


Figure 6: IR Spectra for Sn. complex

Antibacterial activity

For Antibacterial activity the Disk diffusion method is used. This method was first used in 1940. This method is widely used in almost all laboratories for routine antibacterial activity. In this method the disk of standard drug, Doxycycline which is a broad-spectrum antibiotic is used. The media for bacterial growth was Mueller Hinton Agar MHA; the bacterial strands were E.Coli and Staphylococcus aureus. The reason behind the antibacterial activity is that the compound disturbs the structure of membrane and makes holes in it which ultimately cause cell death as cell gradually loses its water and essential nutrients through these holes. The antibacterial activity shows that complexes show more activity than that of a ligand.

Table 5: Antibacterial activities

	Diameter of Zone Inhibition (mm)	
Complexes	E Coli	Staphylococcus aureus
L	6.5	7
FeM2L2	7	9
CoM2L2	8	7
CuM2L2	9	8
SnM2L2	8	7
Standard drug	14	10

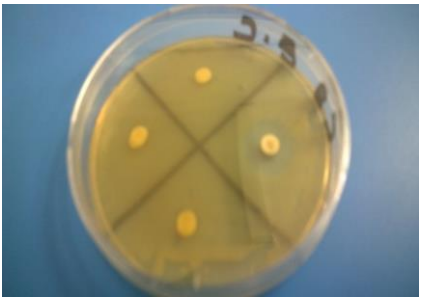


Figure 7: antibacterial activity of Ligand

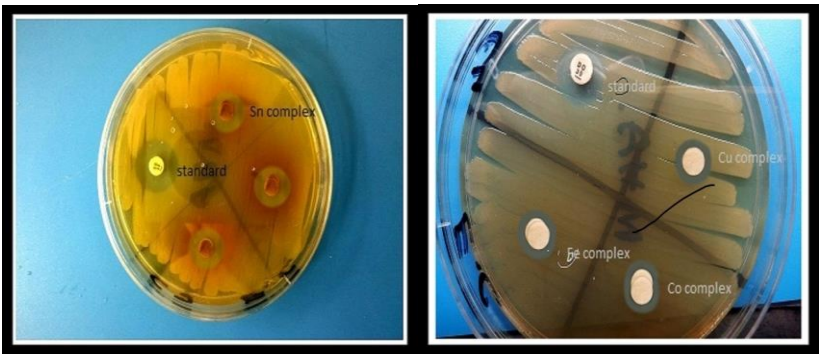


Figure 8: antibacterial activity of complexes

CONCLUSION

The microwave irradiation method is most fast, low cost as it needs lesser amount of solvent and chemicals but gives high yield and also environment friendly method. The antibacterial

studies shows that the complexes have extend to destroy the metabolic activity e.g. protein synthesis and respiration. However this activity is also depended upon the structure of the complexes and Schiff base. The solubility and conductivity also affect their antibacterial activity. The antibacterial activity of complexes is more as compare to the ligand.

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