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Synthesis of Schiff Base Metal Complexes Using Eco-Friendly Catalyst and Study of their Antimicrobial Activities

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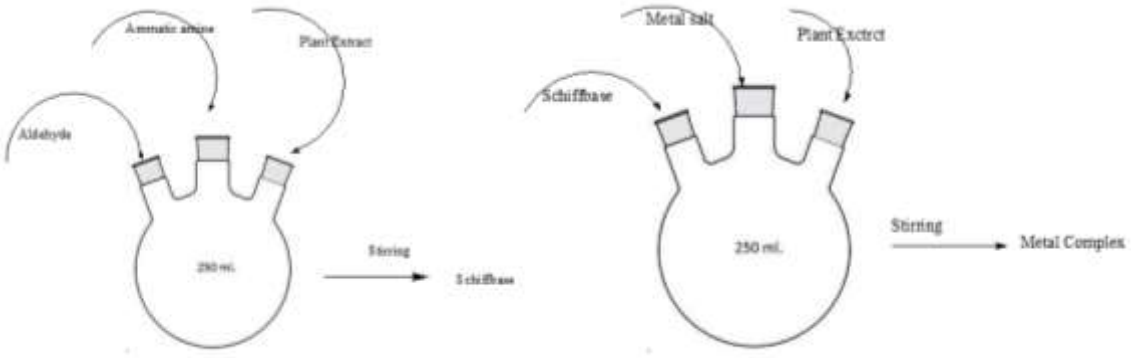
Abstract:
The Schiff base reactions are an important in the formation of Carbon-Nitrogen bonds in synthesis. Schiff bases have a carbon-nitrogen double bond and are named as imine (-RC = N-) . The presence of the imine group in Schiff bases are responsible to show many biological potent activities against microbes. Schiff base compounds are acts as ligands that contain both nitrogen and oxygen lone pair electron donors and are act as polydentate ligands in coordinating ability., it has own application in the field of chemistry. Schiff base expresses a several activities including antibacterial, anticancer, and antiviral. Environment friendly method for the synthesis of Schiff bases in which we use *Tribulus terrestris* L, leaves extract as a green and eco-friendly catalyst. This method is economical, short reaction time, free from organic solvent and give high yield of product. Study of microbial activities against

Keywords: Schiff base metal Complex, Green synthesis, eco-friendly catalyst, Plant extract, antimicrobial activities.

Graphical Abstract:



1. Introduction:

1.1 Schiff base metal Complex:

The usage of Schiff base in chemistry, industry, medicine, drug and pharmacy has increased the interest

in these Schiff base compounds [1-2]. The C atom of the imine (-RC = N-) bond is predisposed to nucleophilic addition, and the nitrogen atom with free electron pair, which is able to form stable complexes with metals [3, 32]. In recent days, there has been notable attention among scientists on Schiff base as a ligands due to their specific of structural characteristics and very large range of physicochemical properties [4]. The interest in Schiff base stems from their structural versatility, due to this the Schiff bases to be effectively employed as asymmetric and stabilizing agents for various complexes in different oxidation states [5-8]. Schiff bases are formed from the compounds of amino and carbonyl groups. These polydented ligands, which are carried out in generating highly substantial complexes with metal ions. They coordinate with metal ions by C=N nitrogen. Schiff base reaction plays a important role in the formation of Carbon nitrogen (C-N) bonds [9-10]. Schiff bases show complex formation property with O, N, and S electron pair donors with that complexes of metal shows a versatile biological activity towards various kinds of diseases, pathogens and tumours [10].

1.2 Antimicrobial activity Schiff base metal Complex:

The biological activities of Schiff base complexes with transition metals and some other are explained [33]. The data refered to the lowest concentration of the tested antimicrobial agent that is able to inhibit the visible growth of the bacterium is investigated [11, 12, 34]. Schiff bases exhibit more potent biological activity compared to free imines [13]. The antimicrobial study was carried out against Staphylococcus aureus, Escheria colii, Aspargillus niger, Candida albacuns.

1.3 Green Synthesis of Schiff base metal Complex:

The use of toxic and hazardous solvents and catalysts in organic synthesis is considered a very important threat for the health and safety of chemist and environmental pollution[35]. These solvents are expensive and their by-products are also toxic for environment and methods by which Schiff bases can be synthesized but these methods are harmful to environment as many organic reagents are toxic and carcinogenic [36]. There are number of Greener methods for the synthesis of Schiff bases are also reported but all these methodologies have some drawbacks like long reaction time, special conditions, special apparatus, cost of dehydrating agent etc. To reduce cost of catalyst herein we are reporting an environment friendly method for the synthesis of Schiff bases in which we use *Tribulus terrestris* L. leaves extract as a eco-friendly catalyst. This method is economical, short reaction time, free from organic solvent and give high yield of product [14]. In ancient medicine, extracts of the aerial parts and fruits have been used for its many medicinal properties [15,16] The preliminary phytochemical study of Tribulus terrestris leaves revealed the presence of saponins, glycosides, alkaloids, flavonoids, and tannins [17]. The furostanol and spirostanol saponins of tigogenin, chlorogenin, ruscogenin, neohecogenin, and sarsasapogenin types are frequently found in this plant [17]. , terrestribisamide, together with 10 known compounds, xanthosine, N-p-coumaroyltyramine, terrestriamide, hecogenin, ferulic acid, vanillin, p-hydroxybenzoic acid, and β-sitosterol, from the dried leaves of TT.[18] The alkaloids present are harmane and norharmane. The β-carboline alkaloid, tribulusterine, is present in minor quantities in fruits[19]. Sterols such as β-sitosterols and stigmasterols were also found to be present[20]. Phytochemical can be act as antimicrobial and antioxidants [21]. These Phytoconstituents due their functional groups can show catalytic properties.

