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Mixed Ligand Oxovanadium (IV) Complexes of 5,6-Dimethyl-1,10-phenanthroline and Amino Acid Schiff Bases Containing Salicylaldehyde: Synthesis and Characterization with Antibacterial Properties

Md. Nur Amin Bitu¹, Md. Saddam Hossain², Md. Ali Asraf³, Md. Faruk Hossen⁴ and Md. Kudrat-E-Zahan^{5*}

¹Department of Chemistry, University of Rajshahi, Rajshahi-6205, Bangladesh. E-mail: nabitu.ru@gmail.com

²Department of Chemistry, Begum Rokeya University, Rangpur-5400, Bangladesh. E-mail: saddamru4535@gmail.com

³Department of Chemistry, University of Rajshahi, Rajshahi-6205, Bangladesh. E-mail: asraf.chem@ru.ac.bd

⁴Department of Chemistry, University of Rajshahi, Rajshahi-6205, Bangladesh. E-mail: fhossen.chem@ru.ac.bd

⁵Department of Chemistry, University of Rajshahi, Rajshahi-6205, Bangladesh. E-mail: kudrat.chem@ru.ac.bd

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Abstract

Five new oxovanadium (IV) complexes have been synthesized from two mixed ligand, 5,6-Dimethyl-1,10-phenanthroline and amino acid derived Schiff bases containing salicylaldehyde. All the synthesized complexes were characterized by some physicochemical properties, molar conductance, magnetic susceptibilities measurements, elemental analysis, UV-Visible and IR spectral studies. The molar conductance values reveal the non-electrolytic nature of the complexes. The magnetic moment values indicate that the complexes are paramagnetic in nature. IR spectral data indicates the coordination of tridentate amino acid Schiff base ligands to the vanadyl (VO^{2+}) ion through O, N, O-donor. ESI-MS spectral data confirms the proposed molecular formula of the complexes. All the analytical data suggested that all the complexes possess to have distorted octahedral geometry. Antibacterial activity of the complexes was tested against two Gram positive bacteria *Escherichia coli* and *Pseudomonas aeruginosa* and two Gram negative bacteria *Staphylococcus aureus* and *Bacillus cereus* with Kanamycin (K-30) standard. The result shows that all the complexes possess to have moderate to strong potential antibacterial activity against all the tested pathogenic bacteria.

Keywords: Oxovanadium (IV) complexes, 5,6-Dimethyl-1,10-phenanthroline, Amino acids, Salicylaldehyde, Schiff base, Antibacterial activity

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1. Introduction

Schiff bases are the condensation products of primary amines and carbonyl compounds. They contain imine or azomethine ($-RC=N-$) group in their structural formula (Bitu et al., 2019). Amino acids are the significant class of organic-based compounds which may take part to prepare Schiff base by condensation of amine group (NH_2) with any carbonyl

* Corresponding author: Md. Kudrat-E-Zahan, Department of Chemistry, University of Rajshahi, Rajshahi-6205, Bangladesh. E-mail: kudrat.chem@ru.ac.bd

compound. They contain potential donor sites such as COOH and/or NH₂ group which have good ability to coordinate with the central metal ions. As an excellent complexing agent Schiff bases can form various complexes with several transition and inner transition metals. Schiff base ligands and their transition metal complexes have a variety of applications including biological, clinical, and analytical applications (Cheng *et al.*, 2010; Ran *et al.*, 2011; and Kudrat-E-Zahan *et al.*, 2019). Vanadium coordination chemistry is a topic of interest over the last few decades due to the role of vanadium in various biological processes, such as phosphorylation (Yue *et al.*, 2006), haloperoxidation (Macedo-Ribeiro *et al.*, 1999) enhancement of oxygen affinity of hemoglobin and myoglobin (Noblia *et al.*, 2004; and Pattanayak *et al.*, 2009) insulin mimicking, etc. (Tasiopoulos *et al.*, 1999; and Thompson *et al.*, 1999). Several studies on the synthesis of vanadium complexes of N-salicylidene amino acids have been reported, most of which were synthesized from amino acids and salicylaldehyde (Pessoa *et al.*, 2002; and 2004).

