Chapter 1

Introduction

1 Introduction

1.1 The Vanadium Element

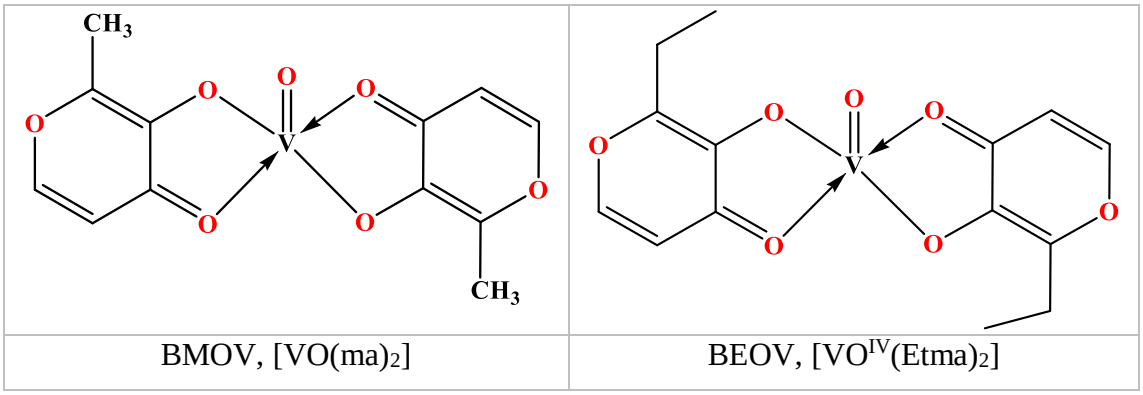
Vanadium (Z = 23) is a transition element and placed in the fourth row in the periodic table with electronic configuration [Ar]$3d^3 4s^2$. It was discovered in 1801 by Del Rio's and rediscovered severally in 1831 by Swedish Chemist Nils Gabriel Sefstrom. Sefstrom named this element vanadium after the Scandinavian goddess Vanadis. It is widely distributed in nature and forms up to 0.02% of the earth's crust with 60 ores. Vanadium metal is hard and steel-grey metal with two naturally occurring isotopes namely ^{51}V (99.76%) and ^{50}V (0.24%). It is mostly used in the steel industry as an additive. Its compounds are useful and used as catalysts, in ceramics, pigments, batteries and industries. This element has got nuclear applications, as rust-resistant elements, in manufacturing superconductive magnets along with within manufacturing of high-speed steel and iron made tools.

Vanadium exists in several oxidation states *viz.*, -1, 0, +2, +3, +4 and +5. Vanadium pentoxide (V_2O_5) is the most common and working form of vanadium. Other commonly used vanadium salts are ammonium metavanadate (NH_4VO_3), sodium metavanadate ($NaVO_3$) and sodium orthovanadate (Na_3VO_4). The toxicity of vanadium is popular and its toxicity differs significantly due to the compound's nature. Vanadium pentoxide is the most toxic and mobile form [1].

Vanadium is an essential element in biological systems. Vanadium is mainly complexed *in vivo*. It easily couples with proteins like transferrin, albumin, haemoglobin and glutathione [2]. It is also nutritionally essential in mammals, but its specific biological role in human is not much explored. The required quantity of vanadium is ca. 100-200 μg [3]. This element is equally useful for the growth and development of goats, rats, mice and chicks [4]. The use of vanadium in humans was suggested in the case of pathological states such as malnutrition, anaemia, tuberculosis and diabetes. It is tested that the diabetic patients prescribed sodium metavanadate excreted less glucose in urine [5].

The inhibition of catalyst activity is thought to be one amongst the effective methods for the treatment of diabetes. Inorganic vanadate complexes have been proposed to have potential in the inhibition of α-glucosidase [6], α-amylase [7] and glucose-6-phosphate [8]. It has been found that athletes took vanadium sulphate as a supplement at doses up to 60 mg/day to increase the weight during training. The toxicity of vanadium compounds is low

due to quick excretion in urine [9]. Although the commercial applications of inorganic compounds are limited due to their poor absorption and fast excretion in the gastrointestinal tract in the human body [10, 11]. Therefore, inorganic chemists shifted their research views to the organic vanadium complexes that have higher absorptive capacity than inorganic vanadium. Encouraging complexes to consist of neutral $V^{IV}O$ species with bidentate anionic ligand (L^-) with composition VOL_2; for example $[VO^{IV}(maltato)_2]$ (BMOV) and $[VO^{IV}(Etmaltato)_2]$ (BEOV) (Fig. 1).



BMOV, $[VO(ma)_2]$	BEOV, $[VO^{IV}(Etma)_2]$

Fig. 1. Vanadium complexes (insulin mimetics).

BMOV is a benchmark complex for the new molecules with antidiabetic feature [12, 13]. These complexes are more effective in lowering the glucose level in blood serum than the inorganic vanadium compounds and are well tolerated in all the animal model of diabetes. Several vanadium complexes with Schiff bases have been reported and their potential in insulin enhancement has been explored [14-17].

Schiff bases are the well-known organic compounds as a result of their manifold biological activities, synthesized by the condensation of aldehydes or ketones and primary amines. This exclusive property is due to the presence of the azomethine linkage and present hetero atom [18, 19]. Recent studies on the antidiabetic activity of Schiff bases and their complexes have also been tested in animal models, resulting in a significant reduction in blood glucose level and also altered biochemical parameters with improved glucose balance in rats [20, 21].

1.2 Coordination chemistry of vanadium

The coordination chemistry of vanadium is dominated with its variable oxidation states which range -I to +V states. A large number of vanadium compounds have been synthesized and characterized using various physicochemical techniques. Most of these vanadium complexes are in higher oxidation states. Many review articles appeared on general coordination chemistry [22-25], structural advances [26], aqueous vanadate chemistry [27] and in other biological systems. Vanadium is important in both reductive and oxidative catalytic transformation in biological systems [28-30].

The blue $[VO(H_2O)_5]^{2+}$ cation is prevalent in the aqueous chemistry of vanadium(IV) and used as beginning material for the synthesis of vanadyl complexes [31,32]. The VO^{2+} (vanadyl ion) is one of the most stable species and most stable diatomic ion with the ground state electronic configuration $[Ar]3d^1$ and exhibit similarities to the Cu^{2+} d^9 system. EPR spectroscopy is often used to detect the VO^{2+} ion and its complexes. The unpaired electron ($3d^1$) coupled to ^{51}V (I = 7/2) to give eight lines with many fold EPR spectra in frozen solution. The hyperfine coupling constant (A) and g-tensor are sensitive to the ligand field of the vanadium atom [33].

