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Synthesis, Spectral Characterization And Antimicrobial Studies Of Metal Complexes Of The Schiff Base Ligand Derived From 2,4 Dihydroxy-5-Acetyl Acetophenone - 1,4 Diaminobutane(Dhadab)

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Abstract
Under the biological domain of chemistry Schiff base has a wide variety of applications like it appropriately and significantly utilised in anti-malarial, anti-inflammatory, antiviral, antifungal, antibacterial characteristics. And brought phenomena these are called as antimicrobial properties or activities of Schiff base. By utilising the complexion of Schiff base with metals usually improves and intensifies antibacterial and antifungal impact when compared with solely Schiff base. One of the most critical antimicrobial activities performed by Schiff base is its working in amine or azomethine groups. Presently, there is very limited research that was concentrated on metal compounds of Schiff base formulated with "2,4-dihydroxy -5-acetylacetophenone and 1,4 diaminobutane (DHADAB)". Henceforth the motive of the research is to comprehensively explore DHADAB and its synthesis with various metallic complexes that have antimicrobial qualities such as Cobalt, Manganese, Copper, and Zinc, among others. The finding shows that the "copper(II) acetate monohydrate [Co(II)] are highly active against *Staphylococcus aureus* as compared with the cobalt(II) acetate tetrahydrate [Co(II)], zinc(II) acetate dihydrate [Zn(II)] cobalt(II) acetate tetrahydrate [Co(II)], and anhydrous ferric chloride (III) [Fe(III)]" which also demonstrate effective activity against *Escherichia coli.* The study recommends to utilise advanced functioning of Schiff bases such as chitosan-based Schiff bases and nano-particles operationalised with Schiff bases as it has excellent possibility to overcome the challenges and provide suitable approach for identifying heavy metal ions that act as a promising ingredient for fighting antimicrobial resistance challenges

Keyword- 2,4-dihydroxy -5-acetylacetophenone and 1,4 diaminobutane (DHADAB), Antimicrobial activities, Schiff Base, Synthesis, spectral characterization and antimicrobial studies.

Introduction

Schiff base is proposed by Hugo Schiff, is a structure (shown in figure 1) of nitrogen analogous which is also known as azomethine, is an analogous of aldehyde or ketone in carbonyl group
($C = O$) and appropriately substituted by imine group [1]. Schiff base being a prominent class of ligands which are widely utilised in the formation of organic compounds in coordination chemistry in the form of chelating ligands [3]. It has applications in other domains like biological, clinical, and analytical chemistry. They are significantly utilised in catalyst, organic synthesis, polymer stabilisers, pigment and dyes and so on. Because of their characteristic qualities like anti-fungal, anti-bacterial, anti-inflammatory, and anti-viral features Schiff base is utilised in the biological domain [2]. The imine group is adequately available in organic, in-organic and natural derivatives. The application of Schiff base complexes in the form of organic catalysts obtained from distinct natural reactions has gained enormous interest in recent times.

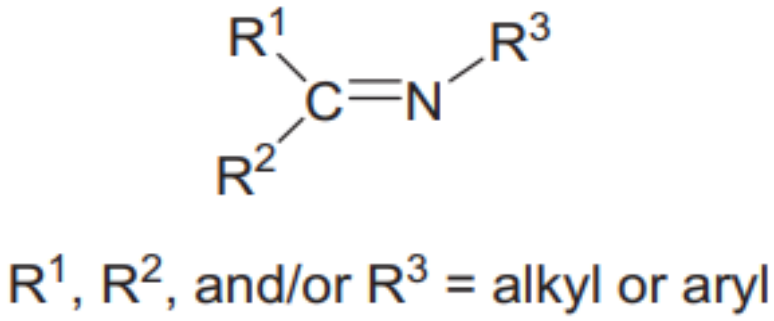

Figure 1: Basic structure of Schiff base [1]

The intramolecular hydrogen bonding between (OH) Hydroxide/base group and C=N nitrogen atom of Schiff base identifies the characteristic of several molecular structures and performs a vital role in distinct biochemical phenomena [5]. The linking between carbon and nitrogen bonds that form as azomethine derivatives play a vital role for biological activities. Schiff base comprehensively functions as ligand in coordination chemistry prominently due to its tremendous donor capability and chelating agents. The metallic complexes that are formed by Schiff base have several industrial functions specifically in the segment of catalysis, dying, and analytical reagent [6]. Schiff bases are considered as opportunities for antibacterial agents. The impact of metals that take place on the biological functioning of elements and their intrinsic chemical activity like multidentate ligands has encouraged research in this theme coordination attribute [4].

