



Review Article

Different schiff base metal(II, III) complexes derived from benzil and its derivatives: A short review

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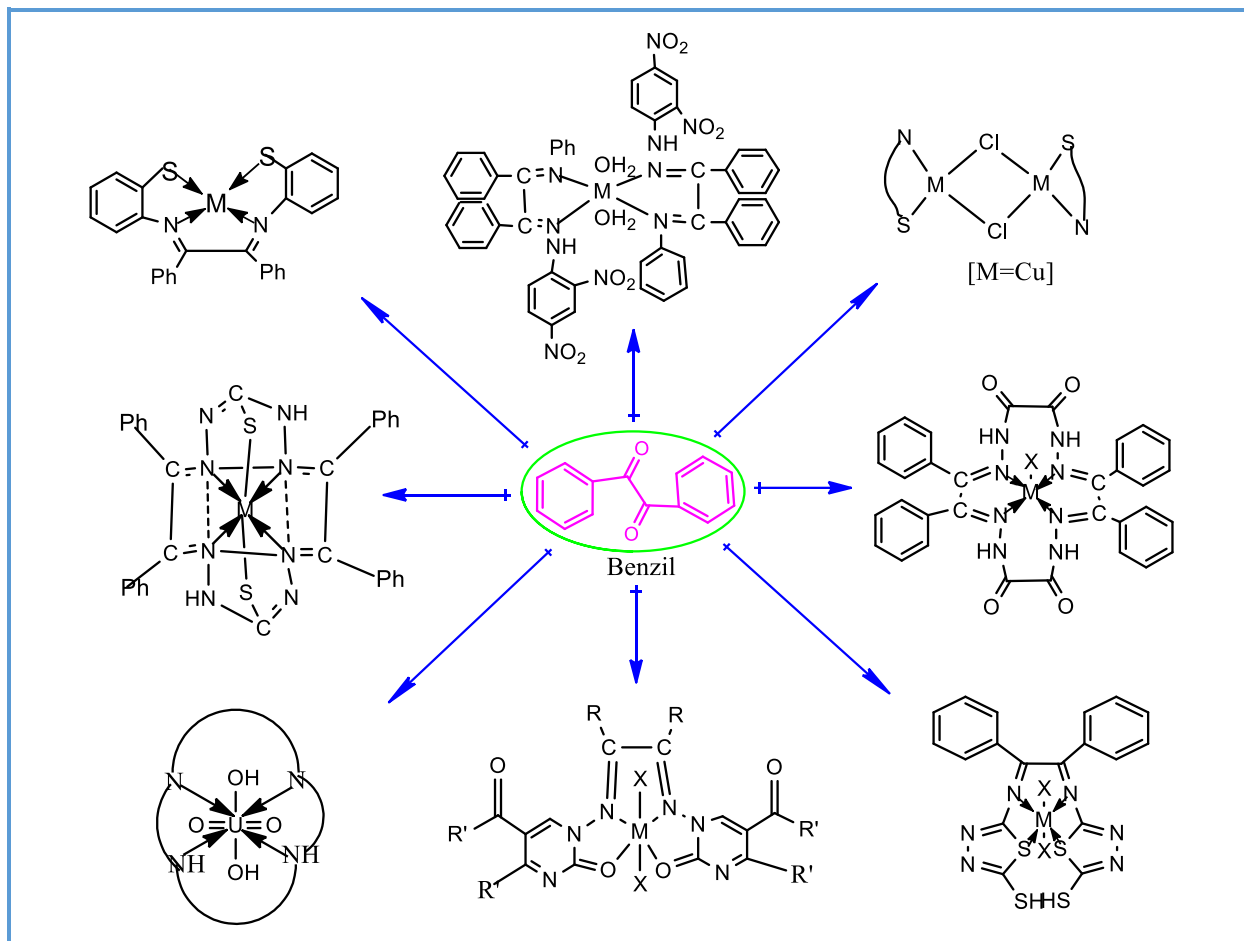
Transition Metal Complexes
Hetero-Atomic Ligands
Antibacterial Activities
Diketones
Different Amines

ABSTRACT

Schiff base transition metal complexes using benzil and its derivatives as a diketone with different amines in 1:1 or 1:2 ratio can be synthesized by simple condensation reaction or mild conditions. They may contain donor atoms such as oxygen, sulphur, and nitrogen which make the ligand a better chelating ligand to coordinate strongly with different metal ions to form Schiff base metal complexes. Here, in this review, the synthesis of different types of Schiff bases customized from benzil and corresponding bidentate, tridentate, and tetradentate metal complexes has been summarized.

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Graphical Abstract



Introduction

Schiff base contains an azomethine groups connected to an aryl or alkyl groups but not to hydrogen [1]. The Schiff bases were synthesized from an amine and a carbonyl compound by nucleophilic addition [1]. From the reaction, a hemi-aminal group was formed which by dehydration generated an imine compound [1]. Metal complexes of Schiff bases have an important role in coordination chemistry [2-4]. Schiff bases and their metal complexes were essential for various biological activities like antibacterial, antifungal, antiviral, antimalarial [5-10]. From various literature surveys, it was found that benzil and its derivatives are

essential and extensively used reagents because of its practical applications in organic and pharmaceutical industry [11-16]. Benzil is an important precursor for various chelating ligands and their corresponding transition metal (II, III) complexes. Metal complexes of Schiff base based on benzil have played a key role in the development of transition metal complexes some of which are commercially important and many macro-cyclic benzil based metal complexes are also known which have been used in analytical, industrial, and medical fields [17-21]. Schiff bases of benzil and their chelating ligands can be synthesized by simple condensation reaction of different mono amines or di-amines.