The majority of vanadium(IV) complexes have magnetic moment ~ 1.78 B.M. closed to spin only value. In these complexes, vanadium ion is not involved in a magnetic exchange with the neighbouring metal ions through exchange forces [1]. Vanadyl complexes are generally synthesized with ligands having O and N atoms. Vanadyl complexes formed are green or blue-green. The most starting salt for the synthesis of these complexes is $VOSO_4.5H_2O$. $[VO(acac)_2]$ is also often used in the synthesis of vanadyl complexes.

Most of the vanadium complexes in the highest oxidation state(V) contain oxygen and fluorine and the electronic configuration remains $3d^0$ that the complexes are diamagnetic. The diamagnetic behaviour of vanadium(V) complexes has allowed extensive study by ^{51}V NMR spectroscopy [34]. ^{51}V NMR spectroscopy is a useful diagnostic tool for the detailed study of vanadium(V) coordination environment.

1.3 Insulin-mimetic vanadium compounds

Diabetes mellitus (DM) is a disease that outcomes in chronic hyperglycaemia due to lack of insulin and associated with impaired glucose and fatty acid metabolism [35]. DM is

generally classified as type 1 and type 2 diabetes. According to world health estimates, up to ~10% of the world population is suffering from diabetes [36]. Type 1 diabetes, advances to ~10% diabetes cases, is due to highly reduced production of insulin. This kind of diabetes generally caused as a result of an autoimmune reaction that destroys the β-cells or it may be due to damage of the pancreases by an accident. Type 2 diabetes usually advances in the age of ~60 years. In old age, insulin is still produced, but the insulin receptors of the tissue cells no longer respond appropriately to insulin. Insulin is a signalling hormone, which is necessary for the metabolism of carbohydrate and fat easily. The insulin-like effects of vanadium compounds were reported long ago [36]. In this context, several vanadium(IV/V) complexes have been designed and tested [37].

Maltol is a commonly occurring compound and a recommended food supplement in most countries. Fig. 2 gives an overview of the uptake and distribution of vanadium compounds in the human body. On administration these compounds partly decompose in strong acidic condition (pH≈2) in the stomach; the potentially destructive effects of such acidity can be circumvented by drug encapsulation. The medium of saliva and small intestine remains slightly alkaline hence, in addition to the expected speciation of the originally applied prodrugs, which passing through these organs with distinct pH-condition, the possible loss of ligands, oxidation and reduction happened as unavailable conditions. Moreover, the original vanadium compounds may have changed upon interaction with low and high molecular weight constituents, which may act as ligands. These constituents are abundantly present in the nutritional compounds along in body fluids and secretions of body fluids stimulated by the intake of found stuff, in reality, vanadium compounds may be changed into sample compounds *viz.*, (I) VO^{2+} salts, possibly then forming insoluble $VO(OH)_2$, which will not be observed and reported out with faces (II) vanadium(IV) compounds, $H_2VO_4^-$. This anion is expected to be observed in the small intestine and hence distributed throughout the body.

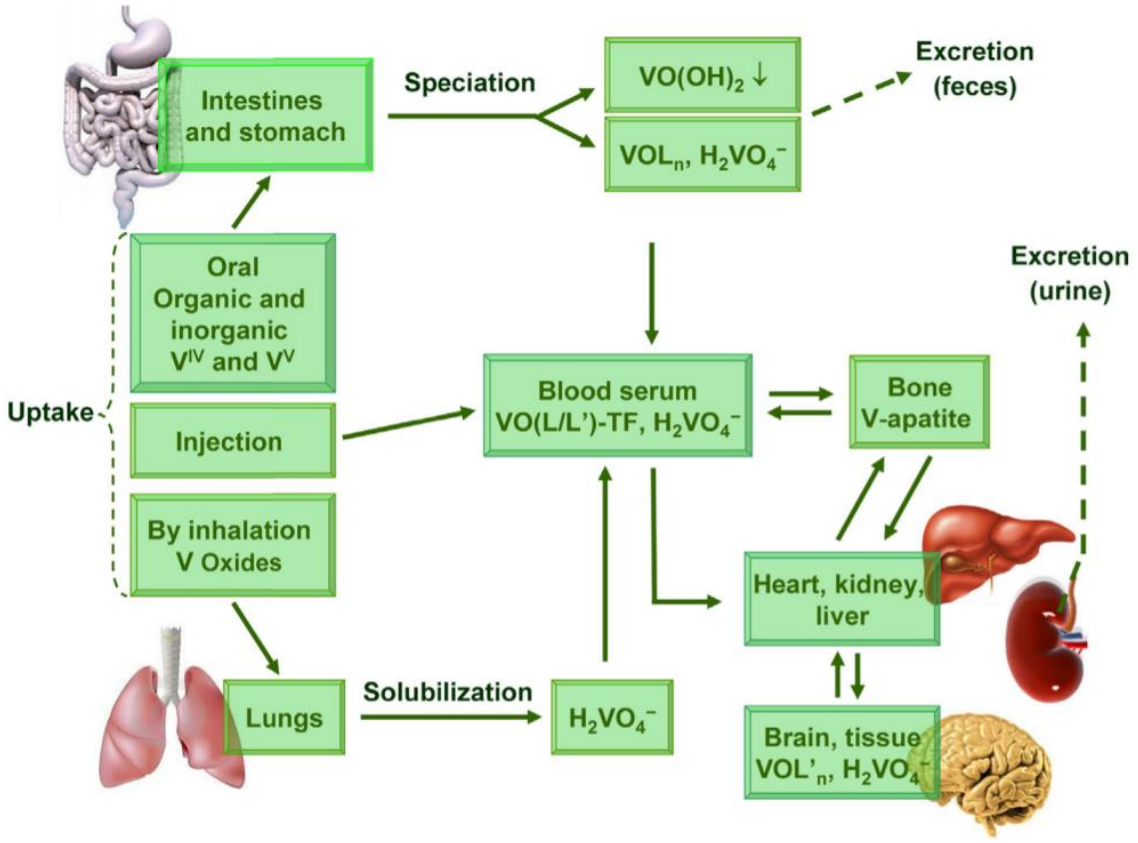
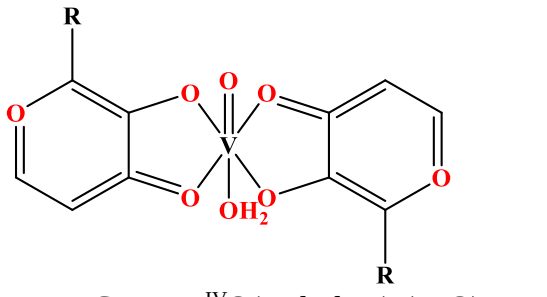
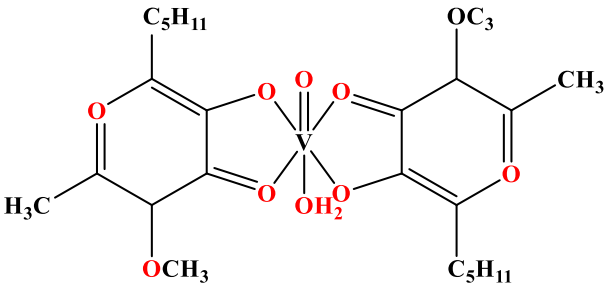


Fig. 2. Absorption and distribution of vanadium compounds in the body.