Several literature work and existence studies focused on Schiff base complexes with first order of transition series covering metal ions because of their excellent pharmacological utility and appropriate physiological function [6]. Although very limited existing research was concentrated on metal compounds of Schiff base formulated with "2,4-dihydroxy -5-acetylacetophenone and 1,4 diaminobutane (DHADAB)". Henceforth the motive of the research is to comprehensively explore DHADAB and its synthesis with various metallic complexes that have antimicrobial qualities such as Cobalt, Nickel, Manganese, Copper, and Zinc, among others. The metals that have antimicrobial qualities are also known as a "series of polymeric complexes like Mn(II), Co(II), Fe(II), Cu(II), and Zn(II)" [7].

Physical Measurement

The study analysed the elements like Carbon (C), Hydrogen (H), Nitrogen (N) and collected the sample from Central Drug Research Institute (CDRI), Lucknow, India. The metallic element then decomposes by using concentration H_2SO_4 and HNO_3. Magnetic susceptibility analysis is conducted based on Gouy's Balance in normal heat with "$Hg[Co(SCN)_4]$ as calibrant and diamagnetic correction in the Pascal's constant". Indian Institute of Technology of Chennai (SAIF) tool uses diffuse reflectance spectra were observed on varian cary-5E UV-visible spectrophotometer. "1_H NMR spectra of ligand (CDCL3+DMSO) was recorded on NMR-JEOL, GSX-400 spectrophotometer and chemical shifts are illustrated in ppm relative to tetramethylsilane". "Thermogravimetric analysis (TGA)" of the prepared components need to be processed on the "TGS-2 Perkin-Elmer caloric profiler" in surrounding air with a warm situation of ten celsius per minute. The ligands and their metallic components were a concealment

for “in vitro antimicrobial functioning confronting the E.Coli, S.Typhi, P. aeruginosa and S.aureus” by cup saucer approach [8].

Experiment

The chemical components were selected based on analytical grade and the diluent was dehydrated and sublimated before utilising it as it fulfilled the guideline and the standard mentioned in the procedural activity [9].

Material

The material utilised for processing all the chemicals metallic ingredient and solvent were selected based on the assessment grading approach.

“Vanadyl (IV) sulphate pentahydrate, anhydrous ferric chloride (III), cobalt(II) acetate tetrahydrate, copper(II) acetate monohydrate, and zinc(II) acetate dihydrate” .

These components were collected and obtained from the laboratory S.D fine limited, providing its services in India. Components are formulated by the knowing procedure with minute alternation and slide changes.

Synthesis of Schiff base ligand (DHADAB)

The Schiff base ligand can be consolidation into two distinct segment, the initial segment comprising the formulation of “2,4- dihydroxy - 5-acetyl acetophenone (DHA)” take place and in the next segment condensation procedure was followed in which formation 1,4 –diaminobutane is processed.

First Segment : “Preparation of 2,4- dihydroxy - 5-acetyl acetophenone (DHA)”

To ready the Schiff base depends on the usual procedure of condensation of the components “5-acetyl-2, 4- dihydroxyacetophenone with 1, 4-diaminobutane”.

The preparedness of Acetophenone was accomplished by a procedure called refluxing in which resorcinol around five grams (0.05 mol) and acetic anhydride about nine millilitre (0.05mol) was taken. All the elements are collected in a circular bottom flask and put for forty-five minutes in the availability of fused zinc chloride of seven grams. The obtained composition of the ingredient was kept at a normal room temperature under endothermic procedure, and afterward utilised to prepare a vigorous shaking by using crushed ice. Once, a procedure was completed a reddish brown precipitate was segregated and after percolating the solvent in a bowl, boiling the bowl under water was performed for approximately fifteen minutes. The obtained solution was then permeated in hot temperatures and the prepared solution was cleaned with warm water again, interbedded from sublimate ethanol and dehydrated in vacuo.

