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Synthesis and Structural Study of Complex Ligands and Derivatives of Carbohydrate Oxazoline from Amino-Sugar

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ABSTRACT

A metal complexes were developed in this study, utilizing ligands derivatives of carbohydrate 1, 3-oxazolidine from amino-sugar modified (L_1 and L_2). The elemental analyses suggested that the stoichiometry is (1:2). The IR data confirmed the binding between the metal ion and the ligands. The crystallinity of the complexes formed was confirmed by the X-ray diffraction. The non-electrolyte nature of metal complexes was confirmed by molar conductance studies. The thermal study suggested the presence of coordinated water molecules in the complexes based on L_2 . The synthesized complexes and their corresponding ligands were tested for their antimicrobial activities against bacteria (*Escherichia coli*, *Pseudomonas aeruginosa*, *Staphylococcus aureus* and *Streptococcus pneumonia*). The complexes ($Zn(L_2)_2$ and $Ni(L_2)_2$) showed significant antibacterial activity compared to the corresponding ligands. The dosage with the radical 2,2-diphenyl-1-picrylhydrazyl at different concentrations for the complexes ($Ni(L_1)_2$ and $Ni(L_2)_2$) showed superior antioxidant activity than the corresponding ligands. The considerable results found proved that the ligands and their complexes are bioactive.

Keywords: Carbohydrate, 1,3-oxazoline, amino-sugar, Metal(II), Complexes, biologic activity.

1. Introduction

Azomethine free and/or bound had many reports of their applications in biology including antibacterial, antifungal, anti-cancer [1-4], obtained results are given as mean standard deviations of three determinations. In line with our interests, we tried to introduce transition metals to form 1,3-oxazolidin-tetra-*O*-acetyl-*D*-amino-sugar -based metal complexes to increase the selectivity, improve the stability, and the biological activity of the ligand (L_1 and L_2). The structures of ligands are presented in Figure 1. In this article, we describe synthesis, characterization, and behavior of metal complexes based on 1, 3-oxazolidine-tetra-*O*-acetyl-*D*-amino-sugar (L_1 and L_2). In addition, the biological activity of the four ligands and their complexes was examined.

2. Experimental

Synthesis of Ligand: The derivatives used in this work have already been synthesized and characterized at the Laboratory of Organic Chemistry 2-Glycochemistry (CO2-GLCO) of the University Claude Bernard-Lyon 1 France. The 1, 3-oxazolidine -tetra-*O*-acetyl-*D*-amino-sugar (L_1 et L_2) (**Figure.1**).

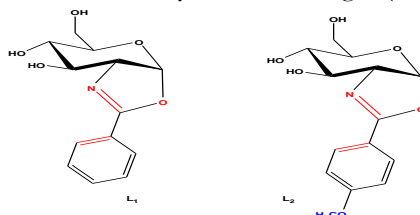


Figure1: structure of 1, 3-oxazolidin-tetra-*O*-acetyl-*D*-amino-sugar (L_1 and L_2)

Preparation of Metal (II) Complexes: A series of Metal(II)- 1, 3-oxazolidin-tetra-*O*-acetyl-*D*-amino-sugar (1:2) complexes were synthesized as described in the literature [5,6]. The complexes based on Zn(II), Ni(II), and Mn(II) and the Ligand (L_1 and L_2) were synthesized by the precipitation reaction of 2 mmol of



the dissolved ligand in 10 mL of absolute ethanol/distilled water (1:1) (v/v) with 1 mmol of metal acetate salt $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, and/or $\text{Mn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ dissolved in the same solvent. At ambient temperature, the reaction mixtures were maintained under magnetic stirring for 2–4 h to obtain a better yield. The mixtures were left to stand for 24 h. The precipitates obtained were filtered, washed with a water-ethanol mixture (1:1), and finally dried at 50°C.

3. Results and Discussion

All the synthesized metal complexes based on ligand (L_1 and L_2) were purified by recrystallization and by flash chromatography in the form of crystals and/or colored powders. In most cases, the yields varied from 88 % to 95%. The yields for the ligand-based complexes (Zn and Mn) are remarkable, while the yield values for the others are acceptable. It should be noted that melting points are lower than 355°C in all complexes, but we noted a decomposition in the complex $[\text{Zn}(\text{L}_1)_2 \cdot (\text{OAc})_2 \cdot 2\text{H}_2\text{O}]$. In contrast, they are all stable in air and do not need special storage precautions and can be stored for a long time. The metal complexes obtained generally do not have characteristic odors. They are poorly soluble in petroleum ether and toluene; they are slightly soluble in acetone and in hexane at room temperature. They even precipitate in the hot medium. On the other hand, all the complexes are freely soluble in DMSO, DMF, methanol, and ethanol, while the solubility of the ligand-based complex (L) is higher compared to the others. We also noticed that the color of the complexes is not the same as the corresponding ligands, which confirms the coordination of the ligand with the metal. This color change may be due to the metal-ligand interaction, which proves that the electronic properties of the complexes are distinct from the ligands used.

4. Conclusions

This research focused on the derivatives of 1, 3-oxazolidin-tetra-*O*-acetyl-*D*-amino-sugar, which were taken as free ligand (L_1 and L_2). The metal (II) complexes of Ni, Mn, and Zn were produced using the coordination reaction with metal salts, giving naissance of new symmetrical metal complexes with the molar ratio (1:2) $[\text{M}(\text{II}):(\text{L})_2]$. The obtained complexes are stable solids and with a different color. The obtained metal complex structures were confirmed using elemental analysis, FT-IR, UV-Vis, and TGA. Molar conductivity measurements indicate that all complexes are non-electrolyte in DMF. Structural study by FT-IR eventually revealed the monodentate coordination of tested ligands and further showed the lowest frequency shift after coordination of the metal ions to the ligand; X-ray diffraction analysis suggests a crystal system in all L -based metal complexes. Antimicrobial tests showed that the $[\text{Zn}(\text{L}_2)_2 \cdot (\text{OAc})_2 \cdot 2\text{H}_2\text{O}]$ and $[\text{Ni}(\text{L}_2)_2 \cdot (\text{OAc})_2 \cdot 2\text{H}_2\text{O}]$ complexes recorded antibacterial efficiencies. Significantly, a study of the free radical scavenging properties of the compounds revealed that the $[\text{Ni}(\text{L}_2)_2 \cdot (\text{OAc})_2 \cdot 2\text{H}_2\text{O}]$ and $[\text{Ni}(\text{L}_2)_2 \cdot (\text{OAc})_2 \cdot 2\text{H}_2\text{O}]$ complexes possessed considerable antioxidant activities.

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