Vanadium compounds $VO(carrier)_n$, with organic carrier ligands (Fig. 3), is an alternative in speciation when entering the body. At low and high pH values the decomposition of $VO(carrier)_n$ may take, the compounds being converted to another distinct compound. Alternatively, the body's ligands (L') can cause reorganization in the coordination sphere, forming VOL'_n or $VO(carrier)_n$ species and finally altering the absorption properties.



BMOV: R = CH_3 = $V^{IV}O(maltolato)_2(H_2O)$ 1a
BEOV: R = $C_2H_5V^{IV}O(ethylmaltolato)_2(H_2O)$ 1b



$V^{IV}O(allxinato)_2(H_2O)$ 2 = $VO(alx)_2$

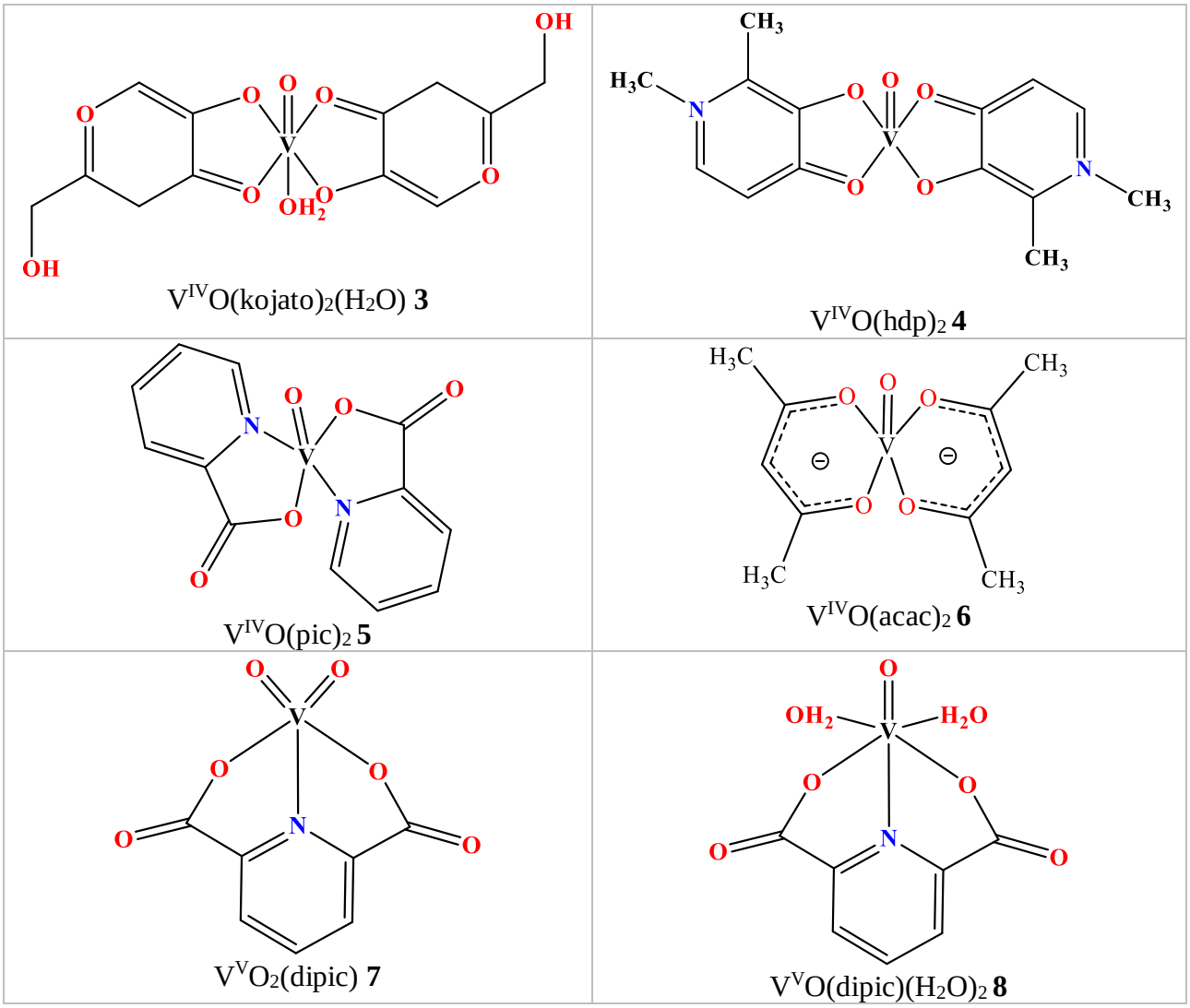


Fig. 3. Some vanadium compounds have been reported to exhibit insulin-like effects.

1.4 Vanadium in the treatment of cancer

An inorganic and organic form of vanadium has been examined in human [38]. The doses of vanadium compounds are usually comparatively lower than those in other animals like rat mice etc. Therefore, only moderate improvements in insulin and glucose metabolism were observed within a few weeks upon the start of the trails. The major disadvantage seen was gastrointestinal distress as only a small amount of vanadium is absorbed generally less than 2% of an oral dose. In this context, a major goal of the research has been the design of appropriate ligands to improve absorption and decrease of close required [39].

Recently the anticancer properties of vanadium compounds have been suggested but the basic mechanics are not well explored. The main targets for antitumor effects of vanadium complexes are the disruption of cellular metabolism through the generation of reactive oxygen species, the alteration of cellular organelles such as lysosomes, mitochondria. Besides, cell proliferation can also be disrupted by the genotoxic effects of vanadium exerted at the nuclei on the cells and on DNA damage. Some vanadium complexes may act as inhibitors of cell proliferation in different cancer cell lines [40]. Such vanadium complexes were tested *in vivo* and *in vitro* for the identification of model vanadium complexes (inhibitors). Although the clinical trials *in vivo* have been performed on mice. Several vanadium complexes as therapeutic have been synthesized and characterized. Among these vanadium(IV) compounds, Metvan was recognized as one of the most promising multitargeted anticancer vanadium compounds (Fig. 4). Metvan activated apoptosis in different tumoral cells lines of human such as leukaemia cells, multiple myeloma cells and solid tumour cells, breast cancer, ovarian, prostate and testicular cancer patients [41,42]. One more advantage of Metvan is that it is potent against ovarian cancer and testicular cell lines which are resistant to cisplatin. In cancer cell lines Metvan generates reactive oxygen species and causes depletion of glutathione along with loss of mitochondrial transmembrane potential.