Second Segment: “Condensation of the 2, 4-dihydroxy-5-acetylacetophenone with 1,4 – diaminobutane (DHADAB)”:

For the condensation of relevant components, the methodology that based on dropping dissolving method was used in which the substance “1,4 -diaminobutane (0.05 mol)” was added in a twenty-five mililitre ethanol solution “2,4- dihydroxy - 5-acetyl acetophenone (0.05 mol)” in a drop - drop pattern in a circular bottom flask and there is a need for continuous steering of the solution. One thing should be noted that the solution of the obtained mixture was heated for about 3 to 4 hours under a water bath and kept overnight at a normal temperature. Afterwards a coloured element that is a yellowish brown solution was prepared. The solution was filtered and washed under the ethanol component (1:1 v/v) and dehydrated in the normal temperature in order to obtain extracted crystallised solution from ethanol “{yield 65% m.p. 1700 C}”.

[1]HNMR of DHADAB

"The [1]HNMR spectrum of ligands exhibits a signal at δ 8.00 due to imine proton. The aromatic protons are observed in the range δ 7.35 - 7.65 (2H, s, phenyl), the phenolic OH appeared at δ 12.20 (2H, S, Phenolic OH), δ 3.10 (4H, t, N, CH2), δ 1.90 (4H, m, CH2), δ 2.6 (6H, s, methyl)."

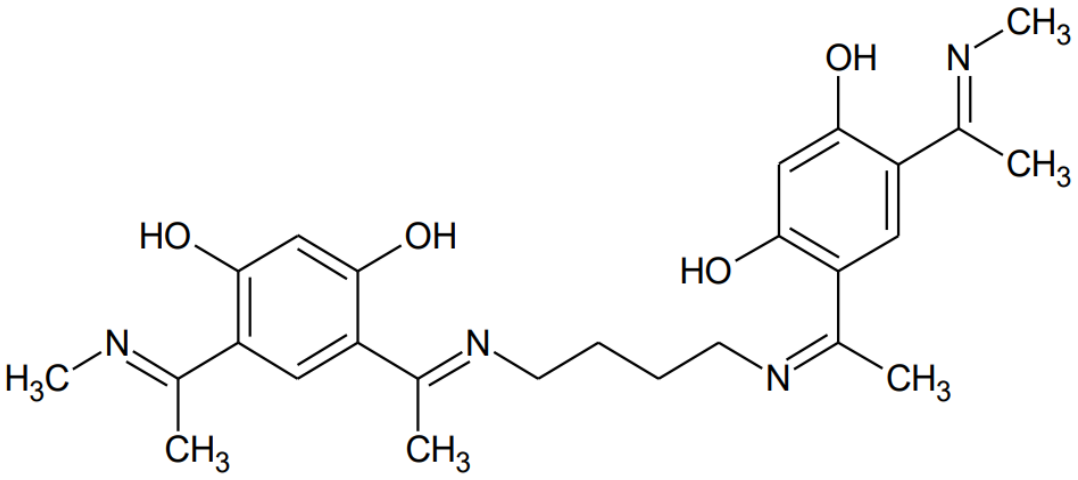

Fig 2: Structure of Schiff base ligand (DHADAB)

Synthesis of the Compound

All the metallic components are designed by the mentioned approach comprising equimolar quantities of adequate metallic salt (1.0 mmol) and ligand (1.0 mmol) were dissolved distinctly in a solution of 25 to 30 ml DMF which is assumed to be minimum quantity. All the solutions were after dissolved go through the filtration face and then combined in heat in a continuous stirring. The prepared solution was then to go through the refluxed procedure where the mixture went through a sand bath for about three to four hour. The obtaining solution is having distinct colour which would again go through the filtration procedure and wash multiple times with a DMF solution that was formed by acetone in order to separate unreacted ligand and metallic salt. Eventually the component was dehydrated in the vacuum over anhydrous CaCl2. Yield: sixty to seventy percent.

Result and Discussion

When Schiff base is compounded with distinct metal and forms a complex, it offers higher functioning in comparability to Schiff base alone. Because the availability of unpaired negative ions of nitrogen elements that are available in the azomethine produce a robust chelating capability to Schiff base [10]. Schiff bases are vitally considered interesting ligands for integrated chemistry as it holds a normal capability because of chelating ligands activity that provide it resilience and convenient approach to offer distinguishability and flexibility on the group.