Schiff base metal complexes of benzil and its derivatives

Schiff base of benzil-2,4-dinitrophenylhydrazone with aniline was prepared and characterised in 1:1 molar ratio in ethanol medium [22]. The corresponding metal complexes of Cu(II), Co(II), Ni(II), and Zn(II) were prepared and characterised by refluxing the metal chloride salt with the Schiff base ligand in ethanol medium keeping the metal ligand ratio 1:2. All complexes show octahedral geometry. Initially, benzil-2,4-dinitrophenylhydrazone and aniline was dissolved in ethanol in 1:1 molar ratio and the resulting mixture was refluxed for 1 hour. The yellow solid was precipitated as Schiff base. It

was then filtered, washed with distilled water, and dried finally. For the synthesis of metal complexes, an ethanol solution (10 mL) of Schiff base (10 mM) was mixed with an ethanol solution (10 mL) of metal chloride salt (5 mM) keeping the ligand metal ratio 2:1. Few drops of acetic acid were added to it. The resulting solution was refluxed for 1 hour. The solid product was precipitated out. Then it was filtered, washed, and dried. Characterizations of the complexes were done by IR, UV-Vis, $^1\text{H-NMR}$, $^{13}\text{C NMR}$, ESR, mass spectral techniques, micro-analytical data, and magnetic susceptibility measurements [22]. The probable structure of the metal complexes is demonstrated in Figure 1.

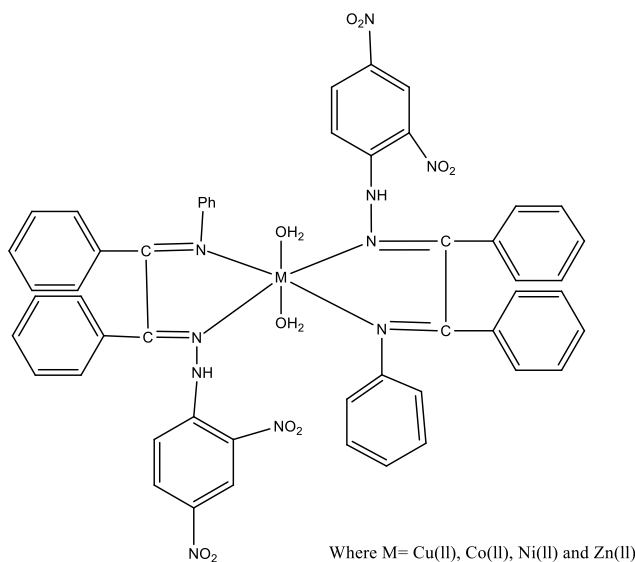


Figure 1. The probable structure of the metal complexes

Chelating complexes of Fe(III), Mn(III), and Cr(III) were synthesised by condensation of oxalyldihydrazide with benzil in hot methanol solution in the presence of the respective trivalent metal salt [23]. Initially, 10 mmol of oxalyldihydrazide was introduced into 50 mL of hot stirring methanol. Then, 5 mmol of trivalent chromium, iron, and manganese salts were added into the solution by dissolving the respective metal salts in minimum quantity of

methanol. The resulting mixture was boiled and refluxed for 30 minutes. After that, 10 mmol of benzil was added to the mixture and the whole solution was refluxed for 7-8 hours. The metal complexes were obtained and characterized by different analytical, spectral, and magnetic data. All complexes possess superior antibacterial activity [23]. The probable structure of the metal complexes is displayed in Figure 2.

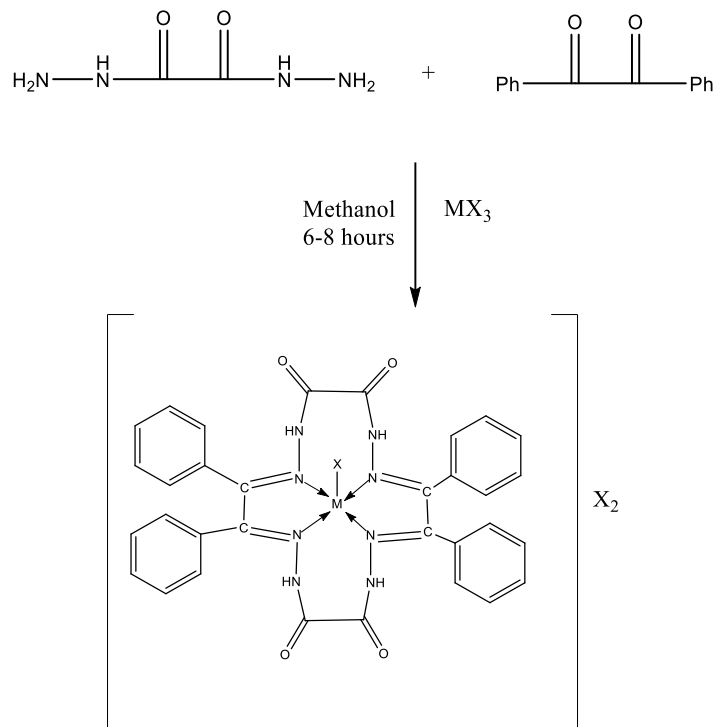


Figure 2. Probable structure of the metal complexes where, M= Mn(III), Cr(III), Fe(III), and X= Cl⁻, NO₃⁻, CH₃COO⁻

Schiff base metal complexes of Mn(II), Cu(II), Co(II), Ni(II), and Zn(II) were prepared from their corresponding chloride salt by template reaction using tetradentate Schiff base derived from benzil, ethanolamine and 4,4'-methylenedianiline with molar ratio 1MDA: 2Benzil: 2Ethanol amine: 2M [24]. Hot ethanolic solution of benzil (0.02 mol) was added to the ethanol solution (25 mL) of MDA (0.01 mol) and the resulting solution was refluxed at 50 °C for 2 hours. Then ethanol amine (0.02 mol) was added to the solution and again refluxed for 60 minutes. After that, an ethanol solution (25 mL) of metal(II) chloride salt (0.02 mol) was added dropwise to that reaction mixture followed by a few drops of conc. H₂SO₄. This resulting solution was refluxed at 70 °C for 3 hours. The complex was formed and it was then filtered, washed, and finally dried in vacuum. The structure of these complexes was identified by different analytical and spectral methods [24]. The

probable structures of the metal complexes are shown in Figures 3 and 4.