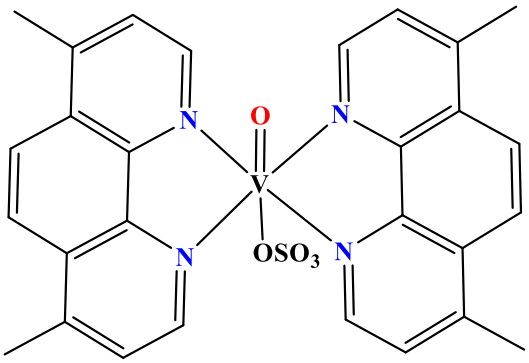


Fig. 4. Structure of Metvan.

1.5 Other therapeutic applications of vanadium compounds

Many vanadium compounds have been reported as antiparasitic, antiviral, antibacterial and antifungal vanadium compounds. Vanadium compounds act as expected agents *Trypanosoma cruzi*, *Leishmania ssp.* and *Entamoeba histolytica*, the protozoan parasites that are the causing agent of American trypanosomiasis (Chagas disease),

leishmaniases and amoebiasis, respectively [43]. Till now only four drugs are reported for the treatment of human African trypanosomiases *viz., pentamidine, suramin, melarsoprol* and *eflornithine* [44, 45].

Sun et al. [46] synthesized vanadium thiourea complexes and vanadium substituted polyoxometalates which exhibited the potent HIV properties towards the infected immortalized T-cells [46, 47].

The antiviral and antifungal features of vanadium complexes have also been examined. Antibacterial study of different polyoxometalates against Helicobacter pylori was reported [48]. Similarly, some vanadium complexes showed antifungal *viz., Aspergillus niger, Colletotrichum falcatum, Colletotrichum pallescence* and antibacterial *viz., Escherichia coli, Salmonella typhi* and *Bacillus subtilis* activities [48].

Ciprofloxacin is an antibacterial metallic element complexes with antibiotic drug substance were synthesized and tested for his or her in vitro medicine activity [49]. These metallic element complexes showed vital medicine activity because of the complexation of this antibacterial.

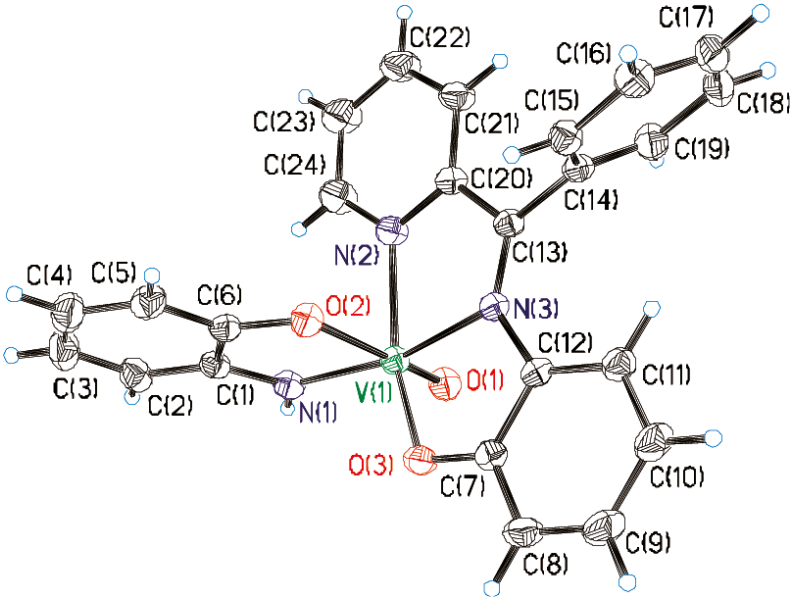
2 Brief Review

Recently, vanadium coordination chemistry and biological chemistry have been explored due to the discovery of vanadium as an essential element in biological systems and antidiabetic properties [50]. Several vanadium complexes possessing different coordination spheres have been suggested to have *in vitro* mimetic activity and *in vivo* antidiabetic blood lowering features [51-61].

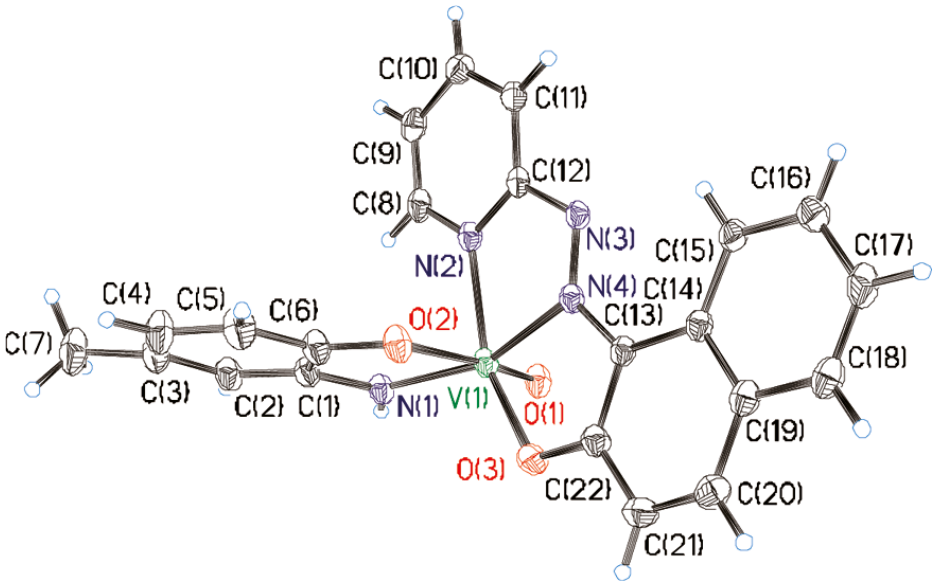
Pal et al. [62-67] have synthesized vanadium(V) complexes with tridentate Schiff bases derived from aroyl hydrazine. These complexes were fully characterized but biological experiments on these complexes were not carried out. The vanadium complexes with α-amino acid Schiff bases and NN donor phenanthroline bases as DNA binder cumphotosensitiser display efficient DNA binding capacity [67]. Synthesis and structural investigations of oxovanadium(IV) complexes of some aroyl hydrazones have been explored [68]. Vanadium(IV/V) complexes containing SNO donor sites have also been studies [69]. Several oxovanadium(IV) complexes with bidentate Schiff bases were synthesized and characterized by Rao and co-workers [70].