One of the most challenging elements in biological chemistry was antibiotic resistance has enhanced in recent years and led to decline in the efficiency of rarely available antibiotics [11]. By utilising the complexion of Schiff base with metals usually improves and intensifies antibacterial and antifungal impact when compared with solely Schiff base [12, 13]. For instance, when Schiff base is compiled and

forms a complex with copper it produces a green cellulose based antibacterial ingredient which shows *E. Coli* and *S. Aureus* improved and intensified by 472% and 823% respectively of Schiff base ligand in comparison to simple Schiff base [14].

The condensation molar ratio formula of a Schiff base is around 1:1. The preparation of the base is assisted by component IR, and ^{1}H NMR details. All the obtained metallic polychaetes are having colouring nature that belongs to surrounding stability for a long duration. Other characteristics like non hygroscopic, insolubility in water, natural solvent but partially dissolved in warm DMF and DMSO. The assessment detail that assists in construction of compound and the metallic assessment assures the availability of metal: ligand ratio in the form of 1:1 [15].

Component	**Weight (/gmol)**	**Colour**	**Reflux duration(hour)**	**Assessment in percentage**			
				M	**C**	**H**	**N**
DHADAB	393.58	Yellow	Approx 3	- - - -	67.00	6.5	12.2
[VO(DHADAB)] H_2O	439.4	Pharaoh	Approx 10	10.29	53.6	4.10	5.70
[Fe(DHADAB)H_2O Cl]	481.8	Pharaoh	Approx 7	10.8	51.04	3.9	5.42
[CO(DHADAB) $2H_2O$]	449.4	Greyish Brown	Around 8	11.7	52.5	4.0	5.6
[Cu(DHADAB)2 H_2O]	453.9	Raven song	Approx 10	12.5	52.0	4.0	5.5
[Zn(DHADAB)2 H_2O]	455.8	Lemon Pie	Around 12	12.8	51.9	3.10	5.5

Table 1: Analysis of DHADAB and metallic components

The detail of all the appropriate components and their extraction like its colour, weight, reflux duration, surrounding stability, unable to dissolve in water while capability to dissolve in organic solvent and other related information are depicted in the (Table-1) that also illustrate the metal: ligand ratio that is obtained to be 1: 1.

Infrared Spectrum (IS) of Ligand:

The IS of ligand illustrated a wide range at 2935 cm^{-1} that belongs to the moderate intensity brand because of its intramolecular hydrogen bonding with hydroxyl group [16]. In the unavailability of this band, the metallic compound shows the separation of hydrogen bond and combining with oxygen atom and the molecules obtained by performing the procedure named dehydrochlorination. Under the spectre of ligand and the medium intensity brand the combination with metallic ions lead to loss of phenolic proton. This leads to upward movement in ν (C-O) phenolic frequency because of larger power by $\leq$ 15-33 cm^{-1} availability in the components that demonstrate the coordination of phenolic oxygen bonding. This medium broadband that supported the upward alteration stretch frequency also indicates shifting toward higher absorption and also trying to formulate the bonding (M-O) [17, 18].