On continuation of the above studies, in our current research five new oxovanadium (IV) complexes containing two mixed ligand, 5,6-Dimethyl-1,10-phenanthroline and Schiff bases derived from amino acids and salicylaldehyde were prepared, characterized and investigated their potential antibacterial activity.

2. Materials and Methods

2.1. Reagents and Chemicals

All the reagents used were of chemically pure grade. Solvents were purified and dried according to standard procedures.

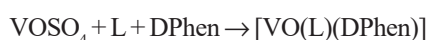
2.2. Physical Measurements

All the weighing operations was carried out by using A Mettler PM-200 electronic balance. The melting point of all the synthesized metal complexes were recorded in an electro thermal melting point apparatus (model No. AZ6512). Conductivities of 1.0×10^{-3} mol/dm³ solutions of the complexes in DMF were measured at 30 °C using a WPA CM 35 conductivity meter and a dip-cell with platinized electrodes. The magnetic moment value was measured by a SHERWOOD SCIENTIFIC Magnetic Susceptibility Balance. Infrared spectra were recorded as KBr disc in a SIMADZU FTIR-8400 (Japan) infrared spectrophotometer, at the Central Science Laboratory of Rajshahi University, from 4000-225 cm⁻¹. The electronic spectra of the complexes were recorded on SHIMADZU Spectrophotometer (Model UV-1800). The ESI-MS spectra were performed using a Shimadzu Ge Ms Qp 100 EX mass spectrometer using the direct inlet system. Carbon, hydrogen and nitrogen analyses for the complexes were carried out by PerkinElmer 2400 organic elemental analyzer-II.

2.3. General Method for the Preparation of the Oxovanadium (IV) Complexes of Type [VO(L)(DPhen)]

Oxovanadium (IV) complexes were prepared by template method in which a mixture of α -amino acids, α -alanine (ala) (0.267 g, 3 mmol)/DL-phenylalanine (pheala) (0.495 g, 3 mmol)/leucine (leu) (0.393 g, 3 mmol)/glycine (gly) (0.225 g, 3 mmol)/DL-methionine (met) (0.447 g, 3 mmol) and NaOH (0.12 g, 3 mmol) in 10 mL methanol were added to a methanolic solution of salicylaldehyde (sal) (0.31 mL, 3 mmol) in a round bottom flask. The resulting solution was refluxed for 1 hour, followed by the addition of a methanolic solution of vanadyl sulfate (0.489 g, 3 mmol). A light precipitate was obtained after refluxing the mixture for 1 h. To this mixture, 5,6-Dimethyl-1,10-phenanthroline (DPhen) (0.625 g, 3 mmol) taken in 10 mL methanol was added. The solution on further refluxing for 1 h gave a precipitate. The precipitate was filtered off on a Buchner funnel, washed with methanol and finally dried in a vacuum desiccator over anhydrous CaCl₂ (Sasmal *et al.*, 2007).

The general synthetic method is shown below:



where, L= N-salicylidene- α -alanine (sal-ala), N-salicylidene-DL-phenylalanine (sal-pheala), N-salicylidene-leucine (sal-leu), N-salicylidene-glycine (sal-gly), N-salicylidene-DL-methionine (sal-met) and DPhen=5,6-Dimethyl-1,10-phenanthroline.

2.3.1. Oxovanadium(IV) complex of α -alanine [VO(sal-ala)(DPhen)]

Color: Brown, Melting point: 217 °C, Yield: 0.825 g (59%), Molar conductance (Ohm⁻¹cm²mol⁻¹): 15.1, Magnetic moment (μ_{eff} /B.M.): 1.65, FT-IR (cm⁻¹): 3454br, ν (OH); 1620vs, ν (C=O); 1537m, ν (C=N); 1312m, ν (C-O); 959s, ν (V=O); 613m, ν (V-O); 456m, ν (V-N), UV-Vis. (λ_{max} /nm): 268-302, 363, 378. Elemental analysis for [VO(C₂₄H₂₁N₃O₃): M.W. 466.38 g/mol: Calculated: C, 61.81; H, 4.54; N, 9.01%. Found: C, 61.07; H, 4.35; N, 9.11%.