The synthesis of novel vanadium complexes bering different coordination sites has been studied [70-72] and their antidiabetic behaviour also investigated. Two kinds of new mixed ligand vanadium complexes have been synthesized and characterized using various physico-chemical techniques [73]. Inhibitory activities of these complexes against protein tyrosine phosphatise 1B (PTP1B), T-Cell protein tyrosine phosphatase (TCPTP) and SRC homology phosphates 1 (SHP-1) were performed. Studies on synthesis, characterization and protein tyrosine phosphates inhibition activities of oxovanadium(IV) with Schiff base (5-bromosalicylidine anthralanic acid) and polypyridyl derivatives (2,2-bipyridyl, 1,10-phenanthroline and their derivatives) has been investigated [74]. These complexes are a good example of protein tyrosine phosphate 1B (PTP1B) inhibitors for futures development as antidiabetic agents.

The experiments have been carried out that the biological activity of vanadium depends on its oxidation state [75-102]. The VO^{2+}-iminobenzosemiquinonate anion radical (**1** and **2**) incorporating tridentate NNO donor ligands have been synthesized and characterized by elemental analysis, FTIR, NMR and UV-Visible spectral measurements [103]. Single crystal structures have also been obtained from these complexes (Fig. 5).



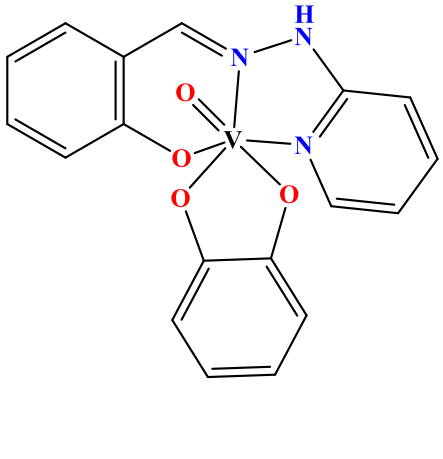
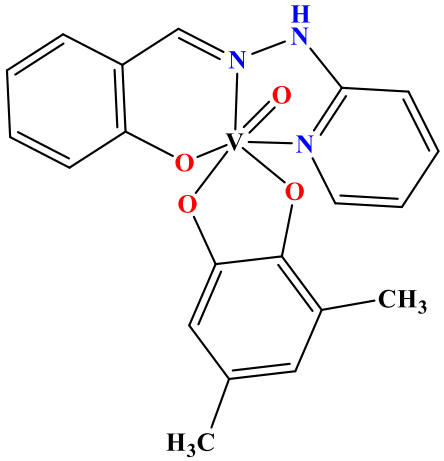
$(L_1^{-})(VO^{2+})(L^{H}{}_{IS}^{\bullet-})\bullet 3/2MeOH$ (1•3/2MeOH) **1**

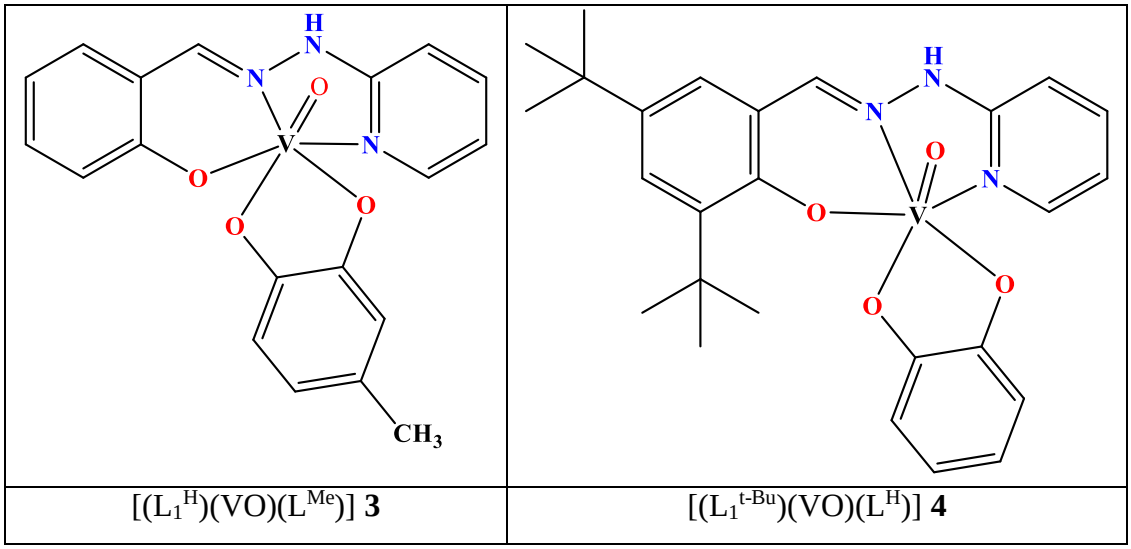


$(L_2^{-})(VO^{2+})(L^{Me}{}_{IS}{}^{\bullet-}).1/2L^{Me}{}_{AP}$ $(3\bullet1/2L^{Me}{}_{AP})$ **2**

Fig. 5. Molecular structures of complexes **1** and **2**.

Kundu et al. [104] have studied oxidovanadium catechol complexes. They have synthesized a new series of oxidovanadium complexes with tridentate ligand (2, 4-di-R-6-{(2-(pyridine-2-yl) hydrazone) methyl phenol} and substituted catechols (Fig. 6). These complexes are analysed by the electro and spectro electrochemical techniques, epr spectra, and DFT calculations. These complexes showed absorption bands at 800 nm due to the closed-shell singlet-open shell singlet (metal to ligand charge transfer (redshifted).

	
$[(L_1{}^{H})(VO)(L^{H})]$ **1**	$[(L_1{}^{H})(VO)(L^{t\text{-}Bu})]$ **2**



[(L_1^{H})(VO)(L^{Me})] **3**	[$(L_1^{t\text{-}Bu})$(VO)(L^{H})] **4**

Fig. 6. Molecular geometries of oxidovanadium catechol complexes **1-4.**

Aslam and co-workers [105] reported that the Schiff bases of hydrazone possess urease inhibitory activity. A pure competitive mechanism of inhibition was introduced by hydrazone complexes. Molecular docking studies of these complexes were also carried out. Zhang et. al. [106, 107] have also reported that the Schiff base complexes also exhibit urease inhibitory activity similar to other urease inhibitors [108-110].