2.0 Materials and methods

Experimental Section

2.1 Preparation of plant extract:-

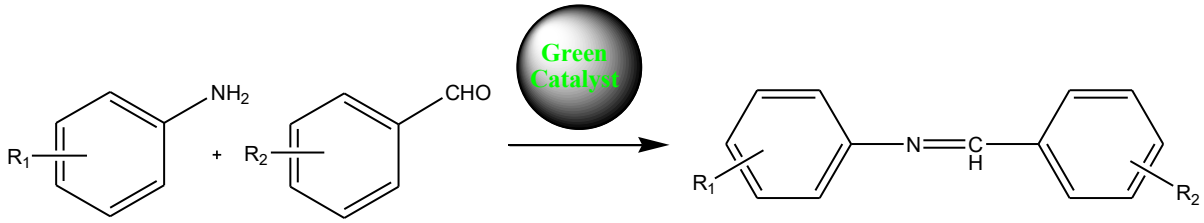
Tribulus terrestris Leaves was collected in local area like waste land, other than agricultural or residential

area (i.e easily available in everywhere in the dhule district, India., no need to cultivate). This plant is wild and not useful for agricultural purpose. This plant material was collected from barren area of College Campus and botanical Garden of S. P. D.M. College Shirpur Dist Dhule, Maharashtra , India. The herbarium was prepared, this plant identified by popular taxonomist Prof. Tayade from PSGVPM's ARTS, Commerce and Science college Shahada affiliated to KBCNMU Jalaon University. The leaves of collected species *Tribulus terrestris* L. were washed in tap water to remove impurities or dirty particals and cleaned well. *Tribulus terrestris* L. leaves was dried in 2-3 days in absence of sunlight (Shade dried). The dried leaves was grinde in grinder to obtain *Tribulus terrestris* L. leaves powder [21]. 50 gm of *Tribulus terrestris* L. leaves powder was dissolved in 150 ml of distilled alcohol in stoppered bottle up to 2-3 days and every 50 min. interval shaking was take place. After 3 days later mixture was filtered and we were obtained plant extract solution. We were used plant extracted solution as catalyst in reaction [22,36].

A) Synthesis of Schiff bases:

The Schiff base are synthesized by using substituted anilines (aromatic aminies) and substituted aromatic aldehydes in presence of green catalyst [23], Extraction was tested for presence of secondary metabolites. As shown in figure no 1 the reactions of aromatic amine and aromatic aldehyde were carried out using *Tribulus terestris* L. leaves extract. To synthesis Schiff base by using 0.01mol substituted aromatic benzaldehyde and 1ml of alcoholic plant extract was added in RBF to this solution 0.01mol aniline added in RBF with constant stirring ,0.5ml plant extract is used as a catalyst the reaction mixture was stirred in magnetic stirrer at room temperature, The reaction was completed within 1 hour[24] .

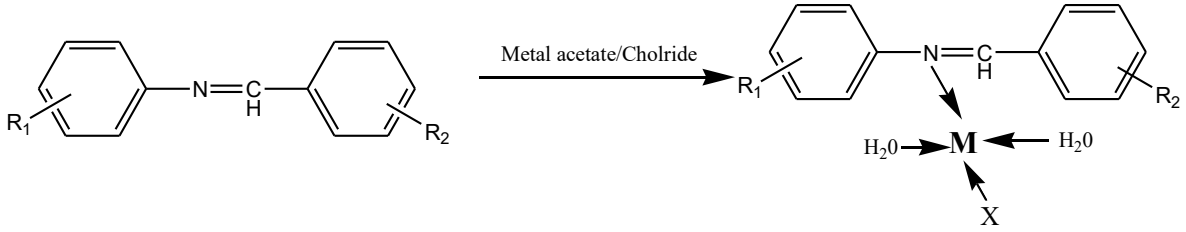
Figure no 1 Synthesis of Schiff base using Aromatic aldehyde and aromatic amine by using *Tribulus terestris* L. leaves extract as a green catalyst.



B) Synthesis of the metal complexes by using the synthesized Schiff bases:

The aqueous solution of metal chlorides salt treated with the ethanolic hot solution of ligand to form the metal complexes with the Schiff base ligands [25,26]. As reaction shown in figure no 2 the Schiff base ligand in 20 mL of ethanol, was added in a 1:2 molar ratio drop wise over 10 min at ambient temperature into a 10 mL solution of $CoCl_2$ stirred for 30 min. The Complex formation Checked by TLC after every 10 min. Crystals of metal complex formed after 30min, Recrystallization of the solid from hot ethanol yielded the compounds [26].