Macro cyclic complexes of Co(II) were synthesised by the condensation reaction of benzil, bis ortho phenylenediamine with acetophenone, benzaldehyde, and P-chloro benzaldehyde [25]. Firstly, benzil (50 mmol, 10.5 g) was added to 100 mL methanol, and then ortho phenylene diamine was added to it and the resulting solution was refluxed for 3 hours. After that, the solution was left in the air. A microcrystalline white crystalline precipitate was appeared after 20 hours, and then it was filtered off and washed with 100 mL of water.

Co-AP: To a solution of CoCl₂ in 15 mL methanol, acetophenone was added, and then benzil bis ortho phenylenediamine was added to it. The resulting solution was stirred for 1 hour and 30 minutes at room temperature. The precipitate was filtered off and dried [25].

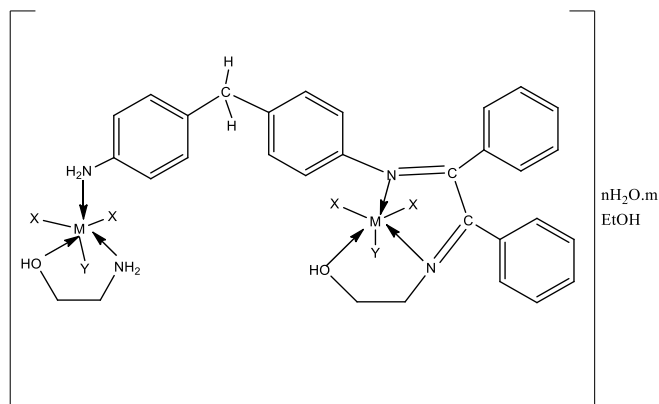


Figure 3. The possible structure of metal complexes where M= Mn(II), Co(II), Ni(II), and Zn(II); X= Cl; Y=H₂O

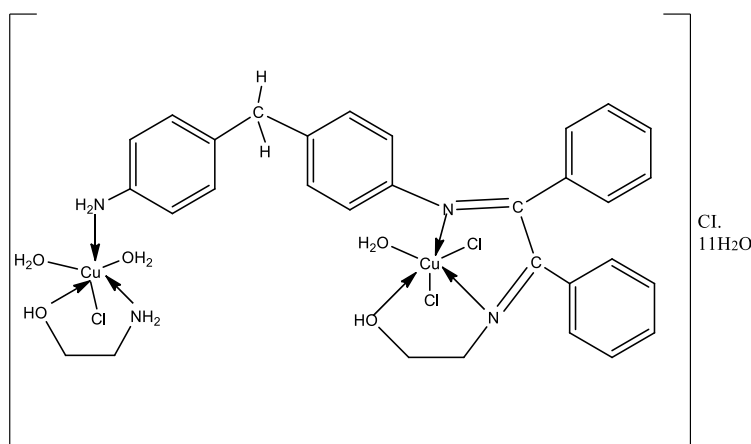


Figure 4. The proposed structure of Cu(II) complex

Co-BZ: To a solution of CoCl₂ in 3 mL methanol, benzaldehyde was added, and then benzil bis ortho phenylenediamine was added to it. The resulting solution was stirred for 2 hours at room temperature. The precipitate was filtered off and dried [25].

Co-P: To a solution of CoCl₂ in 4.5 mL methanol P-chloro benzaldehyde was added, and then benzil bis ortho phenylenediamine was added to it. The resulting solution was stirred for 2 hours at room temperature. The precipitate was filtered off and left for precipitation [25].

The characterization of all these macrocyclic complexes was done by XRD and EXAFS analysis. The particle size and lattice

parameters have been estimated by XRD analysis. XRD analysis established the crystalline nature of all complexes [25].

Macrocyclic binuclear Cu(II), Ni(II), and VO(II) Schiff base complexes were synthesised by a template condensation of 3,3'-diaminobenzidine, with benzyl and dimethyl ortho phenylenediamine [26]. To a hot methanolic solution of 20 mL of 3,3'-diaminobenzidine metal salt (0.2 mmol) of the corresponding metals was added by dissolving the salt in minimum quantity of methanol and the solution was stirred vigorously. This resulting solution was boiled under reflux for 1 hour and 30 minutes. Then, a methanolic solution of benzyl (0.4 mmol) was added and

after that a methanolic solution of dimethyl ortho phenylenediamine (0.2 mmol) was added to the solution. The whole resulting solution was refluxed for 8 hours resulting in the formation of tetradentate macrocyclic Schiff base binuclear complexes of the type $(M_2L.X)$ and $(M_2L).X_2$ (where, $X= 2ClO_4^-$, CH_3COO^- and $2SO_4^{2-}$). The structural characterizations of the complexes were done by elemental analysis, electronic spectra, IR, EPR, cyclic voltammetry,

molar conductivity, and magnetic susceptibility measurements. The geometry was found square planar for the binuclear complex of Cu(II), Ni(II), and square pyramidal environment for VO(II) centre. The complexes show strong antimicrobial activity against gram-positive bacteria (*taphylococcus auerus*), gram-negative bacteria (*pseudomonas fluorescens*) [26]. The probable structure of the metal complexes is shown in Figure 5.