A dioxidovanadium(V) complex of NNO donor Schiff base (H_2PPCH = N'-picolinonylpyridin-1-ium-2-carbohydrazonate) has been synthesized and characterized by elemental analysis and spectral technique [111] (Fig. 7). It potently inhibited PTP1B with IC_{50} 0.13 µM. It exhibited lower cytotoxicity than $VOSO_4$ used as a positive control. Protein tyrosine phosphates (PTPs) are a family of signalling enzymes that play essential roles in controlling cell proliferation, communication and adhesion [112-114]. PTP1B is also considered one of the best-validated targets for the therapeutic candidate of type II diabetes and obesity [115-119].

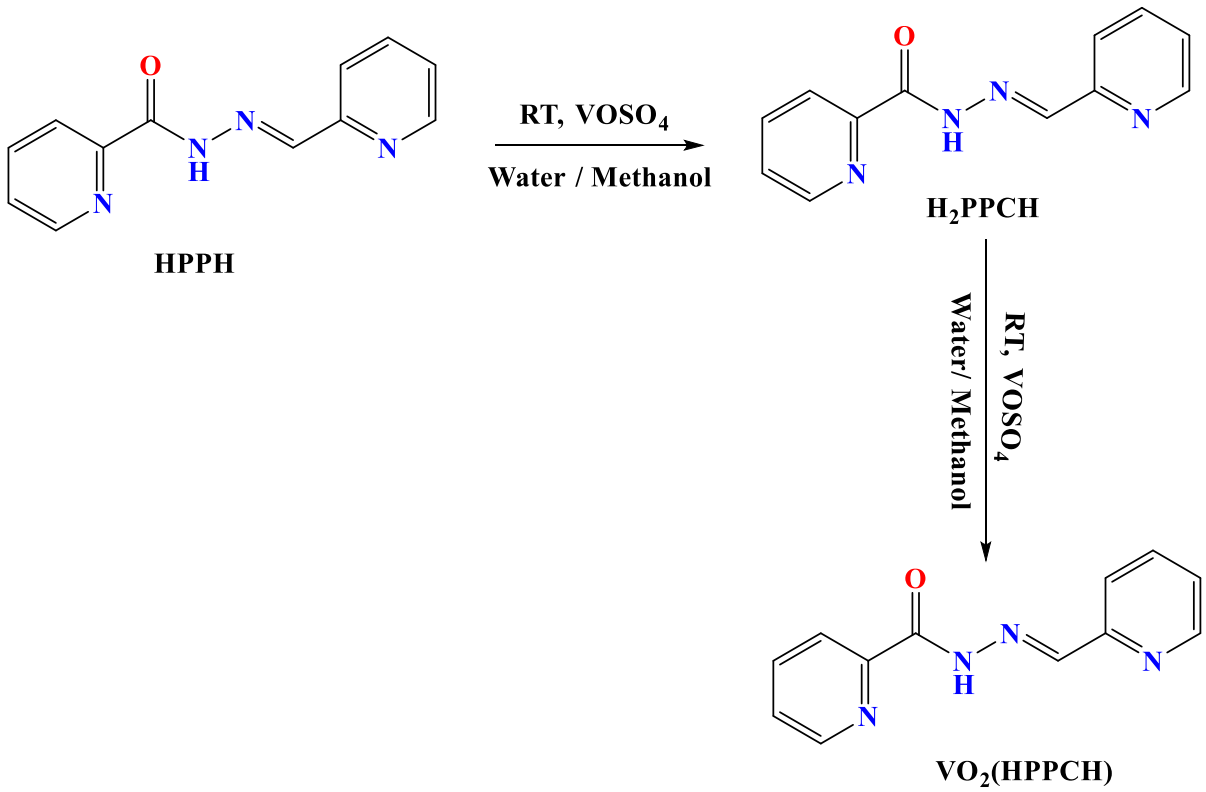


Fig. 7. Synthetic route of complex VO_2(HPPCH).

Mixed ligand vanadyl (IV) complex [VO(L^1)(L^2)] [L^1 =N'-[(z)-phenyl(pyridine- 2-yl)]methylidene benzo hydrazide and L^2 = benzo hydrazide was synthesized and characterized by microanalysis, spectroscopic and electrochemical techniques [120] (Fig. 8). This complex shows paramagnetic behaviour.

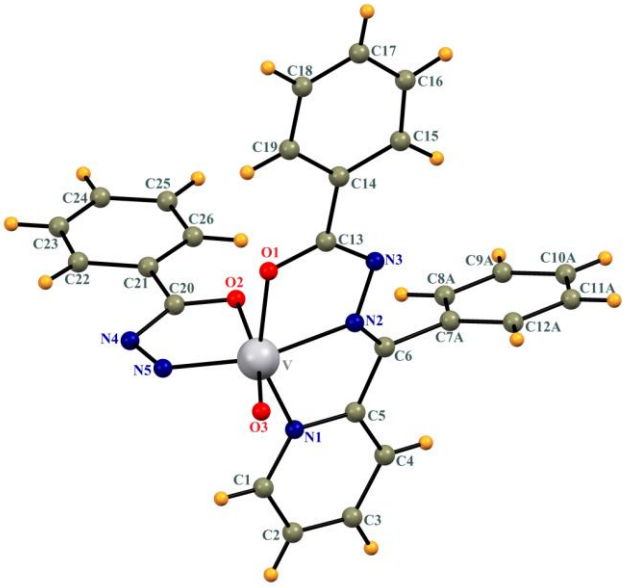


Fig. 8. Molecular structure of the complex [VO(L^1)(L^2)].

The complex was also tested for its anti-diabetic activity in vitro. This event shows moderate α- glucosidase inhibition. Hence, this may be considered as α-glucosidase [121-123].

A novel oxovanadium(IV) complex with tridentate Schiff base (L = 3-{3-bromo-2-hydroxophenyl}-{[1-(3-Bromo-2-hydroxyphenyl)methelidene]amino}methyl pentane-2,4-dione) and co-ligand 1,10-phenanthroline was synthesized and characterized [124]. The complex was tested for its antibacterial and antifungal activities by the MTT method. Such complexes have attracted considerable attention for their biological activities [125-130]. It was observed that complexes bearing the electron-withdrawing group can improve their biological activities [131, 132]. Vanadium complexes with tridentate Schiff base ligands have been reported to have interesting biological properties [133-135]. Rai et al. reported several halogens substituted complexes and observed that they have significant biological activities [136]. Qiu et al. [137] reported the antimicrobial activities of vanadium with hydrazones. A new homobimetallic vanadium(V) complexes were synthesized and structurally characterized using single-crystal X-ray analysis [138]. This complex was successfully employed as a functional catechol-oxidase mimics for the oxidation of 3, 5-ditart-butyl catechol to 3, 5-ditart-butyl-o-benzoquinone. This system followed Michalis-Menten kinetics concerning substrate.