The ligand range demonstrate a band of value 1610 cm^{-1} which shows the strong bonding between the chemical element specially carbon and nitrogen as C≡N it extends the frequency and by appropriate incorporation it can alter and move toward diminishing its frequency that lies around 22-40 cm^{-1} in all the metallic component ranges. This alteration in the frequency is mainly because there is a reduction in the density of electrons on which azomethine is associated. This shows that there is incorporation among the metallic atoms via azomethine nitrogen, that was also assisted by amino nitrogen and phenolic oxygen these elements are in comprised in the complexation along with the metallic items which is effectively manifested and indicated from the value of the spectrum that appeared as a "medium intensity band" and lie in the range of "(590-655 cm^{-1} and 450-496 $cm^{-1)}$" that are appropriately usable by the compound M-O as well as M-N respectively. The infrared absorption band shows strong correlation as the frequency spectrum lies around (967-970 cm^{-1}) among the compound that formulated in DHADAB and a double bond VO^{2+} moiety was formulated that takes assistance of distinct elements because of the flow of electrons. The components Vanadyl (IV) sulphate pentahydrate [VO(IV)] depicting the strong absorption because of double bond (V=O) that formulated with the assistants of integration of two elements σ *and* π and reflecting the band because of electron flow. O(Pπ → Vdπ). These observations of double bonds were extended towards normal values. For the metallic component anhydrous ferric chloride (III) [Fe(III)] ν(M-Cl) bonding reflecting or indicating the frequency ranging 380cm^{-1} [19]. Other antimicrobial components like "cobalt(II) acetate tetrahydrate [Co(II)], copper(II) acetate monohydrate [Co(II)], and zinc(II) acetate dihydrate [Zn(II)]" are comes under the frequency range 3413-3440 cm^{-1} because the hydroxyl bond ν(OH) shares a strong covalent bonding and shows the strengthful band shaping 835 cm^{-1}, as the water substance (H_2O) assists in coordination in the rocking and waging of the model [17, 18,19].

Electronic Spectra and Magnetic Properties:

The geometry coordination that shows four coordination is utilised to illustrate and depict the electronic spectral information as well as magnetic information of the metallic components. The diffuse reflectance spectrum band found approximately at 109890, 188679, and 252525 of Vanadyl (IV) sulphate pentahydrate [VO(IV)] comes under the range of 200-1000nm and demonstrates all the expected band values which are assigned to three distinct bands and relative transitional values "$^2B_2 \rightarrow {}^2E$ (dxy, dxz, dyz), $^2B_2 \rightarrow {}^2B_1$ (dxy, dx^2, y^2), $^2B_2 \rightarrow {}^2A_1$ (dxy, dz^2)". These values are showing the square pyramidal geometry of the complex. The magnetic movement of the compound [VO(IV)] is obtained to be at normal temperature

1.68 B.M which comes under the range of spin and only value that is obtained for one unpaired electron [20, 21]. For the component the magnetic moment obtained at a normal temperature depicts the octahedral geometric value showing the three distinct absorption bands lie in the range of "13071-13422, 15432-15748, and 22222-22624 cm^{-1}". These bands are allotted to "$^6A_{1g}$ (*S*) $\rightarrow ^4T_{1g}$ (G), $^5A_{1g}$ (*S*) $\rightarrow ^4T_{2g}$ (G) and $^6A_{1g}$ (*S*) $\rightarrow ^4A_{1g}$ (G)" showing the octahedral geometric value [22]. The magnetic movement of the compound [Fe(III)] is obtained to be at normal temperature 5.78 B.M which comes under the range of spin and only value that is obtained for one unmated negative ion. The disseminate reflectance frequency range found approximately at 17006-17300, 19074-190455, 188679 and 20408-20920 cm^{-1} of [Cu(III)] comes under the range of 200-1000nm and demonstrates all the expected band values which are assigned to three distinct bands and relative transitional values. These bands are allotted to "$^2B_{1g} \rightarrow ^2A_{1g}$, $^2B_{1g} \rightarrow ^2B_{2g}$ and $^2B_{1g} \rightarrow ^2E_g$", showing the pseudo octahedral geometric value. The magnetic movement of the compound [Cu(III)] is obtained to be at normal temperature 1.89 B.M [23]

The magnetic movement of the compound [Co(II)] is obtained to be at normal temperature 4.75 B.M which comes under the range of high spin octahedral geometry. For the component the magnetic moment obtained at a normal temperature depicts the octahedral geometric value showing the three distinct absorption bands lie in the range of 7704-7812, 10050-9881, and 16129-16722 cm^{-1}. These bands are allotted to "$^4T_{1g} \rightarrow 4T_{2g}$ (F), $^4T_{1g} \rightarrow 4A_{2g}$ (F) and $^4T_{1g} \rightarrow 4A_{1g}$ (P)" showing the octahedral geometric value revolving around the cobalt atom surroundings [17].