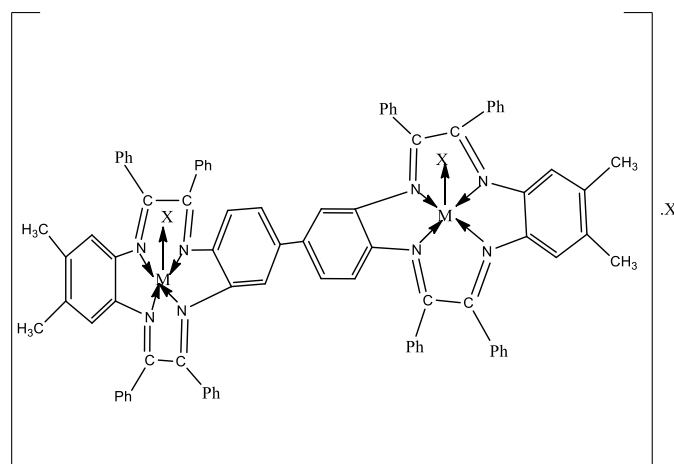


Figure 5. The proposed structure of the complexes Where, $M= Cu(II)$, $Ni(II)$, and $VO(II)$ $X= CH_3COO^-$, $2ClO_4^-$, and $2SO_4^{2-}$

Schiff base complexes of Ni(II), Cu(II), Co(II), and Rh(II) were synthesized from two Schiff base ligands formed by the condensation reaction of benzil and furfural with S-benzylthiocarbamate. S-benzylthiocarbamate (2.0 g) in ethanol (150 mL) was mixed with a solution of benzil (1.6 g) in ethanol (100 mL) [27]. This mixture was then heated on a hot water bath for 10 minutes. The colour of the solution changed from pale yellow to light brown. A oily substance was found by the rotary evaporation of the solution. No crystallisation was occurred in the oily appearance. A pure crystalline product was appeared when the ethanol solution of the oily substance reacted with metal salts. Therefore the ethanol solution of the oil was used for the preparation of metal

salt. All complexes were prepared by a general method. Metal salts (0.005 mol) in ethanol (25 mL) were mixed with the 50 mL ethanol solution of Schiff base (0.01 mol). The resulting solution was heated on a hot water bath for 10 minutes and then allowed to cool and kept for crystallisation. The crystals which had formed were filtered off, washed then thoroughly with ethanol, and dried in a vacuum desiccator over P_4O_{10} . The complexes were characterized by elemental analysis, magnetic data, and spectroscopic measurements. The Ni(II) complexes, $Ni(N-S)_2$, and $Ni(O-N-S)_2$ are sq. planar and octahedral, respectively [27]. The $Cu(N-S)Cl$ complex is paramagnetic having magnetic moment 1.73 B.M [27]. A halogen bridged dimeric structure is proposed for the

complex. The Cu(II) complex, Cu(O-N-S)Cl shows diamagnetism due to the strong antiferromagnetic coupling between two Cu(II) ions in a thio sulphur bridged dimeric or polymeric structure [27]. The Rh(III) complex, Rh(N-S)₂Cl shows dimeric halogen bridged

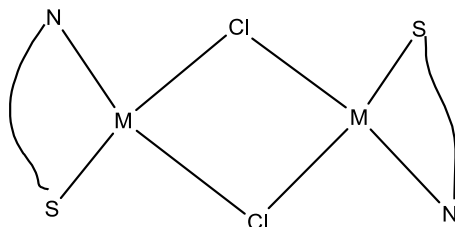


Figure 6. The probable structure of the complex where M=Cu

A new tetradentate acyclic mononuclear or tridentate binuclear metal complexes of the type [ML¹X₂]X, [ML²X₂]X and [Fe₂(L¹)₂Cl₄]Cl₂. H₂O [where M=Fe(III), Cr(III). L¹(benzyl derivative), L²(diacetyl derivative) are N₂O₂/ONO acyclic ligand and X= Cl, NO₃] were synthesised by the template synthesis of 1-amino-5-benzoyl-4-phenyl-1H-pyrimidine-2-one and benzil or diacetyl in presence of FeCl₃.H₂O, CrCl₃.4H₂O salts [28]. Initially, 1-amino-5-benzoyl-4-phenyl-1H-pyrimidine-2-one (0.528 g, 2 mmol) was dissolved in 30 mL *n*-butanol. After that, 0.1 mL (1 mmol) of diacetyl or 0.210 g (1 mmol) of benzil in 20 mL *n*-butanol was added drop wise with constant stirring to the previous solution. To this solution, 1 mmol

of metal salts in 20 mL *n*-butanol [FeCl₃. 6H₂O (0.270g) and Cr(NO₃)₃. 9H₂O (0.400g)] were added dropwise by constant stirring. The resulting mixture was refluxed for 3-6 hours. The volume of the mixture was then reduced to 10 mL by rotary evaporation. The precipitate was then filtered, washed with diethyl ether and cold ethanol, respectively. Finally, the product was dried in a vacuum desiccator. The complexes were characterised by elemental analysis, molar conductance, magnetic measurements, IR, and UV-Visible spectroscopy [28]. Scheme for preparation of acyclic Schiff base complexes is demonstrated in Figure 7. The probable structure of Fe(III) complex is illustrated in Figure 8.