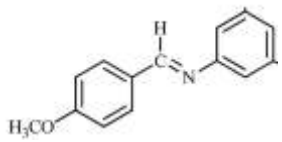
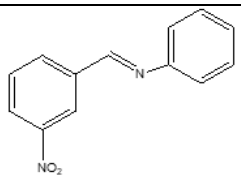
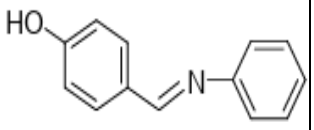
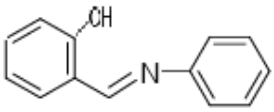
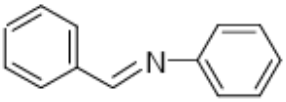
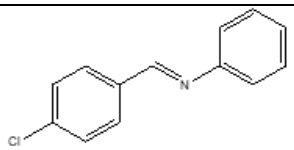
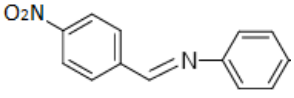
Figure no 2: Synthesis of metal complexes by using the synthesized Schiff bases:



3.0 Results:

Herein, synthesis and to obtained high yield of the Schiff base product with green catalyst (organic material) is developed. This is achieved by adopting the green method for Schiff base reaction between benzaldehyde and aniline with *Tribulus terrestris* leaves extract. As shown in table no 1 it is observed that the desired product N-Benzylideneaniline is formed with 85% yield in 30 min [37] .

Table no 1 Synthesis of Schiffbase and their Co Metal Complexes:

Sr. no	Schiff Base structure	Schiff base name	Metal Complex	Sample code
1		(E)-N-(4-methoxybenzelidine) aniline	[Co((E)-N-(4-methoxybenzelidine) aniline)$_2$ .Cl$_2$.2H$_2$O]	C1-Co
2		(E)-N-(3nitrobezylidene) benzenamine	[Co((E)-N-(3nitrobezylidene) benzenamine)$_2$ .Cl$_2$.2H$_2$O]	C2-Co
3		(E)-4-((phenylimino)methyl)phenol	[Co((E)-4-((phenylimino)methyl)phenol)$_2$ .Cl$_2$.2H$_2$O]	C3-Co
4		(E)-2-(phenylimino)methyl)phenol	[Co((E)-2-(phenylimino)methyl)phenol)$_2$ .Cl$_2$.2H$_2$O]	C4-Co
5		(E)-N-(benzelidine) aniline	[Co((E)-N-(benzelidine) aniline)$_2$ .Cl$_2$.2H$_2$O]	C5-Co
6		(E)-N-(4-chlorobenzylidene)-3-benzenamine	[Co((E)-N-(4-chlorobenzylidene)-3-benzenamine)$_2$ .Cl$_2$.2H$_2$O]	C6-Co
7		(E)-N-(4-Nitrobenzylidene)-3-benzenamine	[Co((E)-N-(4-Nitrobenzylidene)-3-benzenamine)$_2$ .Cl$_2$.2H$_2$O]	C7-Co

3.1 Antimicrobial Testing of synthesized Co Schiffbase metal Complex [27]:

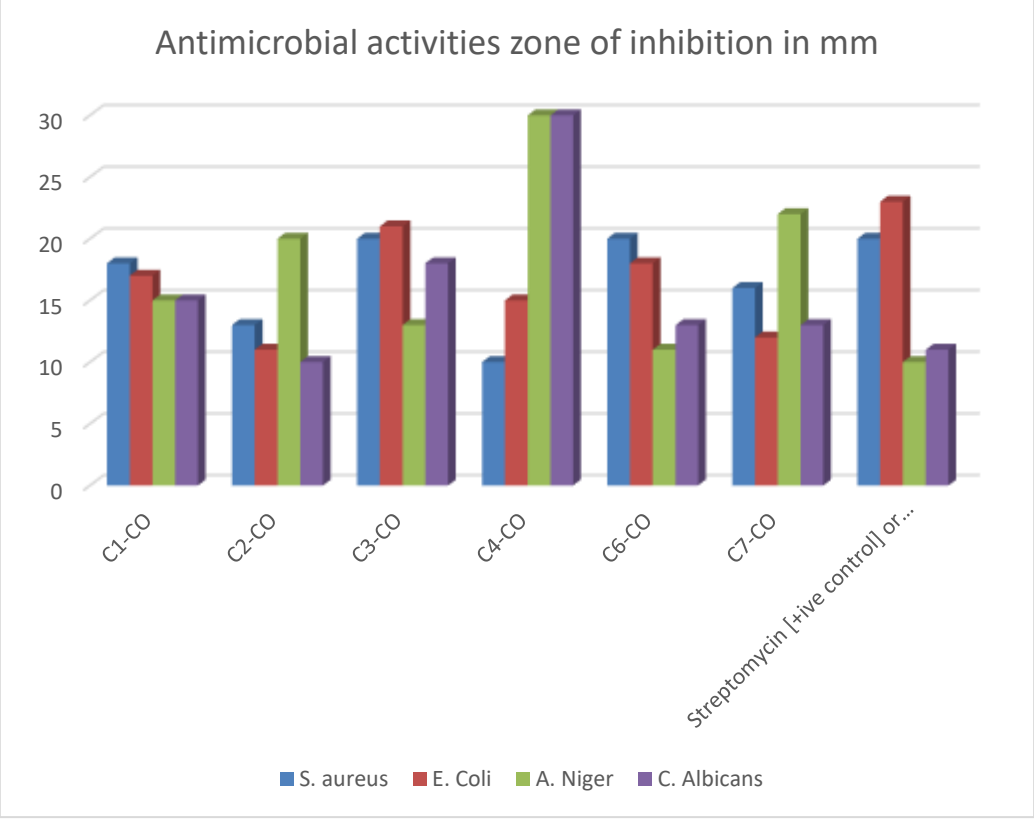
As per shown in table no 2 the synthesized Co Schiffbase metal Complexes are tested against the Bacteria S. Aurius, E. Coli, and Fugi A. Niger and C. Albicans. The Streptomycin [+ive control] was used as