Antibacterial Studies and Analysis

Thermal evaluation of the compound was carried up to the range of 700 degree centigrade prominently because the compound was unexpected until the range of 60 degree then after the complexes showed slide depression up to the temperature reached 120 degree. This shows that all the compounds that consist of carbon hydroxyl bonding depict a mass loss in a moderate and a gentle manner. This shows the decomposition with the enhancement and temperature and depicts the similarity in the pattern in their thermal decomposition that comes under the fragmentation procedure. This loss of mass at a particular range of temperature is with respect to one water molecule for the metallic component [VO(IV)], [Fe(III)] and two water molecules for the other remaining components [Co(II)], [Zn(II)], and [Cu(III)]. After dehydration procedure is accomplished, the anhydrous material reach to the stability condition at a temperature range 320 degree centigrade and afterwards these material shows are swift degradation because of decomposition of organic elements that take part in the molecule formula and shows are the steep decline in the loss of percentage in mass, until and unless the temperature reached up to 620°. Then after the mixture shows the stability through horizontal bar line indicates that the formation of final decomposition of a metallic oxide product is obtained after the temperature level reaches up to 650 degree centigrade. This horizontal stability shows the stable metal oxide which was produced after the eventual decomposition procedure [17, 24].

Compounds	*E.Coli*	*S.Typhi*	*P. Aeruginosa*	*S.Aureus*
DHADAB	16	15	12	17
[VO(DHADAB)] H_2O	11	R	12	15
[Fe(DHADAB)H_2	12	15	16	14

O Cl]				
[CO(DHADAB) $2H_2O$]	15	13	11	16
[Cu(DHADAB)2 H_2O]	12	13	R	14
[Zn(DHADAB)2 H_2O]	13	R	12	18

Table 2: Antimicrobial activity ligand and its compounds [17, 25].

The inhibition impact of ligands and the metal compound with the increase of distinct antimicrobial elements is descriptive in the above table. The increased ligand activities of SB with distinct metal like Copper, Zinc, Iron and others provide promising results as illustrated from its *Escherichia coli and Staphylococcus aureus* column provide more inhabited activity among all the metallic complexes. The table shows that the "copper(II) acetate monohydrate [Co(II)] are highly active against *Staphylococcus aureus* as compared with the cobalt(II) acetate tetrahydrate [Co(II)], zinc(II) acetate dihydrate [Zn(II)] cobalt(II) acetate tetrahydrate [Co(II)], and anhydrous ferric chloride (III) [Fe(III)]" which also demonstrate effective activity against *Escherichia coli.* All the above-mentioned metallic components are repellent or powerless to reasonably active and little sensitivity against " *P.aeruginosa* and *S.typhi*" [26].

Conclusion

It may conclude that Schiff base has wide utility and diverse domains as it can be used in pigment, catalyst, polymer stabiliser, along with cohesion inhibitor for several metallic electrolyte because of the characteristics as it absorb and produce a corrosion mitigation outer covering through electronic enriched nucleus involving the imine moiety. This moiety can formulate and provide robust correlation with metallic elements because of Alpha (α) accepted features.

Schiff base has broadly started in the field of chemistry, as applied chemistry such as it provides several research related with carbohydrate in the amadori production, and the element used in the functioning of solar renewable energy, vitrimers. Some of its utility in organic chemistry is also explored because it provides a prominent and convenient synthetic strategy that operates to obtain a cost effective element. Under the biological domain of chemistry Schiff base has a wide variety of applications like it appropriately and significantly utilised in anti-malarial, anti-inflammatory, antiviral, antifungal, antibacterial characteristics. And brought phenomena these are called as antimicrobial properties or activities of Schiff base. One of the most critical antimicrobial activities performed by Schiff base is its working in amine or azomethine groups.

However, there are certain challenges and limitations that came in front of researchers while exploring the SB as when it works under homogeneous catalyst mechanism its activation functioning reduces during the procedure which requires recycling of catalyst and metal contamination. Another challenge was associated with the ortho hydroxyl group as the double bond among the carbon and nitrogen may lead to distinct activities among the pairing of tautomers. In order to resolve the challenges, the study recommends to utilise advanced functioning of Schiff bases such as chitosan-based Schiff bases and

nano-particles operationalised with Schiff bases as it has excellent possibility to overcome the challenges and provide suitable approach for identifying heavy metal ions that act as a promising ingredient for fighting antimicrobial resistance challenges and provide a new ray of hope for therapeutical specifically in the antimicrobial field.

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