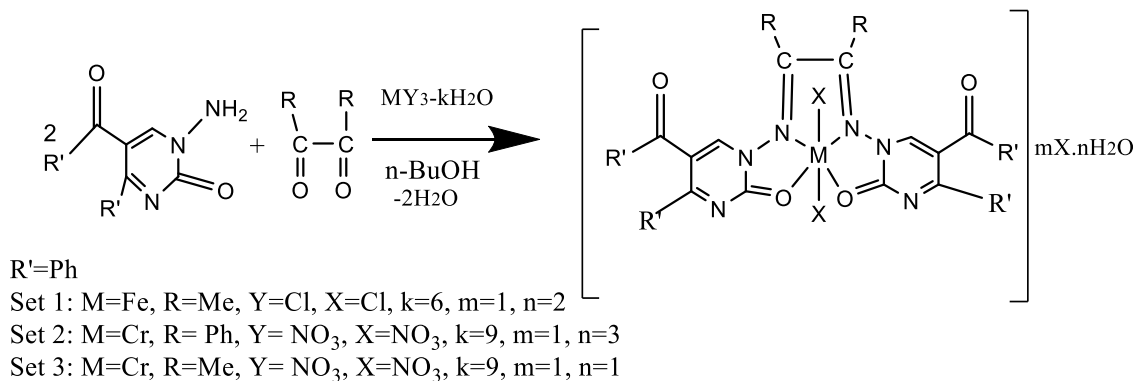


Figure 7. Scheme for the preparation of acyclic Schiff base complexes.

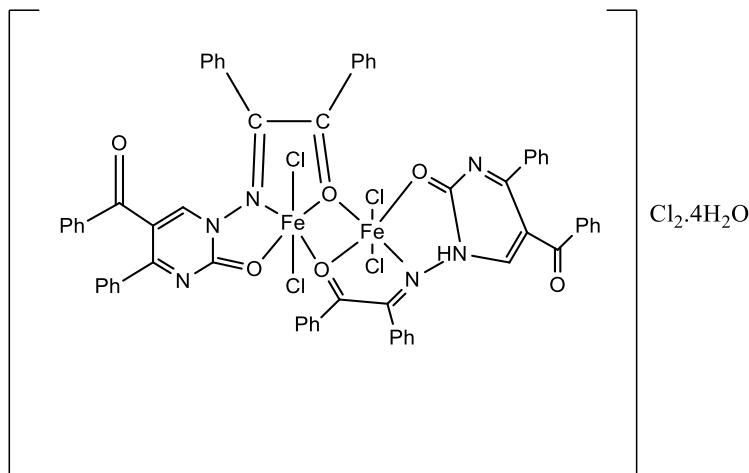


Figure 8. The probable structure of Fe (III) complex

A quadridentate N_2O_2 Schiff base was synthesized by the condensation reaction of 1-phenyl-2,3-dimethyl-4-aminopyrazole-5-one (4-aminoantipyrine) and benzyl [29]. This ligand acts as a quadridentate ligand and forms cationic complexes with Cu(II), Ni(II), Co(II), Mn(II), Zn(II), and VO(IV) salts in ethanol solution. Characterizations of the complexes were done by IR, UV-Vis, 1H -NMR, ^{13}C NMR, ESR, mass spectral techniques, microanalytical data, and magnetic susceptibility measurements. According to the electronic absorption, and IR data the complexes have an octahedral

geometry. VO(IV) complex has a square-pyramidal geometry. The non-electrolytic behaviour of the chelate was established by the low conductance data except for vanadyl complex. The monomeric nature of the chelates was confirmed from their magnetic susceptibility values. The metal complexes showed a strong antimicrobial activity for *Bacillus subtilis*, *Salmonella typhi*, and *Shigella flexneri* than the free ligands [29]. Scheme for the synthesis of the Schiff base is depicted in Figure 9. Probable structure of the $[CuL(Py)_2]X_2$ complexes is demonstrated in Figure 10.

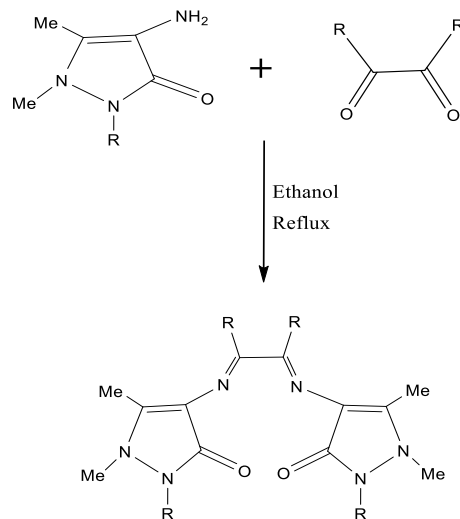


Figure 9. Scheme for synthesis of the Schiff base, where R=Ph

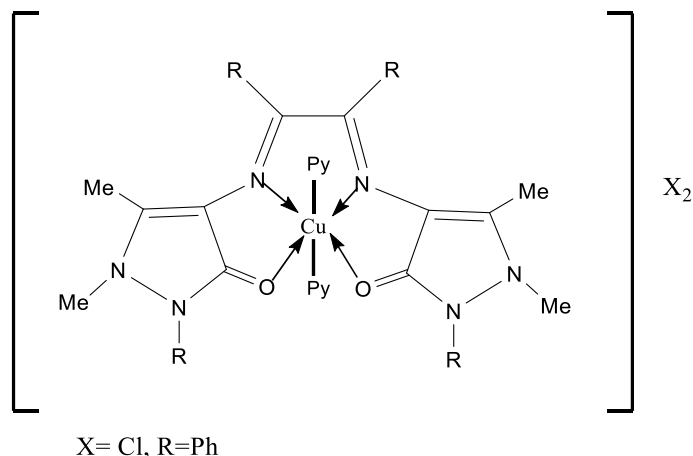


Figure 10. The probable structure of the $[\text{CuL}(\text{Py})_2]\text{X}_2$ complexes

