Characterization of Synthesis and Biological Studies of Some Transition Metal Complexes of New Schiff Bases of Sulfa Drugs

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Abstract: The transition metal complexes find extensive applications in technology, industry and medicine. The recognition of the potential employment of transition metal complexes and chelates in therapeutic applications provide useful outlets for basic research in transition metal chemistry. Metals play a vital role in an immerse number of extensively differing biological processes. Some of these processes are quite specific in their metal ion requirements Co-ordination compounds including Schiff base ligands are significantly important because they play a vital role in life process and are well-known for their antibacterial, antifungal, anti HIV, anti inflammatory, analgesic, antitumor, antimalarial, anti dyslidmic, anti convulsent activityes. The azo methine linkage (-N=CH) is a significant feature that makes Schiff base an important biological active species as well as ligands for co-ordination. Schiff bases have been recently focused by the co-ordination chemists as versatile ligands because of their preparative accessibilities and structural varieties. The interaction between transition metal ions and biologically active Schiff base ligands become an important route in designing new metal based antibacterial and antifungal agents against different kinds of bacteria, fungi and associated viruses that become resistant to the conventional drugs. Literature review suggests that sulphonamides are well known for their wide range of biologically activity. Hydrazono group are also considered as physiologically active group. Therefore it is considered worthwhile to synthesis some new Schiff base ligands by the reaction of dike tones (derived from sulphonamides), with heterocyclic amines. The Ti (III), V (IV), Cr (III), and Co (III) complexes of newly synthesized Schiff bases have been synthesized with corresponding metal halides. The newly synthesized Schiff base ligands and complexes were characterized by elemental and spectral analysis. Geometries of the complexes were established on the basis electronic spectra of complexes and magnetic susceptibility measurement. Biological activity of synthesized Schiff bases and their Ti (III), V (IV), Cr (III), and Co (III) complexes have been evaluated against selected bacteria and fungi.

Keywords: Synthesis, biological, Metal Complexes, Schiff, Sulfa Drugs

1. Introduction

Intensive efforts have been made to design novel compounds to confront new strains of resistant micro organism. The ongoing search for novel and innovative drug delivery systems is predominantly a consequence of the well established fact that the conventional dosages are not sufficiently effective in conveying the drug compounds to its site of action and this has necessitated the need to search for more potent drugs. The recognition of the potential employment of transition metal complexes and chelates in therapeutic applications [1-2] provide useful outlets for basic research in transition metal chemistry.

Metals play a vital role in an immenes number of extensively differing biological processes. Some of these processes are quite specific in their metal ion requirements. A number of antibiotics such as bleomycin, streptomycin and bacitracin have been reported to function properly upon co-ordination with transition metal ions [3] The efficacies of some therapeutic agents are known to increase upon coordination; hence metal based drugs are seen as possible replacement for most of the present drugs [4]. Co-ordination compounds including Schiff base ligands are significantly important because they play vital role in life process and are well-known for their antibacterial, [5] antifungal [6], anti HIV [7-8], anti inflammatory [9-10], analgesic [11], antitumor [12-13], antimalarial [14], anti dyslidmic [15], and anti convulsent [16] activities. Schiff bases have been recently focused by the co-ordination chemists as versatile ligand because of their preparative accessibilities and structural varieties [17-18]. Schiff bases that contain aryl

substituent's are substantially more stable and more readily synthesized, [19] while those which contain alkyl substituent's are relatively unstable and readily polymerizable [20-21].

Schiff bases

Schiff base ligands occupy unique position in co ordination chemistry for complexation. These ligands can be readily synthesized by condensation of primary amines with carbonyl compounds and this general approach allows access to ligand system of various denticity. The general formula of Schiff base is R1R2C = NR3, here R1 is an aryl group, R2 is eitherhydrogen atom or alkyl/ aryl group and R3 is either an alkyl or aryl group. The formation of a Schiff base from an aldehydes or ketones is a reversible reaction which generally takes place on heating with acid or base catalysts.

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The formation is generally driven to the completion by separation of the product or removal of water, or both. Many Schiff bases hydrolyzed back to their reactants under

aqueous condition.