2.3.2. Oxovanadium (IV) Complex of DL-phenylalanine [VO(sal-pheala)(DPhen)]

Color: Orange, Melting point: 210 °C, Yield: 0.928 g (57%), Molar conductance ($\text{Ohm}^{-1}\text{cm}^2\text{mol}^{-1}$): 18.2, Magnetic moment ($\mu_{\text{eff}}/\text{B.M.}$): 1.58, FT-IR (cm^{-1}): 3435br, $\nu(\text{OH})$; 1621vs, $\nu(\text{C}=\text{O})$; 1542s, $\nu(\text{C}=\text{N})$; 1310m, $\nu(\text{C}-\text{O})$; 941s, $\nu(\text{V}=\text{O})$; 612m, $\nu(\text{V}-\text{O})$; 446 m, $\nu(\text{V}-\text{N})$, UV-Vis. ($\lambda_{\text{max}}/\text{nm}$): 269-302, 363, 375. Elemental analysis for $[\text{VOC}_{30}\text{H}_{25}\text{N}_3\text{O}_3]$: M.W. 542.48 g/mol: Calculated: C, 66.42; H, 4.65; N, 7.75%. Found: C, 65.19; H, 4.47; N, 7.58%.

2.3.3. Oxovanadium (IV) Complex of Leucine [VO(sal-leu)(DPhen)]

Color: Brown, Melting point: 225 °C, Yield: 0.901 g (59%), Molar conductance ($\text{Ohm}^{-1}\text{cm}^2\text{mol}^{-1}$): 10.3, Magnetic moment ($\mu_{\text{eff}}/\text{B.M.}$): 1.57, FT-IR (cm^{-1}): 3453w, $\nu(\text{OH})$; 1624w, $\nu(\text{C}=\text{O})$; 1533w, $\nu(\text{C}=\text{N})$; 1313w, $\nu(\text{C}-\text{O})$; 960s, $\nu(\text{V}=\text{O})$; 617m, $\nu(\text{V}-\text{O})$; 460w, $\nu(\text{V}-\text{N})$, UV-Vis. ($\lambda_{\text{max}}/\text{nm}$): 267-303, 364, 373. Elemental analysis for $[\text{VOC}_{27}\text{H}_{27}\text{N}_3\text{O}_3]$: M.W. 508.46 g/mol: Calculated: C, 63.78; H, 5.35; N, 8.26%. Found: C, 63.15; H, 4.92; N, 8.15%.

2.3.4. Oxovanadium (IV) Complex of Glycine [VO(sal-gly)(DPhen)]

Color: Reddish, Melting point: 213 °C, Yield: 0.855 g (63%), Molar conductance ($\text{Ohm}^{-1}\text{cm}^2\text{mol}^{-1}$): 16.5, Magnetic moment ($\mu_{\text{eff}}/\text{B.M.}$): 1.60, FT-IR (cm^{-1}): 3429br, $\nu(\text{OH})$; 1647s, $\nu(\text{C}=\text{O})$; 1538s, $\nu(\text{C}=\text{N})$; 1315m, $\nu(\text{C}-\text{O})$; 962s, $\nu(\text{V}=\text{O})$; 615s, $\nu(\text{V}-\text{O})$; 461m, $\nu(\text{V}-\text{N})$, UV-Vis. ($\lambda_{\text{max}}/\text{nm}$): 271-302, 363, 374. Elemental analysis for $[\text{VOC}_{23}\text{H}_{19}\text{N}_3\text{O}_3]$: M.W. 452.36 g/mol: Calculated: C, 61.07; H, 4.23; N, 9.29%. Found: C, 60.56; H, 4.11; N, 9.13%.

