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Ni(II) N-(2-Pyridylmethyl)-L-Alanine) Complex: Structural Diversity and Photoluminiscent Studies

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Abstract

A novel chiral Nickel (II) complex of N-(2-pyridylmethyl)-L-alanine (Hpyala) **1** has been prepared and structurally characterized by elemental analysis, FT-IR, UV-visible, TGA and single crystal X-ray diffraction techniques. Complex **1** crystallizes in an orthorhombic $P2_12_12_1$ space group. The nickel (II) centre in the complex adopts a distorted octahedral geometry. This compound has been seen to exhibit structural diversity resulting from the number of lattice water molecules. The photoluminescent properties of this compound which have also been investigated, indicates the potential application in luminescence.

Keywords

Nickel (II) Complex, Chiral, Photoluminescent, X-Ray Diffraction

1. Introduction

The desire to rationally design and synthesize useful solid materials through the controlled assembly of molecular components, has led to a recent surge of interest in the concept of crystal engineering [1] [2] [3] [4]. So far, considerable progress has been achieved in tuning and predicting the mode of assembly and orientation of individual building blocks into structures with specific topologies and properties [5] [6] [7], but accurate prediction and control of the 3-D structure of molecular crystals and coordination polymers remains difficult. Reduced Schiff base ligands tend to form a suitable conformational and coordination environment. They have been found to form more flexible and multidentate network complexes because of the reduction of the C=N bond of the Schiff base which helps to overcome ligand stability [8] [9] [10]. In recent years, significant research interests have been devoted to coordination polymers with chiral helical structural motifs [6]. The preparation of chiral coordination complexes is a very well-established procedure and it dates back to Werner, who showed that the complex chirality depends on the formation of *cis*-coordination geometry around the metal ion with appropriate chelating/monodentate ligands. This can be understood based on the symmetry criteria that the absence of improper axis of rotation results in chirality [7]. Metal complexes of N-(2-pyridylmethyl)-amino acid derivatives with different spacers have been reported to have multiple attractive architectures including multidimensional and oligomeric structures with cavities, channels and chiral capsules owing to their robust hydrogen-bonding ability and inherent chirality [11]-[20]. In the pioneering work in these fields, N-(2-pyridylmethyl)-alanine has been used to build the copper, cobalt and zinc complexes [20]. There have been few reports on new nickel (II) with *N*-(2-pyridylmethyl)-alanine in literature [10]. Furthermore, nickel (II) Schiff-base complexes have been reported to have promising applications for their luminescence [21] [22]. Herein, we report on a novel nickel (II) complex with pyridyl reduced Schiff-base ligand of amino acid derivatives $[Ni(II)(L\text{-}pyala)_2]\cdot 4H_2O$, L-Hpyala = *N*-(2-pyridylmethyl)-L-alanine, its synthesis, structural and photoluminescence properties. This article brings out some structural diversity as compared to a similarly reported nickel (II) complex in relation to the role of lattice water due to modified experimental conditions such as change in pH in directing the formation of supramolecular structures in the solid state [10]. In addition, its photoluminescence properties have been evaluated.

2. Experimental

2.1. Materials

All chemicals and solvents used for the synthesis were of reagent grade. Nickel acetate tetrahydrate (Aldrich), 2-pyridine aldehyde (Prolabo) and L-alanine (Prolabo) were used as received. The solvents, ethanol was dried and distilled according to standard methods.

2.2. Analytical Methods

Elemental analysis for carbon, nitrogen and hydrogen were carried out on a Fisons instrument 1108 CHNS-O. Infrared spectra were recorded on a Perkin-Elmer model IR-457 spectrometer and a spectrum 100 FT-IR Perkin Perkin-Elmer spectrometer, while X-ray diffraction was carried out with a CCD bi-dimensional diffractometer using monochromatic radiation, $\lambda\ (M_o - K_\alpha) = 0.71073$ Å, operating at 50 kV and 40 mA. TGA was recorded using a Mettler Toledo TGA/DSC1 SATR system while UV-visible spectrophotometer, HACH DR 3900, MARK Brucker.