standard for antibacterial study. The amphotericin B was used as standard for study of antifungal activity as shown in figure no 3 [28].

Table no 2 Antimicrobial Testing of synthesized Co Schiffbase metal Complex [28,29]

	zone of Inhibition			
Sample ID	*S. aureus*	*E. Coli*	A. Niger	C. Albicans
C1-CO	18	17	15	15
C2-CO	13	11	20	10
C3-CO	20	21	13	18
C4-CO	10	15	30	30
C6-CO	20	18	11	13
C7-CO	16	12	22	13
e control] (	20	23	10	11

Fig no 3 Antimicrobial activities zone of inhibition in mm



3.2 Spectroscopic Analysis Schiff base and Schiff base metal Complexes[30,31]:

The IR and H1 NMR spectra are given in following figures.

Figure no 4 IR spectra of Schiff base (E)-N-(4-methoxybenzelidine) aniline

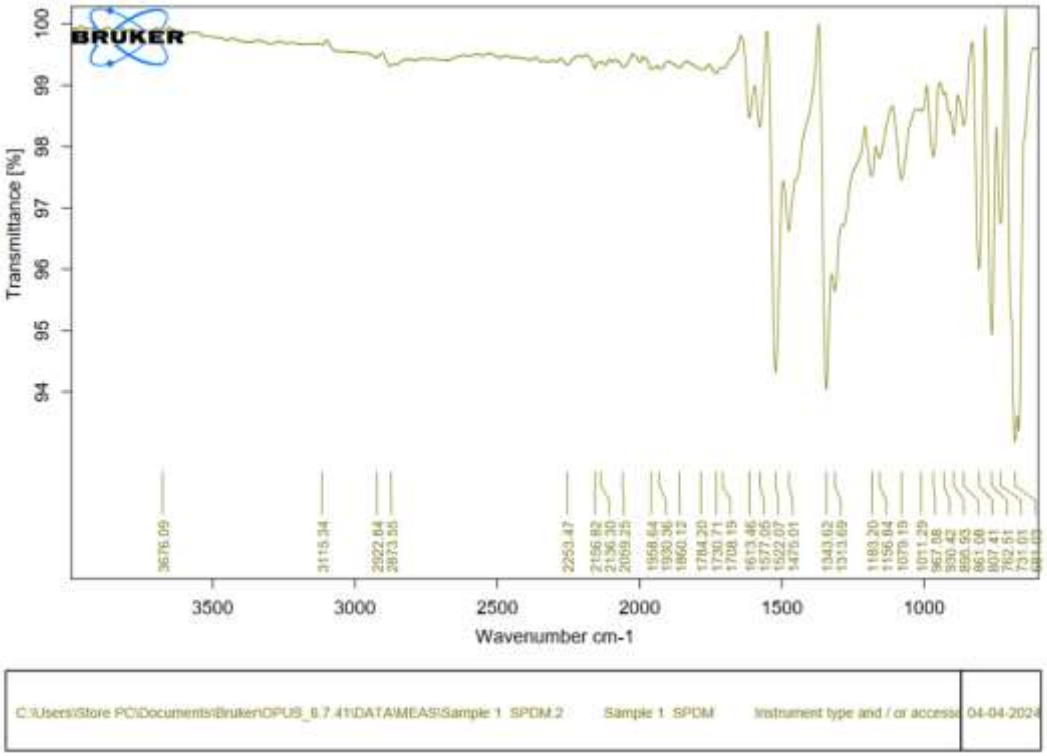


Figure no 5: IR spectra of Schiff base (E)-N-(3nitrobezylidene) Benzenamine:

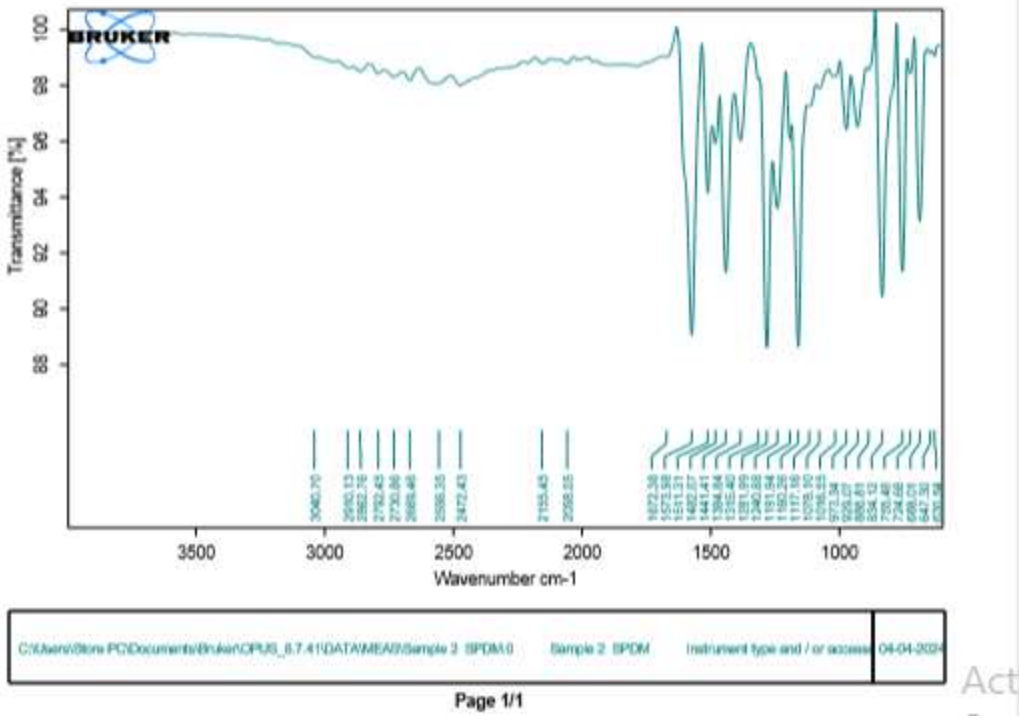


Figure no 6: IR spectra of Schiff base (E)-N-(benzelidine) aniline

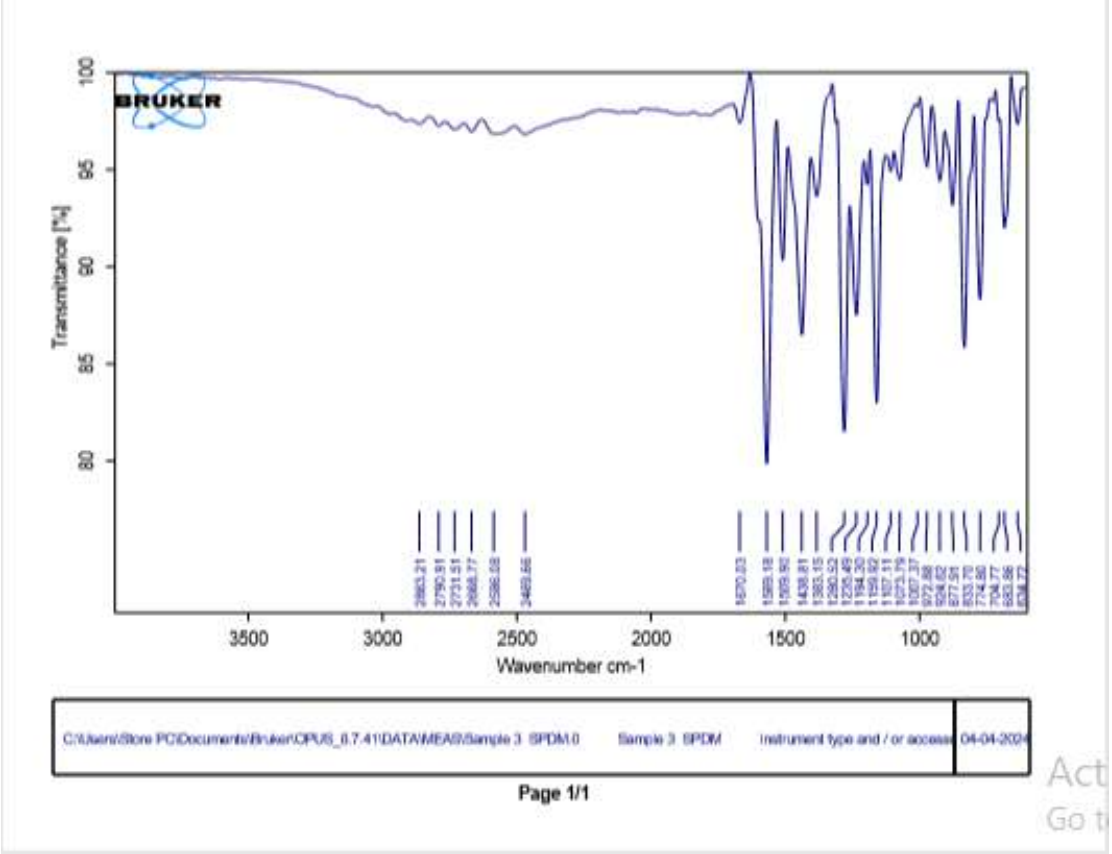


Figure no 7 The H^1 NMR spectra of Schiff base Metal Complex [Co((E)-N-(4-methoxybenzelidine) aniline)2 .Cl2.2H2O]