New thiosemicarbazide and dithiocarbazate based oxovanadium(IV) and dioxovanadium(V) complexes have been studied [139] (Fig. 9). These complexes were characterized by various spectroscopic techniques. All these complexes show excellent catalytic activity and selectivity for the oxidation of benzyl alcohol and ethylbenzene in the presence of H_2O_2. Pessoa et al. [140, 141] reported the vanadium complexes of 2, 6-diformyl-4-methyl phenol derived polydentate ligands in solution as well as vanadium(IV) mononuclear complexes.

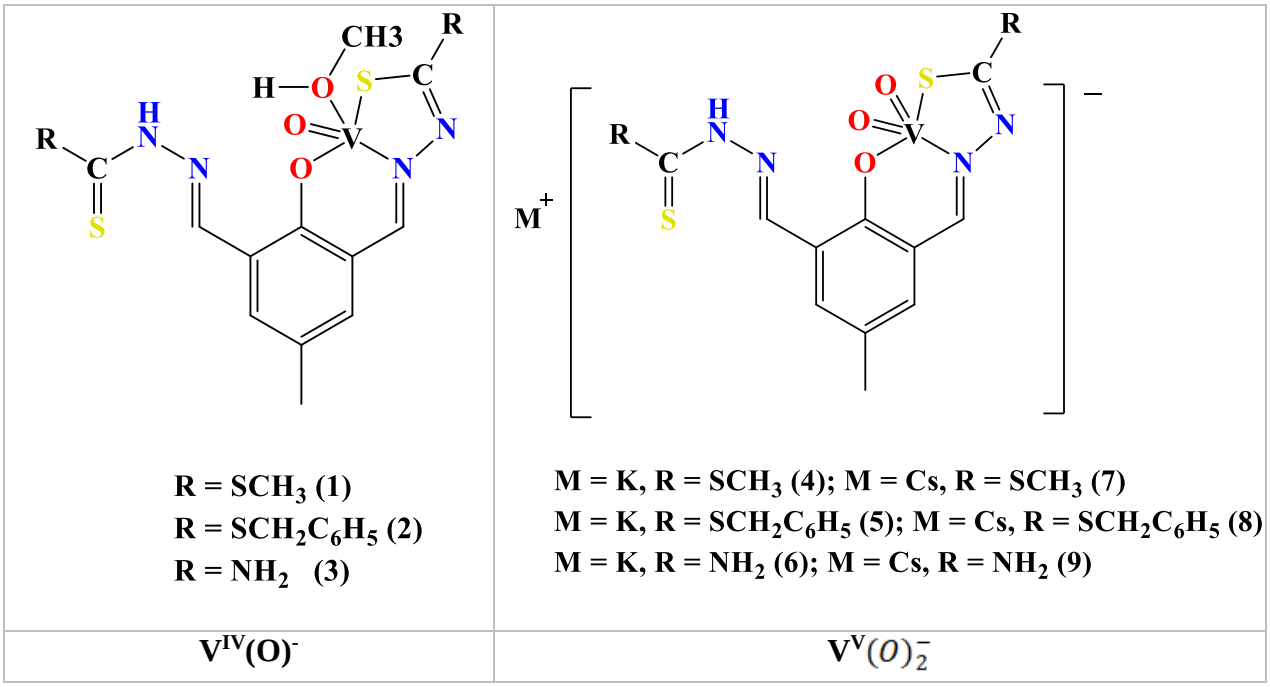
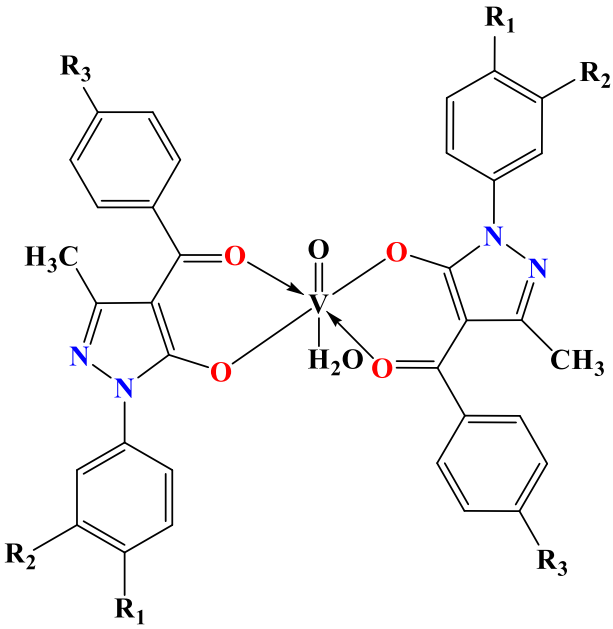


Fig. 9. The proposed structural formula of the $V^{IV}(O)^{-}$ and $V^{V}(O)_2^{-}$ complexes.

Parihar and co-workers [142] have reported the synthesis and crystal structure of a novel oxovanadium(IV) complex with 1-phenyl-3-methyl-4-toluyl-5-pyrazolone ligand and immobilized it over zirconia to use as a heterogeneous catalyst for the oxidation of styrene with hydrogen peroxide (Fig. 10). This research group [143] has also synthesized and characterized some more oxovanadium(IV) complexes with 4-acyl pyrazolone ligands. The catalytic activity of these complexes was explored for the oxidation of benzylic alcohols with H_2O_2 as an oxidant (Fig. 11). A possible pathway for the oxidation of benzylic alcohol was also suggested based on the spectral feature.



$VO(L^1)_2$: $R_1 = CH_3$, $R_2 = H$, $R_3 = H$
$VO(L^2)_2$: $R_1 = H$, $R_2 = H$, $R_3 = CH_3$
$VO(L^3)_2$: $R_1 = CH_3$, $R_2 = H$, $R_3 = CH_3$
$VO(L^4)_2$: $R_1 = Cl$, $R_2 = Cl$, $R_3 = CH_3$
$VO(L^5)_2$: $R_1 = Cl$, $R_2 = Cl$, $R_3 = H$

Fig. 10. Proposed structure for the synthesized complexes.