The mechanism of Schiff base formation involves the neucleophilic addition of amine to the carbonyl group. Thus forming an unstable addition compound called carbinolamine. The carbinolamine looses water by either acid or base catalyzed pathways. The dehydration of the carbinolamine is the rate-determining step of Schiff base formation is catalyzed by acids. However the concentration of acid should not be too high because amines are basic compounds.



And may get protonated and would be non-neucleophilic, so equilibrium may pull to the left and carbinolamine formation cannot occur. Therefore Schiff bases synthesis are best carried out at mildly acidic conditions.

Schiff bases are classified as bidentate (I), tridentate (II) tetradentate (III) and polydentate ligands (IV) capable of forming very stable complexes with transition metals. They can also form a five or six membered ring with metal ions if they bear a functional groups like -OH,-NH,-SH, sufficiently nearer to the site of complexation.











Donget etal [22] synthesised some salen type Schiff bases (V -VIII) which are very active ligands for co-ordination.

нο

OH

(I)

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N, N bis (Pyridin-3 caboxalidene) 1-2 diamino ethane (V)



N, N-bis (3-nitrobenzulidene) 1-4 diamino butane (VI)



N, N'bis (Salicylidene) 1-6 diaminohexane. (VII) N, N'bis (Salicylidene) 1-2 diamino ethane (salen) (VIII)

(VIII)



Schiff bases and their transition metal complexes exhibit many biological activities [23-24]. Schiff bases of N-methyl and N-acetyl isatin derivatives with different arylamines have been prepared and screened for anti convulsant activities [25].

Schiff bases have also been shown to exhibit a broad range of biological activities including antimicrobial [26-38], antiinflammatory [39-43], analgesic [44], anti-tubercular [45-47], antimycobacterial [48-49], antioxidant [50], antiviral [51] andinhibitors [52]. Thiacetazone and Nitrofyrazone (IX & X) are the drugs which have Schiff base in their structures which are responsible for their biological activities





Dimmock et al have synthesized acetylhydrazones XI provided good protection against convulsions while the oxamoylhydrazones XII were significantly less active [53]



(XII)



4-Aminobutyric acid (GABA) is the principal inhibitory neurotransmitter in the mammalian brain. GABA hydrazones XIII were synthesized and evaluated for their anticonvulsant properties in different animal models by Ragavendran et al [54].



Lima and coworkers have synthesized [(4-dimethylamino benzylidene-3-(3, 4methylenedioxyphenyl) propionylhydrazine] XIV was more potent than dipyrone and indomethacine are used as standard anti-inflammatory/ antinociceptive drugs [55].

XIV



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Kucukguzel and coworkers have synthesized-(4methoxybenzamido) benzoyl]-2-[(5-nitro-2-furyl) methylene] hydrazine XV inhibited the growth of several bacteria and fungi [56].



Turan-Zitouni et al have prepared some 5-Bromoimidazo [1, 2-a] pyridine-2-carboxylic acid benzylidenehydrazide XVI and screened their antimicrobial activity [57].



Antibacterial activity of monobasic bidentate Schiff base ligand with N, O donor have been reported by K. N. et. al [58]. Schiff bases are some of the most widely used organic compounds. They are used as pigments and dyes, catalysts, intermediates in organic synthesis, and polymer stabilisers. Some of them were used as antifertility and enzymatic agents [59]. L-Amino acid Schiff bases with N, O donorsystem have been reported by Taqui Khan et al [60], are used as catalyst of enantio selective epoxide of 1, 2-di hydro-naphthalene. Nitro substituted benzaldehyde Schiff bases were used in organic catalytic reactions [61]. Schiff bases were also used as optical and electrochemical sensors and have applications in chromatographic methods and in various other analytical processes [62-65]. They are also effective corrosion inhibitors [66]. Schiff bases of ethylenediamine/ triethylenetetramine with benzaldehyde/ cinnamic aldehyde/salicylaldehyde were reported by Desai et al [67] as corrosion inhibitors.

In recent decade's interest have been created to Schiff base derived from heterocyclic amines due to their immense biological importance. Literature survey reveals that Schiff bases derived from heterocyclic amines are used as antitumor [68-69], anticancer [70-72], antibacterial [73-74], antifungal [75] and diuretic agents.