Another novel Schiff base ligand was prepared by the condensation reaction of benzil with triethylenetetraamine [30]. Hot ethanolic solution (25 mL) of triethylenetetraamine (5 g, 34.19 mmol) was mixed with a hot ethanol solution (25 mL) of benzil (7.18 g, 34.19 mmol) and the mixture was refluxed for 2 hours at 60 °C. The yellow coloured solid product was filtered, recrystallized in ethanol, washed with di-ethyl ether, and finally dried under vacuum. A series of transition metal complexes of this Schiff base ligand (H_2L) was prepared. Complexes of Mn(II), Cu(II), Ni(II), Co(II), Cd(II), Zn(II), Fe(III), Th(IV), and $\text{UO}_2(\text{II})$ were prepared from their corresponding chloride or acetate salts. The hot ethanol solution (25 mL) of the metal salts (1 mmol) was added to the hot ethanol solution (25 mL) of Schiff base H_2L (1 mmol) and the solution was kept under refluxed while stirring at 60 °C. The complexes formed in this way were collected by filtration, washed with di-ethyl ether. The ligand and all complexes were characterised by UV, IR, NMR, mass, elemental analysis, magnetic moment, molar conductance, and thermal analysis. The complexes have more anti-bacterial activity than the parent ligand [30]. The proposed structures of metal complexes are shown in Figure 11.

A Cd(II) complex of benzil -2- Mercaptoanil was prepared by Hans Jadamus *et al.* [31]. Initially, the ligand was prepared when an ethanol solution (20 mL) of benzil (2.1 g, 0.01 mol) was mixed with ortho aminobenzenethiol (2.5 g, 0.02 mol) at 50 °C. The solution was then kept at room temperature for 1 hour. The yellow crystalline product was formed and then filtered, washed with methanol, and finally dried in air. Scheme for the preparation of the Schiff base is shown in Figure 12. For the formation of complex, 30 mL methanolic solution of Cadmium acetate dehydrate (267 mg, 0.001 mol) was added into the hot solution of that ligand (951 mg, 0.003 mol) in methanol, and then 10 mL of 0.2 (M) solution of NaOH was added drop wise to the solution with constant stirring. The complex was formed; this way was collected by filtration, washed, and dried in air [31]. The probable Structure of the Cd(II) complex is shown in Figure 13.

Cu(II), Ni(II), Co(II), and Pd(II) complex of N_2S_2 and N_2S_4 ligand was synthesized from benzildithiosemicarbazone (BDTSC) and thiocarbohydrazone [32]. For the preparation of BDTSC, thiosemicarbazide (0.08 mol) was dissolved in 50 mL hot water and this solution was added to the methanolic solution (50 mL) of benzil (0.04 mol).

