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Research Article

SYNTHESIS OF SALICYLALDEHYDE BASED SCHIFF BASES AND THEIR METAL COMPLEXES IN AQUEOUS MEDIA -CHARACTERIZATION AND ANTIBACTERIAL STUDY

Shamly. P[1]., Mary Priya Nelson[1]., Anitta Antony[2] and Jaya T. Varkey[1]*

[1]Department of Chemistry, St.Teresa's College, Ernakulam, 682035, Kochi
[2]Department of Chemistry, St.Albert's college, Ernakulam, 682018, Kochi

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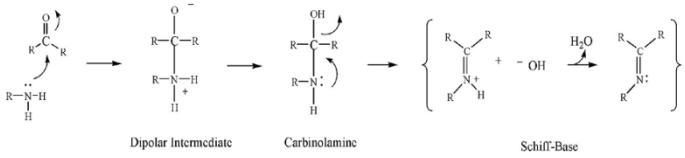
ABSTRACT

A new, efficient and environmental friendly procedure for the synthesis of a series of salicylaldehyde-based Schiff bases and their metal complexes in aqueous media were conducted. The work involved the condensation of salicylaldehyde with various amines, both aromatic and aliphatic and the Schiff bases formed were complexed with a transition metal Ni and an alkaline earth metal Mg. This green synthetic approach was compared with conventional procedure and found to have advantages like good yield and reduction in reaction time and byproducts. The synthesized Schiff bases and their complexes were characterized by FTIR. The antibacterial activity of complexed and uncomplexed Schiff bases were compared against Escherichia coli and the Mg complex is found to have better antibacterial activity than the corresponding Ni complex.

INTRODUCTION

The recent interest in green chemistry has posed a new challenge for organic synthesis in that new reaction condition need to be found which reduce the emission of volatile organic solvents and the use of hazardous toxic chemicals. The use of water as the reaction medium offers several advantages as it is cheap, non-inflammable, non toxic and safe to use. Its unique physical and chemical properties often increase the reactivity or selectivity, which sometimes is unattainable in organic solvents and it eliminates the additional effort required to make the substrate dry before use and thus reduce the consumption of drying agents, energy and time.[1]

A Schiff base is a nitrogen analogue of an aldehyde or ketone in which the C=O group is replaced by C=N-R group. It is usually formed by the condensation of an aldehyde or ketone with a primary amine[2]. In general mechanism (Scheme 1) of synthesis of Schiff bases, an amine reacts with a carbonyl compound by nucleophilic addition. This forms a hemiaminal, followed by a dehydration to produce imines[3, 4]. Schiff bases have a large number of synthetic uses in organic chemistry. Schiff bases appear to be an important intermediate in a number of enzymatic reactions[5].



Scheme 1 Mechanism of formation of Schiff base

Schiff base complexes have attracted wide attention due to their important role in analytical chemistry, organic synthesis, refining of metals, electroplating and photography[6]. The nature of metal ion determines the properties of the complexes. Schiff bases can acts as a potential site for biologically active compounds. Various transition and inner transition metals form complexes with bi, tri and tetra dentate Schiff bases containing nitrogen and oxygen donor atoms [7, 8]. Several studies have revealed that by condensation of salicylaldehyde with different primary amines and their metal complexes showed potential antibacterial activity. The antibacterial potency of Schiff bases increased upon chelation/complexation against tested bacterial stain.

It is worthwhile to mention here that the synthesis of these Schiff bases known in the literature involves tedious process using methanol/ethanol as solvent and requires large reaction

Corresponding author:* **Jaya T. Varkey
Department of Chemistry, St.Teresa's College, Ernakulam, 682035, Kochi

time. We here report a green and efficient method for the synthesis of Schiff bases in aqueous media. The easy workup, facile conditions, fast reaction rate and good yield of the reaction makes the present methodology attractive.

Experimental

Materials

Chemicals like salicylaldehyde, aniline, ethylene diamine and 3-aminobenzoic acid was provided by Sigma Aldrich Company and they were used as supplied. All metal (II) salts were used as chlorides. The FTIR spectra were recorded on a Bruker IFS-55 spectrometer using KBr pellets.

Methods

Synthesis of salicylaldehyde-based Schiff bases: Schiff bases were prepared by condensation of 0.01mol of amines; 3-amino benzoic acid, aniline, ethylene diamine in 10 ml of water and 0.01mol of salicylaldehyde. The mixture was stirred at room temperature for 10 min. The yellow precipitate formed was filtered, washed with water and dried[9].