2.3.5. Oxovanadium (IV) Complex of DL-methionine [VO(sal-met)(DPhen)]

Color: Brown, Melting point: 216 °C, Yield: 0.947 g (60%), Molar conductance ($\text{Ohm}^{-1}\text{cm}^2\text{mol}^{-1}$): 10.1, Magnetic moment ($\mu_{\text{eff}}/\text{B.M.}$): 1.75, FT-IR (cm^{-1}): 3425br, $\nu(\text{OH})$; 1639s, $\nu(\text{C}=\text{O})$; 1536s, $\nu(\text{C}=\text{N})$; 1311m, $\nu(\text{C}-\text{O})$; 958vs, $\nu(\text{V}=\text{O})$; 613m, $\nu(\text{V}-\text{O})$; 453m, $\nu(\text{V}-\text{N})$, UV-Vis. ($\lambda_{\text{max}}/\text{nm}$): 269-305, 364, 385. Elemental analysis for $[\text{VOC}_{26}\text{H}_{25}\text{N}_3\text{O}_3\text{S}]$: M.W. 526.50 g/mol: Calculated: C, 59.31; H, 4.79; N, 7.98%. Found: C, 59.14; H, 4.59; N, 7.78%.

Here, the related band intensities for FT-IR are denoted by *vs*, *s*, *m*, *w* and *br* representing *very strong*, *strong*, *medium*, *weak* and *broad band* respectively.

3. Results and Discussion

3.1. Solubility and Melting Point Measurement

All the complexes are soluble in dimethylformamide (DMF) and dimethylsulfoxide (DMSO) but insoluble in common organic solvents such as methanol, ethanol, chloroform, Carbon tetrachloride, benzene etc. Melting point (210-225 °C) gives an approximate idea about the nature of the complexes and can suggest whether it is covalent or ionic (Arun et al., 2009).

3.2. Elemental Analysis and Conductivity Measurement

The molar conductance of the complexes was measured in DMF (10^{-3} M solution) at 30 °C. The molar conductance values (10.1 to $18.2 \text{ W}^{-1}\text{cm}^2\text{mol}^{-1}$) are lower than that expected for an electrolyte and indicate that all the complexes are non-electrolytic in nature. Based on the elemental analysis, conductivity and magnetic susceptibility measurements, IR spectral and UV-Visible spectral analysis, the structure of the complexes has been proposed (Al-Resayes et al., 2012). Also, the analytical data are in good agreement with their proposed empirical formula.

3.3. Magnetic Moment and Electronic Spectra

The magnetic susceptibility of the complexes was measured by a Sherwood Scientific magnetic susceptibility balance at room temperature (30 °C) (Kriza et al., 2005). The observed values of effective magnetic moment (μ_{eff}) (1.57 to 1.75 BM) at room temperature indicated that all the complexes are paramagnetic in nature. The electronic spectra of the complexes were recorded in DMSO in the wavelength of 200-800 nm range. All the complexes exhibit a shoulder in the range 373-385 nm due to ligand-to-metal charge-transfer (LMCT, $\text{PhO}^- \rightarrow \text{V}$) transition, and the remaining bands appearing in the UV region are assignable to the intraligand transitions (Dutta et al., 1997). All the complexes exhibit bands in between 267-305 nm which are assignable to the $\pi \rightarrow \pi^*$ transition (Sasmal et al., 2007).

3.4 IR Spectral Studies

The IR spectral data of oxovanadium (IV) complexes show a broad band in the $3425\text{-}3454 \text{ cm}^{-1}$ region which is possibly