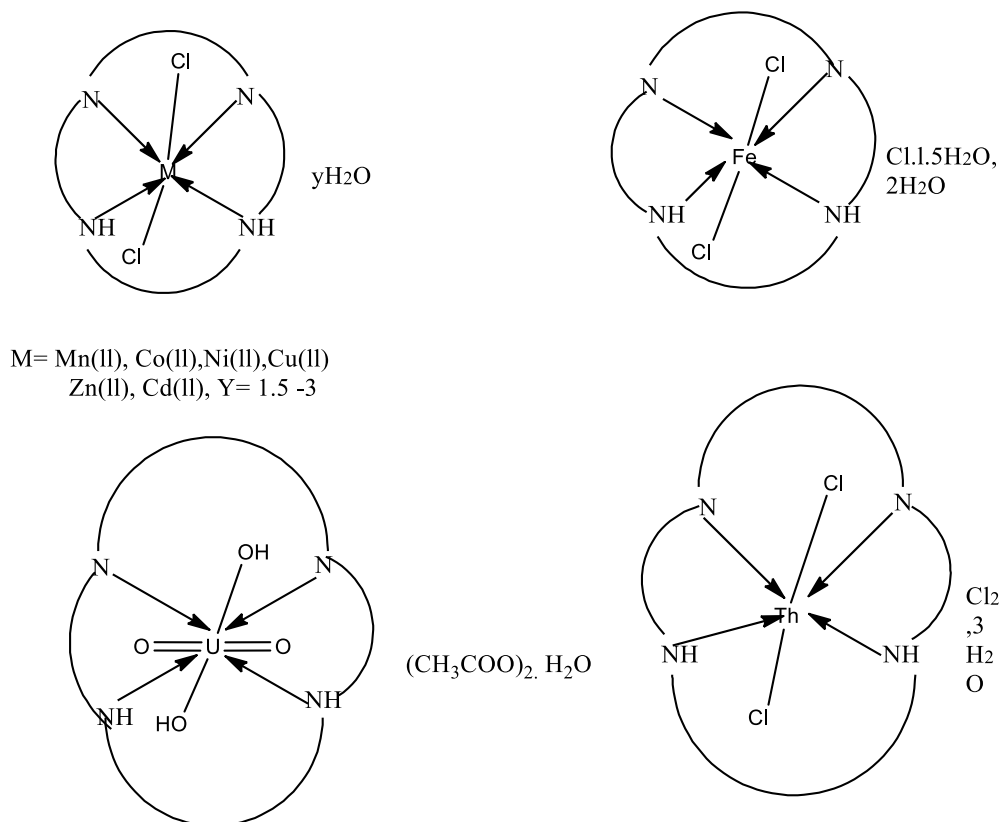


Figure 11. The proposed structure of H₂L metal complexes

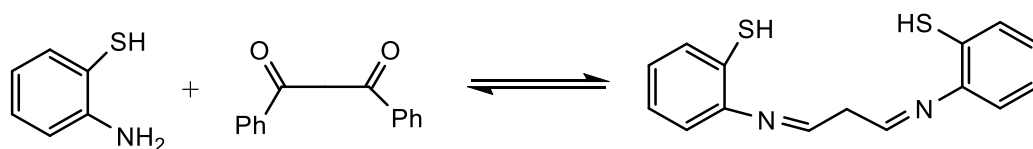


Figure 12. Scheme of the preparation of Schiff base

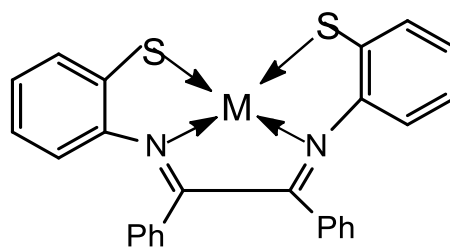


Figure 13. The possible structure of the complex where M= Cd(II)

To this mixture, 3 mL conc. HCl was added, and then the mixture was refluxed for 30 minutes. A light yellow coloured product was appeared which was filtered, washed, and recrystallized from ethanol. Now for the

preparation of the complex, 0.01(M) metal salt was dissolved in distilled water and this solution was added to the methanolic solution of BDTSC (0.01 M). This mixture was refluxed for 2 hours, and then allowed to cool. The

obtained solid product was filtered, washed with hot water and followed by methanol, recrystallized from ethanol, and then dried at 80 °C. Tetraaza macrocyclic complexes of Ni(II), Co(II), Cu(II), and Fe(II) were synthesized from benzil and thiocarbohydrazide. For this, 0.01 (M) benzil and 0.01 (M) thiocarbohydrazide were mixed in a round bottom flask. To this mixture, a sufficient amount of metal salt solution was added and the solution was refluxed for 2 hours. On cooling, a solid precipitate was obtained. The solid product was filtered, washed with hot water followed by methanol, and recrystallization was done from ethanol [32]. The possible structures of the metal complexes and macrocyclic metal complexes are indicated in Figures 14 and 15, respectively.

A novel Schiff base ligand was synthesized by the condensation reaction of benzil with 5-amino-1,3,4-thiodiazole-2-thiol in 1:2 ratio [33]. The ligand was produced in this reaction named as benzil bis(5-amino-1,3,4-thiodiazole-2-thiol). The scheme for the synthesis of Schiff base ligand is shown in Figure 16. The synthesized ligand behaved as a tetradentate ligands. Ni(II) and Cu(II) complexes were synthesized with this nitrogen sulphur(N₂S₂) donor ligand of thiol ring and imine ring,

respectively. A hot ethanolic solution of benzil (1 mol, 2.102 g) was added to the hot ethanolic solution of 5-amino-1,3,4-thiodiazole-2-thiol (2 mol, 2.664g) with constant stirring and then the mixture was refluxed for 5-6 hours at 60 °C. The solution was allowed to cool at room temperature, and then kept in the refrigerator overnight. The yellow colour solid was precipitated out, filtered, and washed several times with cold ethanol, and finally it was dried in vacuum. To this hot ethanolic solution of Schiff base ligand (0.001 mol), a hot ethanolic solution of metal salts was added and the whole solution was refluxed for 10-12 hours at 90 °C. The pH of the solution was maintained at 5-6 by adding 2-3 drops of the aqueous ammonia. The final metal complex was formed in this way, was then filtered, washed with ethanol, and dried under vacuum over P₄O₁₀. The complexes were characterised by elemental analysis, molar conductance, magnetic measurements, IR, UV-Visible, EPR spectroscopy, thermal, and molecular modelling. The synthesized ligand and complexes showed biological activities. Metal complexes have strong biological sensitive nature than free ligand [33]. The probable structures of the metal complexes are depicted in Figure 17.

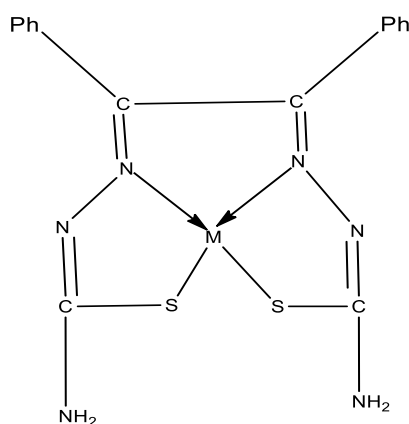


Figure 14. The possible structure of metal complexes. M= Co(II), Ni(II), Cu(II), and Pd(II)

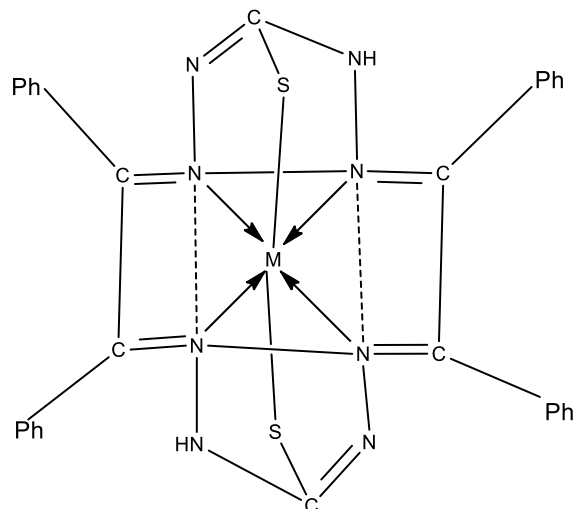


Figure 15. The probable structure of macrocyclic metal complexes. M= Co(II), Ni(II), Fe(II), and Cu(II)