Schiff bases appear to be an important intermediates in a number of enzymatic reaction involving interaction of an enzyme with an amino group or a carbonyl group of the substrate [76], One of the most prevalent types of catalytic mechanisms in biochemical processes involves condensation of a primary amine in an enzyme, usually that of a lysine residue, with a carbonyl group of the substrate to form an imines, or Schiff bases.

Stereo chemical investigation [77] carried out with the aid of

molecular models showed that Schiff bases formed between methylglyoxal and the amino groups of the lysine side chains of proteins can bend back in such a way towards the N atoms of peptide groups that a charge transfer can occur between these groups and the oxygen atoms of the Schiff bases. In thisrespect, pyridoxal Schiff bases derived from amino acids have been prepared and studied [78]. Schiff bases derived from pyridoxal and amino acids are considered very important ligands from the biological point of view. Transition metal complexes of such ligands are important enzyme models. The rapid development of these ligands resulted in an enhanced research activity in the field of coordination chemistry leading to very interesting conclusions.

Certain polymeric Schiff bases have been reported which possess antitumor activity [79]. The antitumor activity of the Schiff bases towards ascetic tumors increases considerably with a slight increase in water solubility [80].

Transition Metal Complexes of Schiff Bases

Schiff base metal complexes have been widely studied as they possess significant biological activities [81-83]. Chelating ligands containing N, S and O donor atoms are of special interest because of the variety of ways in which they are bonded to metal ions. It is known that the existence of metal ions bonded to biologically active compounds may enhance their activities [84-86].

Metal-Schiff base complexes had been known since the mid nineteenth century [87]. They gain tremendous importance after the work of Jorgensen and Werner [88] Ettlings isolated a dark green crystalline product from the reaction of cupric acetate, salicylaldehyde and aqueous ammonia. H. Schiff prepared metalcomplexes of Schiff bases obtained by the reaction of salicylaldehyde with primary amines, and urea respectively [89-91]. Delepine [92] prepared complexes by reacting metal acetate, salicylaldehyde and a primary amine in alcohol and demonstrated a 2: 1 stoichiometry. However, there was no comprehensive, systematic study until the preparative work of Pfeiffer and associates [93]. Pefiffer and his coworkers reported a series of complexes derived from Schiff bases of salicylaldehyde [94-103]. Dubsky and Sokol [104] isolated N-N-Bis-Salicylidene ethyledimine Cu (II) and Ni (II) complexes and formulated their structure.

The chemistry of Schiff base complexes has developed rapidly in the last 30 years, solving problems related to stereochemistry and fertilizing related field in coordination chemistry. Stereochemistry and electronic factors largely govern the reactivity and stability of chelate compounds [105]. The stereochemistry and the electronic structure of chelates determine many properties, such as their redox properties reactivity and ability to form adducts [106]. These properties are important for understanding manycatalytic processes and phenomenone occurring in living organisms [107]

Sulphonamides

Sulphadrugs are a group of compounds used for eliminating a wide range of infections in human and other animal system. Many chemotherapeutical important sulpha drugs

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possess SO2NH moiety as an important toxoforic functional group. They are frequently used in medicine on account of their anti bacterial activity. They inhibit some enzyme reactions vital to bacteria and this hinders the formation of bacterial wall [108-109]. They possess the following general structures:



Structure of sulphonamide

Biological activity of sulpha drugs depends upon the free or potentially free Para amino group. The Sulphonamides containing this group either free or substituted by such group which can be converted in to free amino group in body. All sulphur drugs exert bacteriostatic effect rather than bactericidal effect on the micro organisms. Some important sulpha-drugs are given below.



Sulpha drugs had attracted special attention dueto their therapeutic importance as they were used against a wide spectrum of bacterial ailments and used against most of the gram positive and some of the gram negative bacteria, These drugs were the first efficient treatment to be employed systematically for the prevention and cure of bacterial infections. In most instances, they are bacterial static that is, they inhibit the growth and multiplication of the bacteria, but do not kill them [110-112]. Several metal based sulphonamides have been reported as potentially biologically active [113-114].