due to the hydrated water molecule in the complexes (Maurya et al., 2001). The complexes exhibit $\nu(\text{C}=\text{O})$ bands at 1620-1647 cm^{-1} and $\nu(\text{C}-\text{O})$ bands at 1310-1315 cm^{-1} which are significantly lower than the values for respective bands of uncoordinated amino acids. Again, the appearance of $\nu(\text{V}-\text{O})$ modes in the range 612-617 cm^{-1} confirms the coordination of carboxylate ion to the central metal ion (Dutta and Lal, 1988). The $\nu(\text{O}-\text{H})$ band usually appeared at $\sim 3600 \text{ cm}^{-1}$ for the phenolic $-\text{OH}$ is absent in the present complexes which supports the coordination of phenolic oxygen to vanadyl ion. The bands appeared in the range 1533-1542 cm^{-1} may be assigned to $\nu(\text{C}=\text{N})$ stretching frequency suggesting the coordination of the azomethine nitrogen and heterocyclic nitrogen to the VO^{2+} moiety. The coordination of nitrogen of azomethine group and heterocyclic nitrogen is further evident by the appearance of $\nu(\text{V}-\text{N})$ modes in the 446-461 cm^{-1} region (Singh et al., 1989). The present oxovanadium (IV) complexes exhibit the $\nu(\text{V}=\text{O})$ stretching frequency in the 941-962 cm^{-1} region characteristic of metal-oxygen multiple bond (Rao et al., 1988).

3.5 ESI-Mass Spectra

The ESI-Mass spectra of the complexes are presented in Figure 1. The obtained m/z values are similar to the formula weight of the respective complexes which further supports the proposed structure of the oxovanadium (IV) complexes.

On the basis of the elemental analysis, conductivity measurements, magnetic moment values, spectroscopic studies and literature review the structure of the complexes (Figure 2) could be illustrated as shown below:

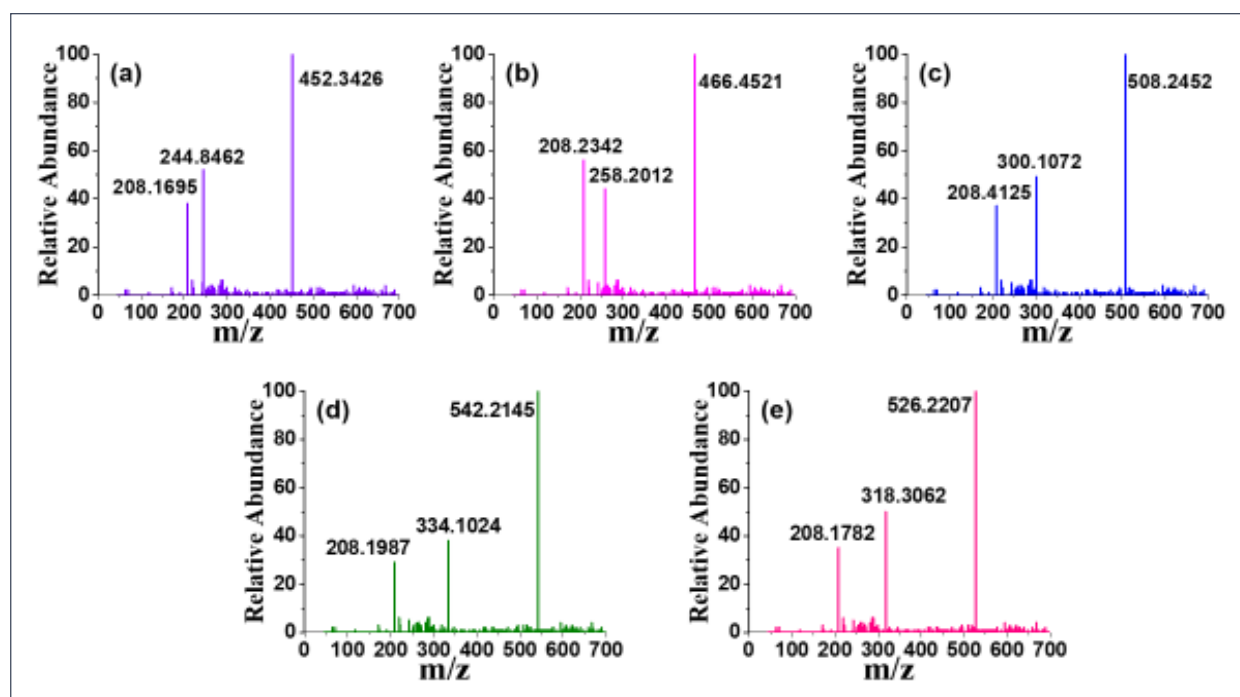


Figure 1: ESI-MS Spectrum of the Complexes; (a) [VO(sal-gly)(Dphen)], (b) [VO(sal-ala)(Dphen)], (c) [VO(sal-leu)(Dphen)], (d) [VO(sal-pheala)(Dphen)] and (e) [VO(sal-met)(Dphen)]