Synthesis of metal complexes of Schiff bases: Magnesium and Nickel were used as metals for derivation of metal complexes. Warm ethanolic solutions of Schiff bases were added to magnetically stirred solution of metal salt in distilled water. The mixture was refluxed for 1 hour and cooled to room temperature. On cooling, schiff base metal complex was precipitated, filtered, washed with water and dried[10].

Analysis

FTIR analysis: Fourier Transformation Infrared Spectrophotometer (FT-IR) analysis of each schiff base and its metal complexes were done to confirm the formation of metal complexes.

Antimicrobial study: Antimicrobial substance kills or prevents the growth of microorganisms and metal complexes showed very good antimicrobial activity. E. coli is a gram-negative bacterium found in the lower intestine of humans. In the present work antimicrobial activity of synthesized Schiff base ligands and its metal complexes were studied against E. coli bacterium. The bacterial culture was incubated at 30±0.1°C for 24 hours by incubation into nutrient agar. Gram negative microbe *E.coli* was selected for study. For assay, solution with desired concentration of Schiff base with metal complexes were incubated with plates containing microbes and zone of inhibition was observed after 24 hours[10].

RESULTS AND DISCUSSION

The solid Schiff base metal complexes are intensely coloured, insoluble in water, but soluble in methanol, ethanol and THF.

IR Spectra: The IR spectra of the Schiff bases showed a strong band in the region 1580-1680 cm^{-1}, which is characteristic of azomethine (stretching frequency C=N) group. In all complexes, this band is slightly shifted to lower frequencies indicating coordination of the Schiff base through azomethine nitrogen atom. The IR spectrum of ligands exhibited a broad band of stretching frequency of OH group at 3369-3445 cm^{-1}. These peaks were absent in the IR spectra of metal complexes. In the case of complexes, the bands in the region 650-680 cm^{-1} and 440-490 cm^{-1} are attributed to ν (M─O) and ν (M─N) stretching vibrations respectively, conforming coordination of Schiff base to metal ions (Table 1) (Figures 1-9).