Literature on hydrazones

Hydrazone is an important class of chemical substances used for the treatment of several disease and found applications in synthetic and analytical chemistry [115-117]. These compounds containing grouping –NH-, N=C< can be distinguished from other classes by the presence of two interlinked nitrogen atoms. In analytical chemistry hydrazones have been used for identification and detection of large number of metal cations.

Biological formation and reaction of hydrazono compounds got further prominence after the use of -Isoniazid as an antitubercular agent. During treatment of tubercular patients with hydrazono compound -- Iproniazid , the drug was found to stimulate the central nervous system and inhibit theactivity of hydrazonoamine oxidase (MAO) both in vivo and invitro. Furthermore hydrazono exhibit physiological activities in the treatment of several disease [118-122]. This activityes conributated to the formation of stable chelate complexes with transition metal which catalyses physiological process [123-124]. They also act as herbicides, insecticides, nematocides, rodenticides, plant growth regulators, and sterins for house flies. They also have applications in analytical chemistry and used in colorimetric and fluorimetric determination [125-126]. Terra et. Al report synthesis and characterization of hydrazono Schiff bases compound, Schiff bases are used as substrate in the preparation of long bio-active and industrial compound via ring closure, cycloaddition, replacement reactions. In addition several hydrazono Schiff bases are well known to have biological activities [127-129].

Chemistry of transition metals Chemistry of Titanium

Titanium has an electronic configuration of (Ar) $3d^24S^2$. Titanium exhibits oxidation states of +2, +3, and +4, in the compounds titaniummonoxide, TiO, dititanium trioxide, Ti2O3, and titanium dioxide, TiO2, respectively. The +4 oxidation state is the most stable.

The chemistry of titanium in the +2 state is rather restricted. By contrast, many compounds are formed by titanium in the +3 state. One of the most important is TiCl3, a crystalline form of which is particularly useful as a catalyst in the stereospecific polymerization of propylene to make the commercially valuable polymer polypropylene.

Among the compounds formed by titanium in its +4 state, the dioxide, TiO2, is the most important. This nontoxic, pure white powder is used extensively as a pigment in paints, enamels, and lacquers. It occurs in nature as the minerals brookite, octahedrite, anatase, and rutile.

Chemistry of vanadium

Vanadium has an electronic configuration of [Ar]3d³4s² and can exist in eight oxidation states ranging from -3 to +5[130]. The three highest, oxidation state +3, +4 and +5, are important for biological systems [131-133]. Under ordinary conditions, the +4 and +5 oxidation states are the most stable [130]. The coordination chemistry of vanadium is strongly influenced by the oxidation/reduction properties of the metal centre and the chemistry of vanadium ions in aqueous solution is limited to oxidation states of +2, +3, +4 and +5. Vanadium compounds of oxidation state of +2 and +3 are unstable to air and their compounds are predominantly octahedral. Many oxovanadium (V) complexes contain the VO2 + entity and the cis geometryin dioxo complexes have been confirmed by structural determination [134]. The oxo complexes of the halides, alkoxides, peroxides, hydroxamates and amino carboxylates have been

characterized [135-136]. Compounds of vanadium (IV) which are not of vanadyl type include tetra halides.

Chemistry of Chromium

Chromium is a member of the transition metals, in group 6. Chromium has an electronic configuration of $[Ar]3d^54S^1$, owing to the lower energy of the high spin configuration. Chromium exhibits a wide range of possible oxidation states, where the +3 state is most stable. The +3 and +6 states are most commonly observed in chromium compounds, whereas the +1, +4 and +5 states are rare.

Chemistry of Cobalt

Cobalt has an electronic configuration [Ar] $3d^74S^2$ in its compounds cobalt nearly always exhibits a +2 or +3 oxidation state, although states of +4, +1, 0, and -1 are also known. The compounds in which cobalt exhibits the +2 oxidation state (Co^{2+} , the ion being stable in water) are called cobaltous, while those in which cobalt exhibits the +3 oxidation state (Co^{3+}) are termed cobaltic. Both Co^{2+} and Co^{3+} form numerous coordination compounds, Co^{3+} forms more known complex ions than any other metal except platinum. The coordination number of the complexes is generally six.