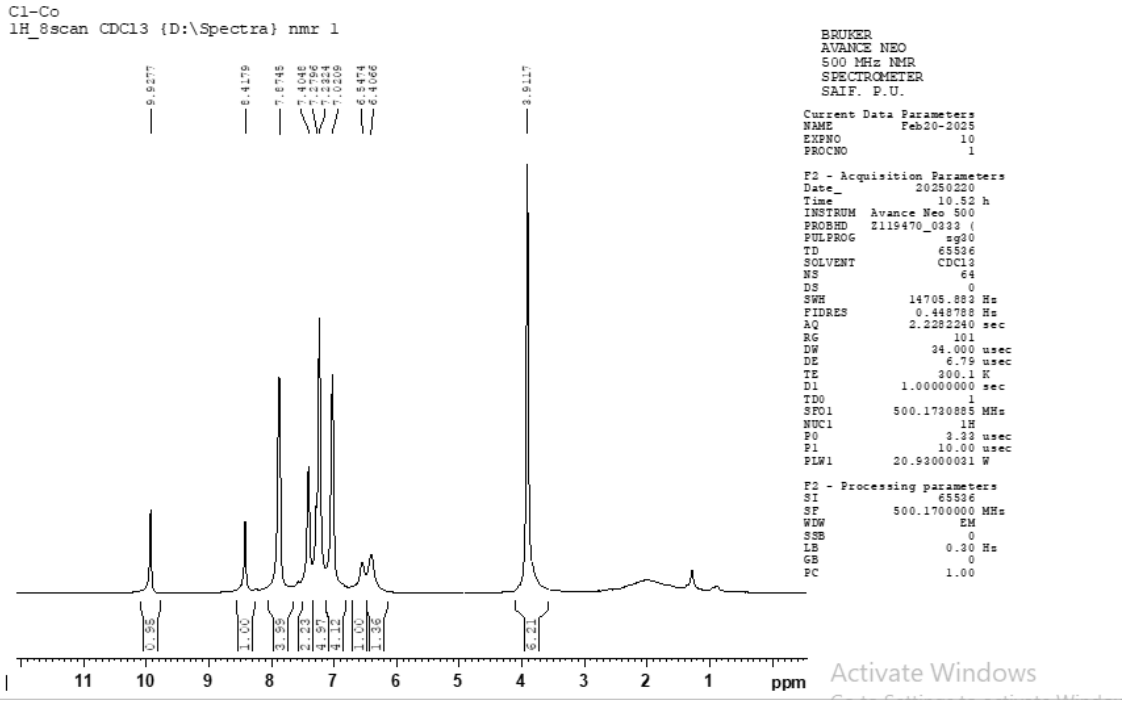


Figure no 8: The H^1 NMR spectra of Schiff base Metal Complex [Co((E)-N-(3nitrobezylidene) (benzenamine)2 .$Cl_2.2H_2O$]