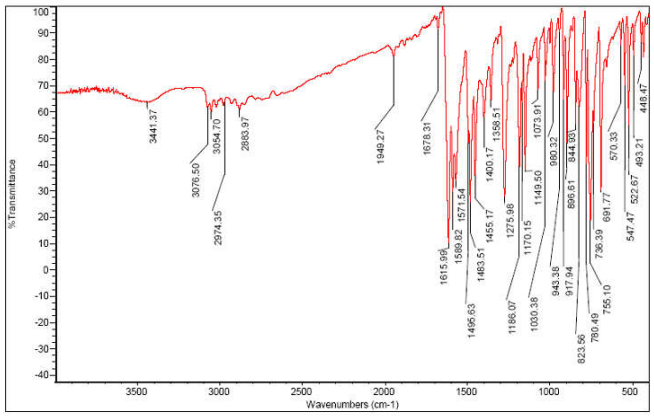


Figure 1 IR spectra of salicylaldehyde – aniline Schiff base

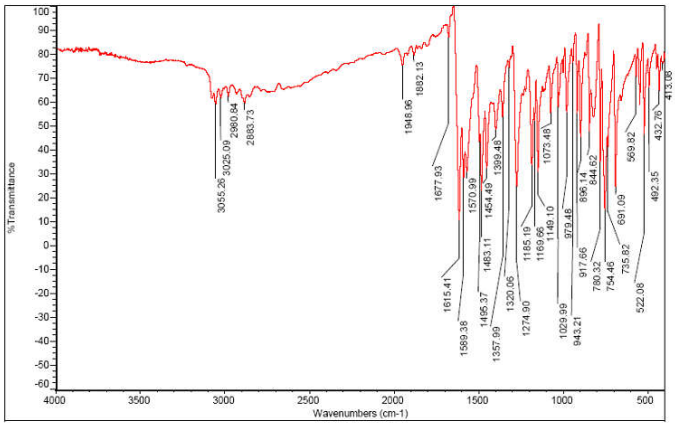


Figure 2 IR spectra of salicylaldehyde – aniline Schiff base Mg complex

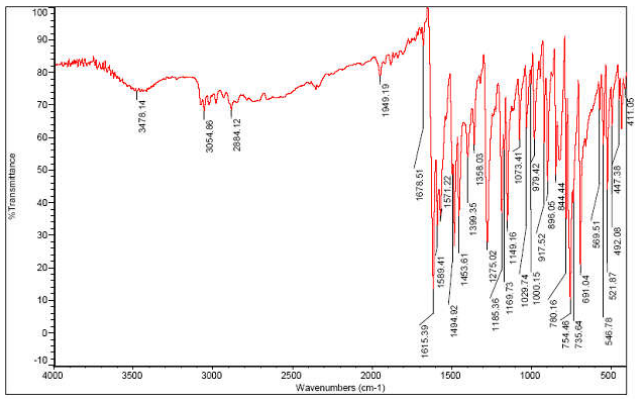


Figure 3 IR spectra of salicylaldehyde – aniline Schiff base Ni complex

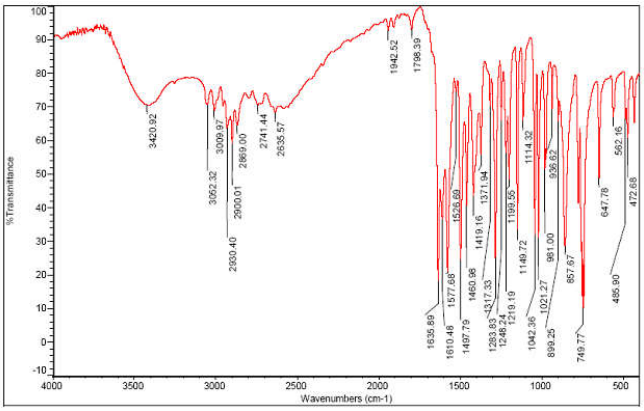


Figure 4 IR spectra of salicylaldehyde – ethylene diamine Schiff base

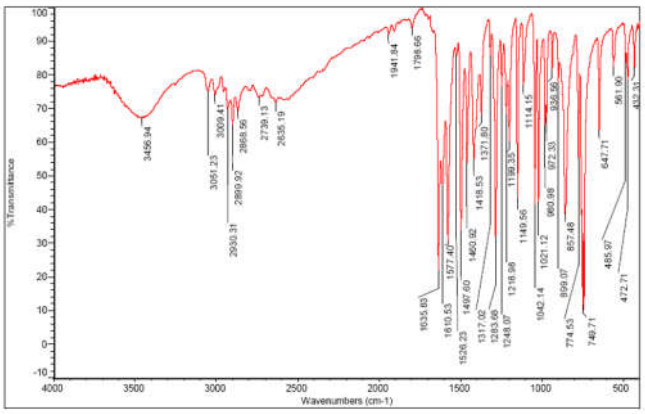


Figure 5 IR spectra of salicylaldehyde – ethylene diamine Schiff base Mg complex

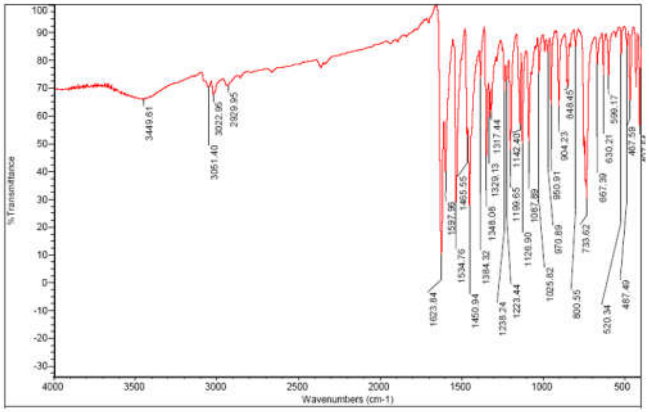


Figure 6 IR spectra of salicylaldehyde – ethylene diamine Schiff base Ni complex

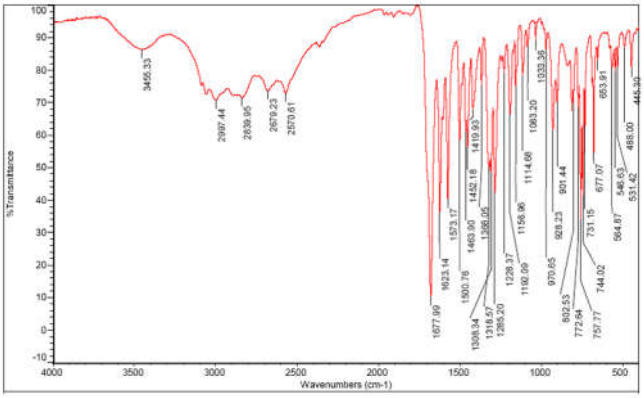


Figure 7 IR spectra of salicylaldehyde – 3-aminobenzoic acid Schiff base

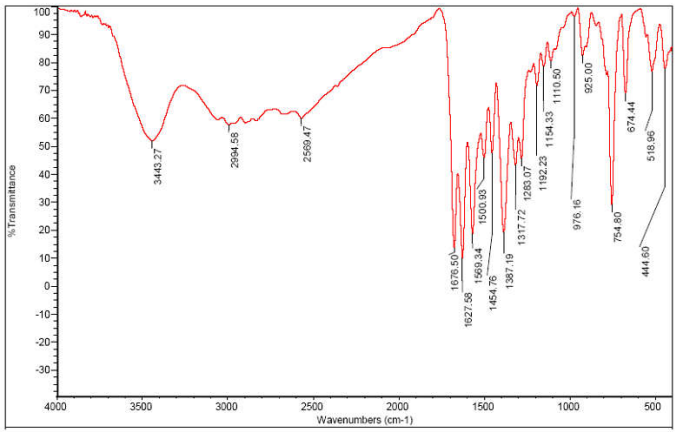


Figure 8 IR spectra of salicylaldehyde – 3-aminobenzoic acid Mg complex

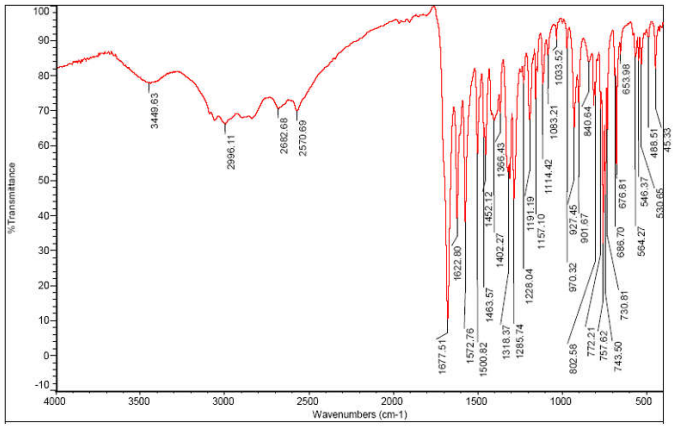


Figure 9 IR spectra of salicylaldehyde – 3-aminobenzoic acid Ni complex

Table 1 IR spectra of Schiff bases and corresponding metal complexes

Compound	ν (C=N) c.m^{-1}	ν (O–H) c.m^{-1} (stretching)	ν (M–O) c.m^{-1}	ν (M–N) c.m^{-1}
Salicylaldehyde -aniline Schiff base	1678.31	3441.37		
Salicylaldehyde -aniline Schiff base Mg Complex	1677.93		691.09	413.08
Salicylaldehyde-aniline Schiff base Ni Complex	1678.51	3478.14	691.04	411.05
Salicylaldehyde-ethyelenediamine Schiff base	1635.89	3420.92		
Salicylaldehyde-ethylenediamine Schiff base Mg complex	1635.83	3456.94	647.71	432.31