Physicochemical techniques

1. Magnetic susceptibility

For studying the electronic structure of transition metal complexes, the measurement of magnetic moment is a very useful method. It provides fundamental information about the bonding and stereochemistry of metal complexes. The magnetic properties of coordination compounds are based on the effect of ligands on the spectroscopic terms of metal ions [137]. The Gouy method is the simplest method of measuring magnetic moments. It consists of a suspension of a uniform rod in a non homogeneous magnetic field of about 5000 Oersteds and measuring the force exerted on it by conventional weighing technique. The caliberants usually used are Hg [Co (SCN) 4] and [Ni (en) 3S2O3] which are easy to prepare, do not decompose or absorb moisture and pack well in the sample tube. Their susceptibilities at 20 °C are; 16.44 x 10⁻⁶ and 11.03 x 10⁻⁶ c. g. s. units, respectively and may decrease from 0.05×10^{-6} to 0.04×10^{-6} per degree risein temperature.

All substances those possess magnetic properties are affected by the application of a magnetic field [138]. The substances may be diamagnetic when an apparent reduction in mass is caused in the applied magnetic field and paramagnetic when an apparent increase in mass is caused in the magnetic field. The molar susceptibility is a measure of magnetic field, of a substance. It is an algebraic sum of the susceptibilities of all the constituent atoms, ions or molecules. The susceptibility per gram atom of a paramagnetic metal ion in a particular compound is determined by measuring the molar susceptibility of the compounds and applying diamagnetic corrections for the other ions or molecules in the compound. The diamagnetic corrections can be estimated by various methods [137-138]. Pascal's correction gives satisfactory results for inorganic compounds.

1.1 The theory of magnetic susceptibility

When a substance is placed in a magnetic field of strength H, the magnetic induction or density of lines of force, B, within the substance is given by:

$$\mathbf{B} = \mathbf{H} + 4\pi \mathbf{I} \qquad (\mathbf{i})$$

Where I is the intensity of magnetization or magnetic moment per unit volume and the term $4 \pi I$ is a contribution to B by the substance itself. Dividing eq. (i) by H gives:

$$P = 1 + 4 \pi \kappa \qquad (ii)$$

$$(P=B/H, and K = I/H)$$

Where P and κ are the magnetic permeability of the material and the magnetic susceptibility per unit volume respectively, which may be considered dimensionless.

Thus the volume susceptibility of a vacuum is zero, since in a vacuum B/H = 1. The volume susceptibility of a diamagnetic substance is negative while paramagnetic substances have positive susceptibilities. In practice, susceptibility is usually expressed per unit mass (gram susceptibility) rather than per unit volume.

$$g = \kappa/\rho$$
 (iii)

where $\chi g = \text{gram susceptibility} = \text{density of the substance in gcm}{-1}$

$$\chi m = \chi g M$$
 (iv)

where M is the molar mass of the sample. Similarly, χa and $\chi a+$ refer to the atomic and ionic susceptibilities respectively and can be obtained from equation (v).

$$\chi a = \chi m - \Sigma \chi L$$
 (v)

where χL is the molar susceptibility of all other constituents of the ligands (diamagnetic correction). The magnetic moment is given by

$$\mu eff = 2.828 \sqrt{\chi aT BM} \qquad (vi)$$

where T is the temperature in K. Experimental values of χa are very small and are generally independent of both field strength and temperature.

1.2 Types of magnetic behavior

There are four type of magnetic behavior shown by complexes of transition metal ions viz. diamagnetic, paramagnetic ferromagnetic and anti ferromagnetic

If P < 1 (i. e. I, κ and χ are negative) the substance is said to be diamagnetic. It causes a reduction in the intensity of the magnetic field and in an in homogenous field moves to the region of lowest field strength. The molar susceptibility of diamagnetic substances is very small and negative (-1 to 100 10^{-6} c. g. s. e. m. u) and are usually independent of field strength and temperature. Diamagnetism is a property of all matter and arises from the interaction of paired electron with the magnetic field. Diamagnetic susceptibilities of atoms in molecule are additive; and this is of particular use in estimating the diamagnetic susceptibilities of ligand atoms and counter-ions in a transition metal complex [138]. Additively of atomic susceptibility is contained in the Pascal's constants.