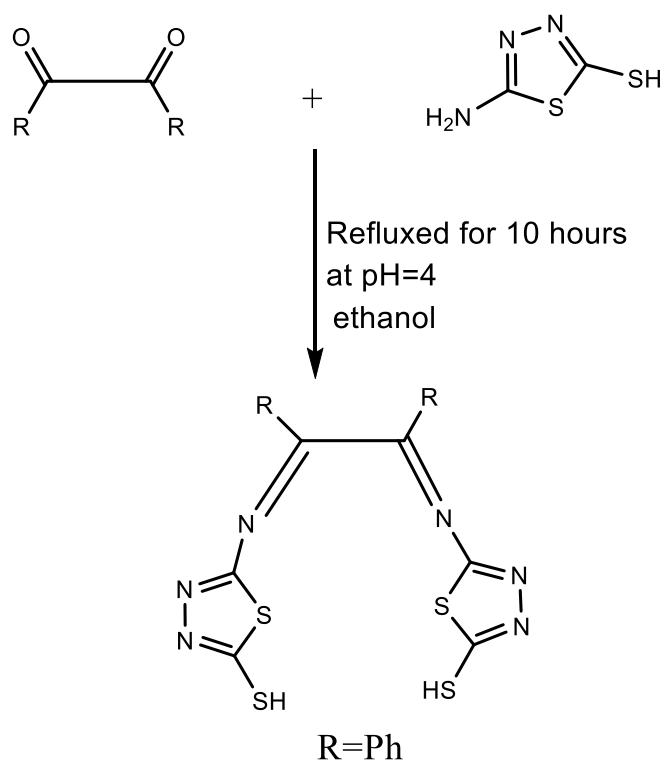


Figure 16. Scheme for the synthesis of Schiff base ligand benzil bis(5-amino-1,3,4-thiodiazole-2-thiol)

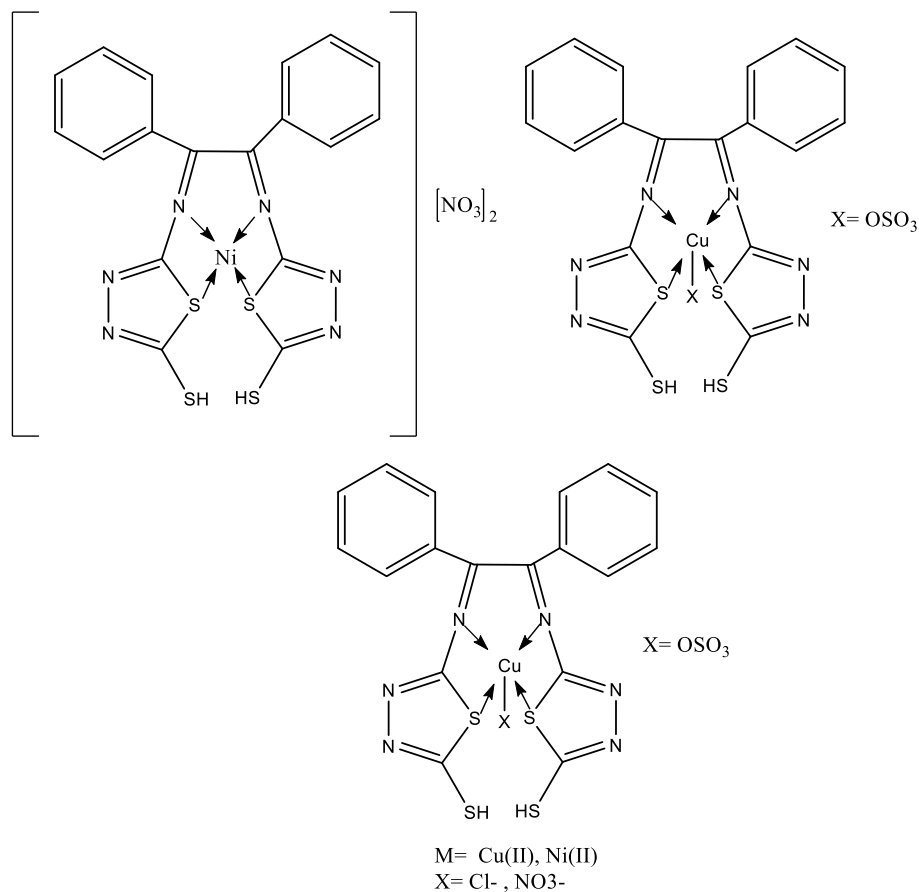


Figure 17. The probable structure of the metal complexes

Conclusion

Metal complexes can be prepared from different Schiff base ligands of benzil and its derivatives. Schiff base ligands of benzil and its derivatives can act as a good chelating agent. These Metal-Schiff base complexes have broad applications in various biological activities including antifungal, antibacterial, antimalarial, antiproliferative, anti-inflammatory, antiviral, and antipyretic properties. The Schiff base complexes of benzil have an important role in coordination chemistry. The simple synthesis procedure of the ligands, binding capacity with the metals with their different denticities, combination of donor atoms like oxygen, sulphur, and nitrogen, strong stability have

made them an important ligand in coordination chemistry and transition metal complexes.

Disclosure Statement

No potential conflict of interest was reported by the authors.

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Authors' contributions


All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

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