Salicylaldehyde-ethyelediamine Schiff base Ni complex	1623.84	3449.61	630.21	407.54
Salicylaldehyde-3-aminobenzoic acid Schiff base	1677.99	3455.33		
Salicylaldehyde-3-aminobenzoic acid Schiff base Mg complex	1676.50	3443.27	674.44	444.60
Salicylaldehyde -3-aminobenzoic acid Schiff base Ni complex	1677.51	3449.63	676.81	488.51

Antimicrobial activity: The sensitivity of gram negative bacteria *E.coli,* towards the Schiff base and their complexes were analyzed by measuring the size of their zone inhibition. The coordination of metal ions with bulky Schiff bases through chelation decreases the polarity of the metal ion. This is due to the overlap of the ligand orbital and partial sharing of the positive charge of the metal ion with donor groups. It also increases the delocalization of Π electrons over the whole chelate ring. This enhances the lipophilic nature of complexes. The lipid membranes lipophilicity is an important factor in determining antibacterial activity. Lipid membrane of cell favors the passage of only lipid soluble materials. This increased lipophilicity enhances the penetration of the complexes into lipid membrane and thus blocks the metal binding site on enzymes of microorganisms. The metal complexes also disturb the respiration of cell and inhibit cell growth. The antimicrobial potency of metal complexes depends on the impermeability of the cells of the microbes. The results indicate that the Mg complex shows highest activity against

E.coli than Ni complex. The antimicrobial studies were shown in Images 7 and 8 and the results were given in Tables 2-4.