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Para magnetism results when P >1 i. e. I, κ and χ are positive, and it causes an increase in the intensity of the field and in an in homogenous field it moves to the region of highest field's strength. Paramagnetic susceptibilities are positive and relatively large (100 to 100, 000 10^{-6} c. g. s. e. m. u). They are independent of field strength but they are temperature dependent. However, temperature independent paramagnetism (TIP) can arise in system containing unpaired electron due to the coupling of the ground states with the excited states under the influence of a magnetic field. Ferromagnetism and anti-ferromagnetism are both special classes of paramagnetism. They arise from the interaction of individual paramagnetic species with one another. In anti-ferromagnetism, the magnetic vectors of the neighbouring centers tend to couple anti-parallel so as to cancel one another. It reduces the susceptibility and hence magnetic moment of a compound while in ferromagnetic substances the moments of the separate ions tend to align themselves parallel and thus to reinforce one another. They are both temperature and field strength dependent.

1.3 Magnetic properties of transition metal complexes

For ions of the first transition series, the magnetic moments due to electron spin (spin only moment) is given by the formula:

$$\mu so = [4S (S+L)]^{1/2}$$
 (vii)

Where S = sum of the spin quantum numbers, $s = \pm$ hence the number of unpaired electrons n=2S

Equation VII can therefore be rewritten as;

$$\mu$$
so = [n (n+1)]¹/₂ (viii)

Where there is orbital motion, the magnetic moment can be written as: $\mu S+L = [4S (S+L) + L (L+1)] \frac{1}{2} ix$

2. UV-Visible spectrophotometry

The excitation of a molecule from its electronic ground state to an electronic excited state corresponds to absorption of light in the near-infrared, visible or ultraviolet regions of the spectrum. For transition metal complexes, the absorption bands in the first two of these regions (infrared and visible) are relatively weak and are associated with transitions largely localized on the metal atom. The ultraviolet bands are intense and they are associated with the transfer of an electron from one atom to another and so are called chargetransfer bands.

The spectra of transition metal complexes arises due to transition of unpaired electrons from the ground state to an excited state. Transitions may occur between the split d-levels of the central atom, giving rise to the d-d or ligand field spectra. The spectra region where these bands occur spans the near infra-red, visible and UV. Most of the transition metal complexes are coloured due to d-d transitions in the visible region. The atomic overlap in metal—ligand bonds allows d electrons to penetrate from the central atom to the ligand, and *vice versa*. The transitions are affected by the effect ofligands on the energies of the d orbital of the metal ions. Since octahedral, square-planar and

tetrahedral fields cause splitting of d orbitals in different ways, the geometry will have a pronounced effect on the d-d transitions in a metal complex. Thus spectral data of transition metals provide useful information about the structure of complexes

3. Powder X-ray diffraction

X-ray powder diffraction (**XRD**) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined.

X-ray powder diffraction is most widely used for the identification of unknown crystalline materials.

Aim and objectives of the Research

It is apparent from literature review that Schiff bases possess several important biological activities. The azo methine linkage (–N=CH) is a significant feature that makes Schiff base an important biological active species as well as ligands for co-ordination. It is well accepted that metal ions play an important role in the enhancement of biological activity of organic compounds/ligands. The interaction between metal ions and such biologically active ligands represents an important route in designing new metal based antibacterial, antifungal and anticancer therapies against different kinds of bacteria, fungi, and associated viruses that become resistant to the use of conventional drugs.

Therefore it is worthwhile to synthesis some new Schiff base ligands derived from diketones with sulphonamoyl azo moiety and heterocyclic amines. The study of biological activity of new Schiff bases ligands vi a co-ordination to transition metal ions Ti (III), V (IV), Cr (III), Co (III), is carried out with expectation that these studies result is achieving new targets, in synthesizing/designing of metal base compound that could fight more aggressively against such bacterial/fungal studies which become resistant to certain presently available and commonly used anti microbial agents.

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