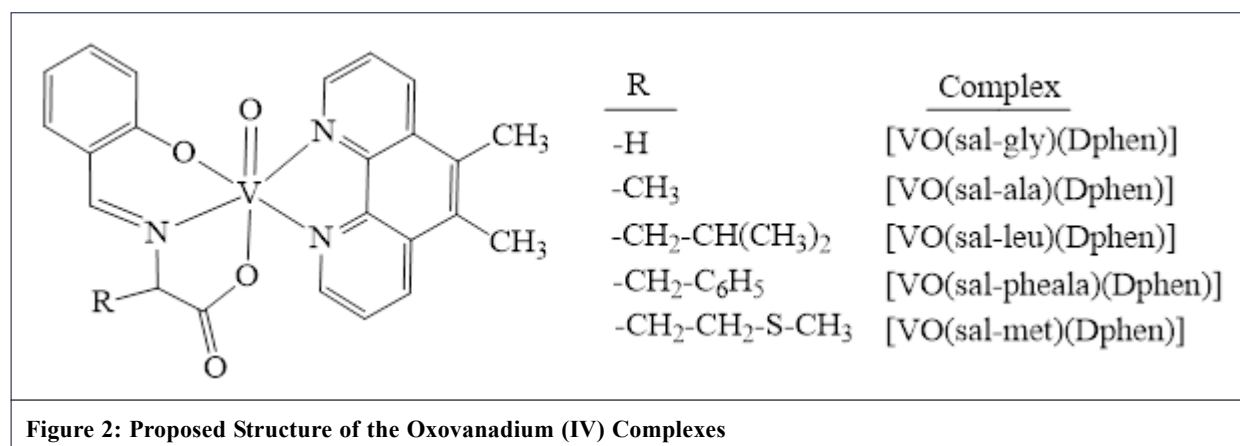
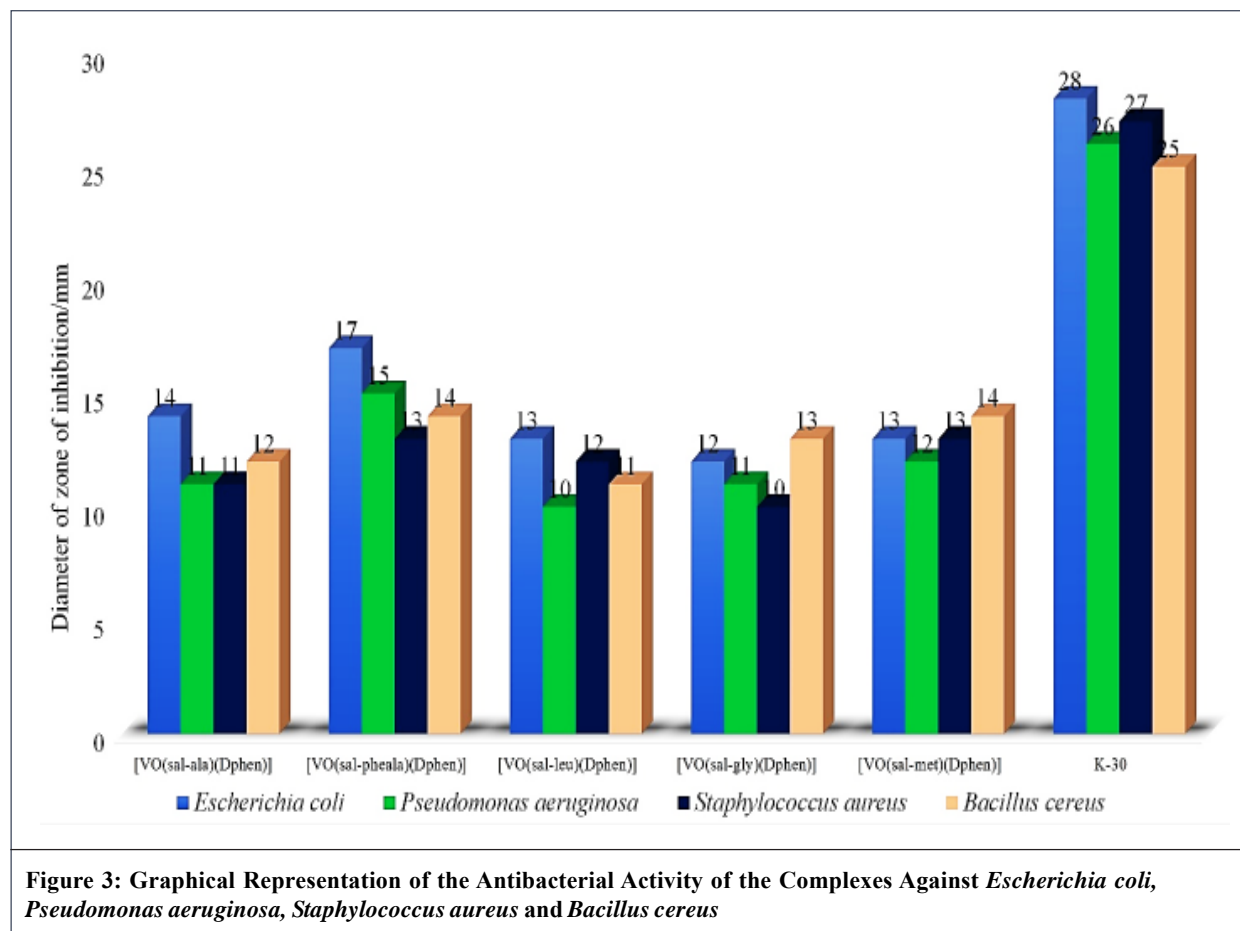