Table 2 Antibacterial activity of Salicylaldehyde-Aniline Schiff base and its metal complexes

Sl. No.	Organism	Chemical	Zone Size
1	*E.coli*	Salicylaldehyde-aniline Schiff base	4mm
2	*E.coli*	Salicylaldehyde-aniline Schiff base Mg complex	14 mm
3	*E.coli*	Salicylaldehyde-aniline Schiff base Ni complex	7 mm
4		Solvent control-DMSO	Negative

Table 3 Antibacterial activity of Salicylaldehyde-ethylenediamine Schiff base and metal complexes

Sl. No.	Organism	Chemical	Zone Size
5	*E.coli*	Salicylaldehyde-ethylene diamine Schiff base	Negative
6	*E.coli*	Salicylaldehyde-ethylene diamine Schiff base Mg complex	Negative
7	*E.coli*	Salicylaldehyde-ethylene diamine Schiff base Ni complex	Negative
8		Solvent control-DMSO	Negative

Table 4 Antibacterial activity of Salicylaldehyde-3-aminobenzoic acid Schiff base and metal complexes

Sl. No.	Organism	Chemical	Zone Size
9	*E. coli*	Salicylaldehyde-3-aminobenzoic acid Schiff base	11mm
10	*E. coli*	Salicylaldehyde-3-aminobenzoic acid Schiff base Ni complex	20mm
11	*E. coli*	Salicylaldehyde-3-aminobenzoic acid Schiff base Mg complex	22mm
12		Positive control	18mm

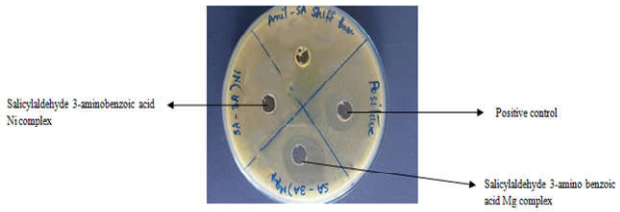


Image 1 Zone inhibition against *E. coli* bacteria with positive control

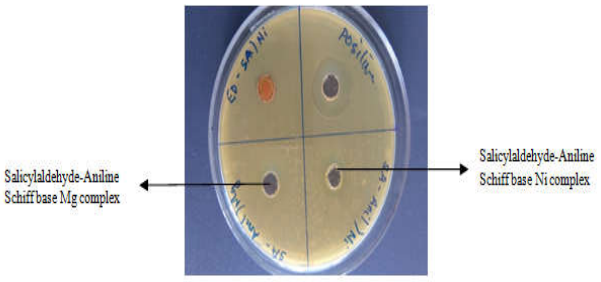
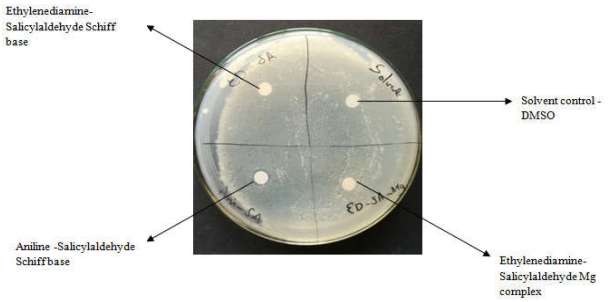


Image 2 Zone inhibition against *E. coli* bacteria with positive control



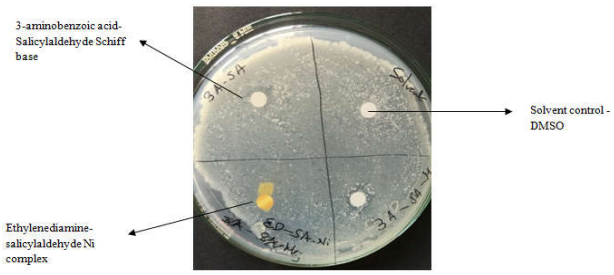


Image 3 Zone inhibition against *E. coli* bacteria with solvent control

CONCLUSION

The method shown here is most convenient way to synthesize the salicylaldimines, in which water plays the role of eco friendly solvent. The synthesis using water as the reaction medium offers advantages over conventional methods like good yield, reduction in reaction time and byproducts. The synthesized ligands were successfully complexed with the metal ions Mg (II) and Ni (II). FTIR Spectroscopic analysis have indicated the successful formation of salicylaldehyde based Schiff bases and their complexes. Based on antibacterial study, it was found that the metal complexes of Schiff bases are more efficient antimicrobial agents than its native form. Thus metal complexes of these Schiff bases could find potential applications in designing new therapeutic agents. However, the antimicrobial potentiality of metal complexes is highly dependent of the metal ions used for complex formation. Results of antimicrobial activity reflected that the Schiff base-metal complex of Mg has more antimicrobial activity than Ni complex. They can also be used in water purification to kill or inhibit the growth of water borne bacteria.

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Conflict of interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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