2.3. Synthesis

2.3.1. Synthesis of Ligand

The synthesis of the ligand was as described in literature [17].

2.3.2. Synthesis of $[Ni(C_9H_{11}N_2O_2)_2]\cdot 4H_2O$ (1)

Pyala (0.36 g, 2 mmol) in 10 mL water/ethanol mixture (1:1) was added drop wise to a 5 ml aqueous solution of $Ni(OOCH_3)_2\cdot 4H_2O$ (0.245 g, 1 mmol), while stirring magnetically at room temperature. Stirring continued for two hours. Suitable purple crystals for X-ray analysis were obtained from the solution by slow evaporation after two weeks. Yield 70%; Anal. Calcd. For $C_{18}H_{30}NiN_4O_8$; C, 41.52; H, 4.12; N, 22.01; Found: C, 41.46; H, 4.07; N, 22.59.

2.4. X-Ray Crystal Structure Determination

The crystal structure of the complex was determined by single crystal X-ray diffraction on a Brucker *APEX*-II using monochromatic Mo*K*α radiation (λ = 0.71073 Å) at a temperature of 198 K and integrated with SAINT-Plus program [23], and absorption corrections were carried out by multi-scan method by SADABS [24]. The Structure was solved by direct methods and refined against F^2 by full-matrix least-squares techniques with SHELTL [25]. All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydrogen atoms were included from calculated positions and refined riding their respective parent atoms with isotropic displacement parameters.

3. Results and Discussion

3.1. Infra-Red Spectra

The IR spectra of the ligand showed a characteristic weak absorption band at 2982 cm^{-1} due to ν (N-H) stretching of amines [26]. This band confirms the formation of imine (-C=N) group of Schiff bases which was reduced during the synthesis by $NaBH_4/H_2O$ to the amine group -N-H. Furthermore the IR spectra of the ligand revealed a strong absorption band at 1672 cm^{-1} due to ν (C=C, C=N) stretching of the pyridyl group [27]. This band in the metal complex shifted to 1577 cm^{-1} and 1597 cm^{-1}. The decrease in wave number as a result of reduction in electron density around the ν (C=C, C=N) bond of the ring, suggests the par ticipation of the pyridyl ν (C=N) bond in the complexation through the nitrogen atom [28]. In addition the Ni(II) complex exhibited a very weak absorption band around 3288 cm^{-1} which suggests the deprotonation of the ν (OH) bond and the participation of the oxygen atom in complexation [28].

3.2. Crystal Structure of $[Ni\ (C_9H_{11}N_2O_2)_2]\cdot 4H_2O$(1)

The structure of **1** with atomic numbering scheme is shown in **Figure 1**. The Crystal data and structure refinement details for the title compound are summarized in **Table 1** while selected bond lengths and angles are listed in **Table 2**. The X-ray single crystal diffraction analysis reveals that complex 1 crystallizes in the

Table 1. Crystal data and structure refinement for 1.

Compound	**1**
Empirical formula	C_{18} H_{30} N_4NiO_8
Formula weight	489.17
T (K)	198 (2)
Space group	$P2_12_12_1$
Wavelength (Å)	0.71073
Crystal system	Orthorhombic
Unit cell dimensions	
a (Å)	9.7812 (4)
b (Å)	10.7564 (4)
c (Å)	21.3721 (8)
α (˚)	90
β (˚)	90
γ (˚)	90
Volume ($Å^3$)	2248.57 (15)
Z	4
D_X ($g·cm^{-3}$)	1.445
μ (mm^{-1})	0.91
F (000)	1032
Crystal size (mm^3)	0.24 × 0.22 × 0.14
θ range (˚)	2.7 - 29.8
Miller Index range	V12 ≤ h ≤ 13, −14 ≤ k ≤ 15, −30 ≤ l ≤ 29
Reflections collected	179,527
Independent reflections (R_{int})	6570 [R(int) = 0.030]
Completeness to θ_{max} (%)	99.9
Max. and min. transmission	0.811 and 0.883
Data/restraints/parameter	6570/7/322
Goodness-of-fit (GOF) on F2	1.111
Final R indices [I > 2σ(I)]	R1 = 0.019, wR2 = 0.048
R indices (all data)	

Table 2. Selected bond lengths (Å) and Bond angles (˚) of 1.