Figure 2: Proposed Structure of the Oxovanadium (IV) Complexes

4. Antibacterial Activity of the Complexes

Antibacterial activities of the synthesized complexes are investigated by using the traditional disc diffusion method (Elachi et al., 2019; and Bitu et al., 2020). The complexes (50 mg/disc in DMSO solution) were screened for their antibacterial activity against two Gram positive bacteria *Escherichia coli* and *Pseudomonas aeruginosa* and two Gram negative bacteria *Staphylococcus aureus* and *Bacillus cereus* with Kanamycin (K-30) standard (Haque et al., 2005; Ali et al., 2020a; 2020b; and 2020c). The result shows that all the complexes have moderate to strong potential antibacterial activity against all the tested pathogens. The result is graphically represented in Figure 3.



5. Conclusion

The VO²⁺ complexes of O, N, O-donor amino acid Schiff bases and 5,6-Dimethyl-1,10-phenanthroline have been synthesized and characterized. The molar conductance values shows that the complexes are non-electrolytic in nature. The magnetic moment values reveal that the complexes contain paramagnetic behavior and are in accordance with the d¹ electronic configuration of the V^{IV}O²⁺ moiety. IR spectral data indicates the coordination of tridentate amino acid Schiff base ligands to the vanadyl (VO²⁺) ion. UV-Visible spectra appeared in the range 373-385 nm is due to LMCT transition. Thus, on the basis of physicochemical properties, elemental analysis, conductivity measurements, magnetic moment values, spectroscopic studies and literature review, the structure of the complexes (Figure 2) may be proposed as distorted octahedral geometry with VO₃N₃ coordination environment. All the complexes were screened for their antibacterial activity against four human pathogenic bacteria; two Gram positive *Escherichia coli* and *Pseudomonas aeruginosa* and two Gram negative *Staphylococcus aureus* and *Bacillus cereus* with Kanamycin (K-30) standard. The result shows that all the complexes have moderate to strong potential antibacterial activity against all the tested organisms.

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