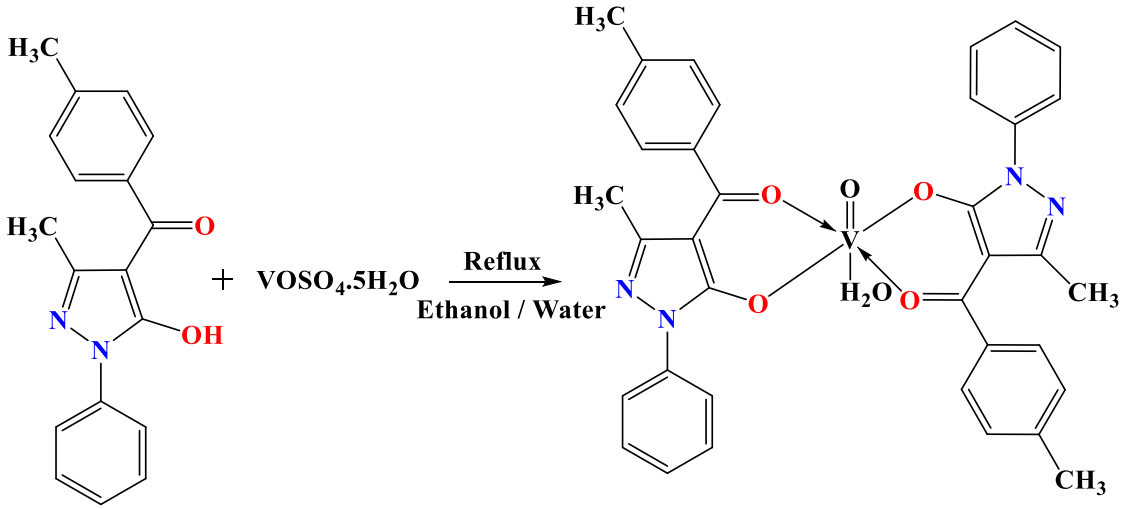
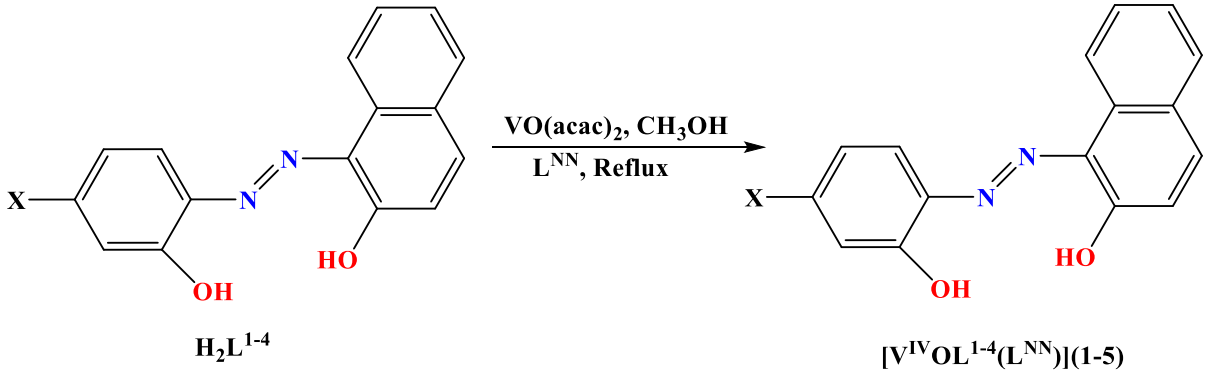


Fig. 11. Synthetic route for oxidovanadium complex.

A series of oxovanadium(V) complexes [VOLL'] L=ethyl malonate and L'= aroyl hydrazones, where synthesized and characterized by various physicochemical techniques [144]. The biological study of these complexes shows that they are insulin-like agents, which stimulates glucose utilization in a doses dependent manner. This research group also

synthesized and characterized a few more complexes to explore new drugs that enhance the properties of insulin [145-147].

Lima et. al. [148], reported synthesis, structure and biological evaluation of mixed ligand oxidovanadium(V) complexes incorporating 2-(aryl azo) phenolates (Fig. 12).



H_2L^{1-4} $[V^{IV}OL^{1-4}(L^{NN})](1\text{-}5)$

Ligand	X	L^{NN}	Complex
H_2L^1	H	Bipy	$[V^{IV}O(L^1(bipy)].(H_2O)$ (**1**)
H_2L^1	H	Phen	$[V^{IV}O(L^1(phen)]$ (**2**)
H_2L^2	CH_3	Bipy	$[V^{IV}O(L^2(bipy)]$ (**3**)
H_2L^3	NO_2	Bipy	$[V^{IV}O(L^3(bipy)]$ (**4**)
H_2L^4	Br	Bipy	$[V^{IV}O(L^4(bipy)]$ (**5**)

Fig. 12. Schematic route for the synthesis of $[V^{IV}O(L^{1–4})(L^{NN})]$(**1**–**5**).

The interaction of synthesized mixed ligand vanadium(IV) complexes with bovine serum albumin were also investigated. The antiproliferative properties of these complexes were also explored against A-549 (lung cancer) cancer cell lines by the MTT assay. Some research group studied vanadium(IV/V) complexes and their biological properties have been explored [149-160].

3 Aims and objective

Vanadium compounds showing insulin-like effects are generally known as insulin mimetics or insulin enhancing compounds. Simple vanadium salts like vanadyl sulphate or sodium metavanadate mimic antidiabetic compounds. The advantage of vanadium compounds relative to insulin is that they may be taken orally. Therefore, the aim of research

in the work field of vanadium chemistry is to make vanadium compounds that can approach the target cells with high efficiency. It is now believed that low molecular weight vanadium complexes, should be neutral with optimal lipophilicity to be mobile and cross the cell membrane easily. The VO(ethylmaltolato)$_2$ has successfully crossed the clinical phase I and II tests. It is found that the activities of the vanadium complexes were remains in the range of 30 ~70% of the activity of insulin. The inorganic salts due to very low oral bioavailability have been synthesized. In the thesis work, various oxidovanadium(IV/V) of Schiff base complexes have been synthesized and well-characterized using elemental analysis, UV-Vis, FTIR, EPR and the electrochemical (cyclic voltammetry and differential pulse voltammetry) techniques. Finally, these complexes were analysed by single crystal and powder X-ray diffraction techniques to get molecular structures of synthesized complexes. Quantum chemical calculations (DFT studies) suggest a similar structure and same mode for all synthesized complexes. Biochemical assay (antidiabetic activity) demonstrate that these vanadium complexes are potent inhibitors of insulin-mimetic agents. Additionally, a series of dioxidovanadium complexes also synthesized and characterized which are formed by *in situ* generated Schiff bases.

References

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