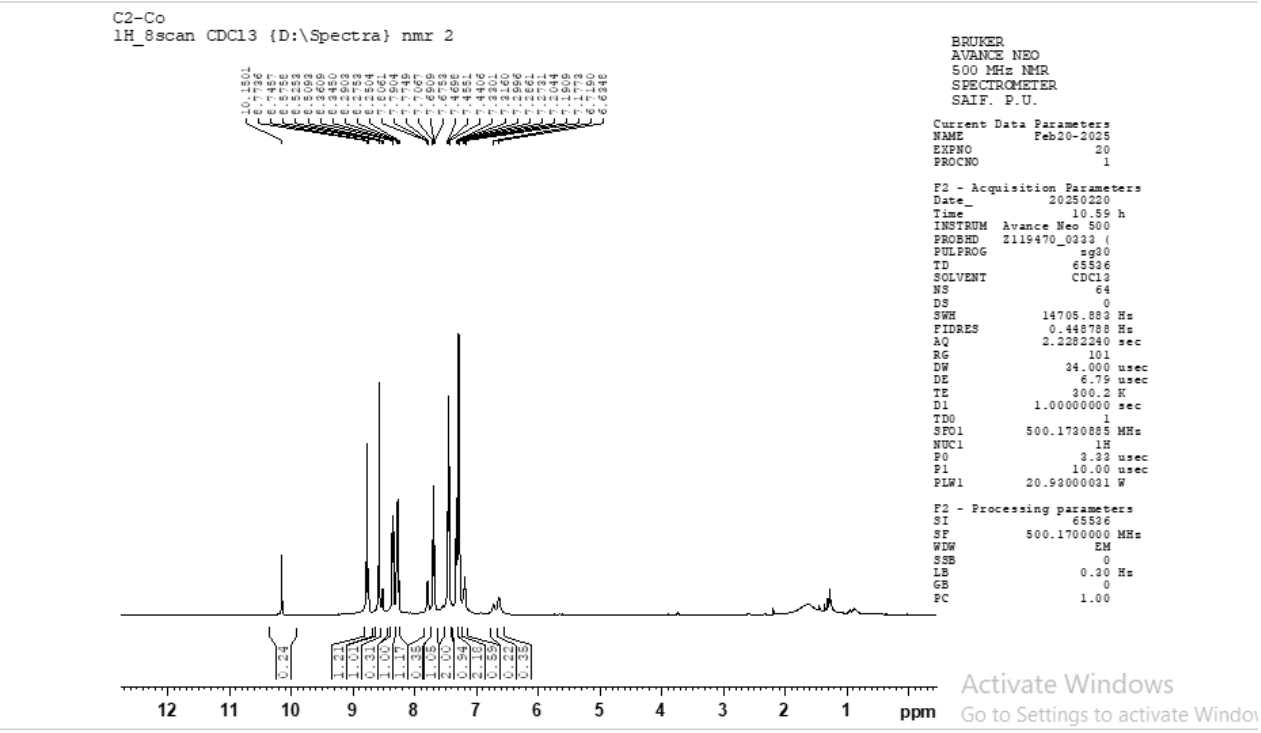


Figure no 9: The H^1 NMR spectra of Schiff base Metal Complex [Co((E)-4-((phenylimino)methyl)phenol)$_2$.$Cl_2.2H_2O$]

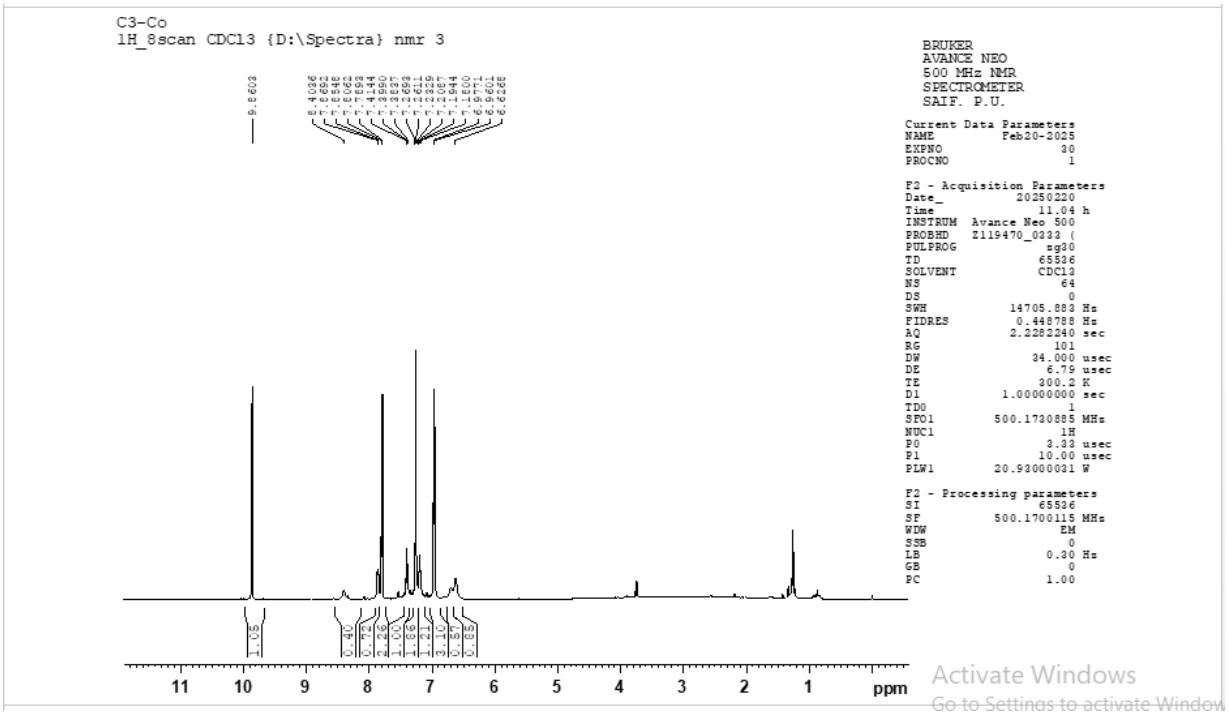
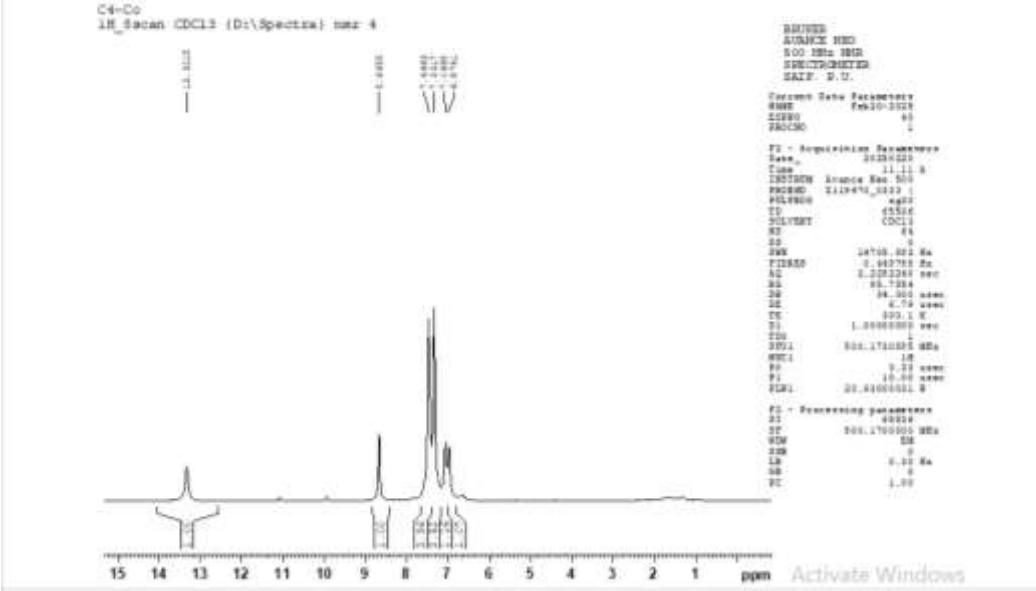


Figure no 10: The H1 NMR spectra of Schiff base Metal Complex [Co((E)-2-(phenylimino)methyl)phenol)$_2$ .Cl$_2$.2H$_2$O]



4.0: Discussion

The IR data of the spectra of Schiff base ligands (L1 and HL2) and their complexes are presented in figure 4,5 and 6. The IR spectra of the complexes were compared with those of the free ligands in order to determine the coordination sites that involved in chelation [38]. There were some guide peaks in the spectra of the ligands, which were helpful in achieving this goal. The position and/or the intensities of these peaks are expected to change upon chelation. New peaks are also guide peaks, as is water, in chelation. These guide peaks are shown [39]. Upon comparison, it was determined that the υ(C=N) stretching vibration is found in the free ligands at 1614 and 1659 cm−1 for the L1 and HL2ligands, respectively. This band was shifted to higher or lower wavenumbers in the complexes, indicating the participation of the azomethine nitrogen in coordination (M—N) 30. The SH stretching vibration, υ(SH), is not useful, since it displayed very weak bands in both the free HL2ligand and complexes spectra.

1HNMRspectra

A review of the literature revealed that NMR spectroscopy has been proven to be useful in establishing the nature and structure of many Schiff bases[33] are shown in figure no 7, 8, 9 and10, as well as their complexes in solutions. The NMR spectra of Schiff bases were recorded in d6-dimethylsulfoxide (DMSO) solution, using tetramethylsilane (TMS) as internal standard.

Microbial Activity:

According to the results of the antibacterial activity screening shown in table no 2, the Co(II) complexes possess effective and selective antibacterial activity against 1 gram-positive bacteria *Stephyllococcus* spore-forming zone of inhibition , 1 gram-negative bacterium E. Coli forming zone of inhition. Co-Schiff base complexs shown highly potent inhition activity against gram positive as well as gram negative bacteria is clearly shown in figure no 3. The Streptomycin [+ive control] was used as standard for antibacterial study. The antifungal activities of the Co-complexes of compounds against 2 yeast strains A. niger and C. albanicans. The Co-Schiffbase Complexes shown potent inhibition again fungi. The amphotericin B was used as standard for study of antifungal activity [40]. [Co((E)-N-(4-

methoxybenzelidine) aniline)2 .Cl2.2H2O] formed 18mm of zone of inhibition against S. aurius, 17mm against E. coli, 15mm against A. niger 15C. albanicans. [Co((E)-N-(3nitrobezylidene) benzenamine)2 .Cl2.2H2O] formed 13mm 11mm 20mm 10mm of zone of inhibition against S. aureus, E. coli, A. Niger, C.albicans respectively. [Co((E)-4-((phenylimino)methyl)phenol)2 .$Cl_2.2H_2O$] 20mm, 21mm, 13mm, 18mm of zone of inhibition against S. aureus, E. coli, A. Niger, C.albicans respectively.
[Co((E)-2-(phenylimino)methyl)phenol)2 .$Cl_2.2H_2O$] formed 10mm 15mm, 30mm, 30mm of zone of inhibition against S. aureus, E. coli, A. Niger, C.albicans respectively.
[Co((E)-N-(4-chlorobenzylidene)-3-benzenamine)2 .$Cl_2.2H_2O$] formed 20mm,18mm,11mm, 13mm of zone of inhibition against S. aureus, E. coli, A. Niger, C.albicans respectively. [Co((E)-N-(4-Nitrobenzylidene)-3-benzenamine)2 .$Cl_2.2H_2O$] has formed 16mm, 12mm, 22mm, 13mm of inhibition against S. aureus, E. coli, A. Niger, C.albicans respectively.

5.0 Conclusion:

Co-Schiffbase metal complexes formed by using eco-friendly and chip catalyst. This catalyst is easily available. The Co-Schiffbase metal complexes formed was charecterised and identified by spectroscopy. On the microbial testing these metal complexes shown highly potent antimicrobial activity. In [Co((E)-N-(4-chlorobenzylidene)-3-benzenamine)2 .$Cl_2.2H_2O$] formed 20mm,18mm,11mm, 13mm of zone of inhibition against S. aureus, E. coli, A. Niger, C.albicans respectively. It is more potent than standard used for testing.

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6.0: References:

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