Bond lengths		Bond angles	
Ni1-O1	2.0232 (3)	O1-Ni1-O3	90.916 (13)
Ni1-O3	2.0381 (3)	O1-Ni1-N12	90.916 (13)
Ni1-N12	2.0947 (4)	O3-Ni1-N12	93.328 (14)
Ni1-N1	2.0950 (4)	O1-Ni1-N1	92.749 (14)

Continued

Ni1-N19	2.1030 (3)	O3-Ni1-N1	95.509 (13)
Ni1-N8	2.1177 (3)	N12-Ni1-N1	171.737 (15)
N12-C17	1.3398 (6)	O1-Ni1-N19	170.037 (13)
N12-C13	1.3444 (6)	O3-Ni1-N19	82.608 (18)
N1-C2	1.3405 (6)	N12-Ni1-N19	79.887 (14)
N1-C6	1.3447 (5)	N1-Ni1-N19	95.083 (13)
N19-C20	1.4860 (5)	O1-Ni1-N8	82.324 (13)
N8-C9	1.4882 (5)	O3-Ni1-N8	169.686 (13)
		N12-Ni1-N8	94.792 (14)
		N1-Ni1-N8	80.106 (14)
		N19-Ni1-N8	105.075 (13)

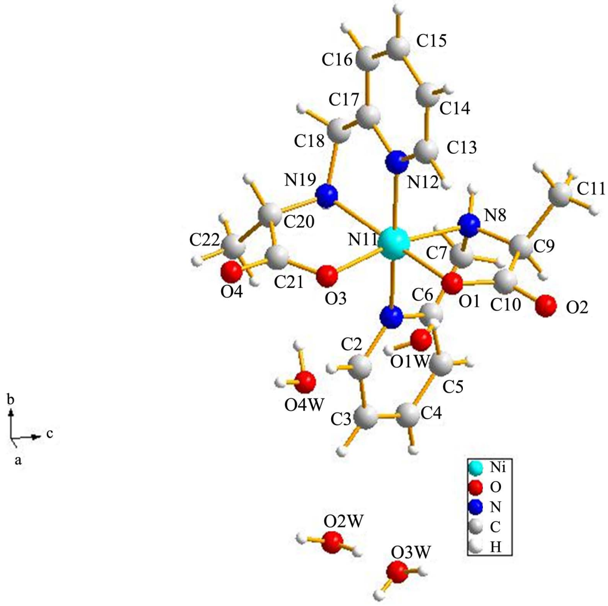

Figure 1. Coordination environment of Ni(II) center with atom labeling in 1.

orthorhombic system $P2_12_12_1$ space group. The Ni(II) centre lying on the C2 axis (x, 1, 1), has a distorted octahedral geometry, which is surrounded by two oxygen and four nitrogen donors from two monodentate carboxylate Ni1-O1, 2.0232 (3) Å and Ni1-O3, 2.0381 (3) Å, two amino nitrogen atoms Ni1-N1, 2.0947 (4) Å, Ni1-N1, 2.0950 (4) Å and two pyridyl nitrogens Ni1-N19, 2.1030 (3) Å; Ni1-N8, 2.1177 (3) Å. The O1-Ni1-O3, O1-Ni1-N12, O3-Ni1-N12, O1-Ni1-N1 and O3-Ni1-N1 bond angles are 90.916 (13)˚, 90.916 (13)˚, 93.328 (14)˚, 92.749 (14)˚ and 95.509 (13)˚ respectively. These values are comparable to previously reported results of a similar nickel (II) complex with the same ligand having Ni-O bond distances in the range of 2.0496 (12) - 2.035 (2) Å and Ni-N bond distances in the range 2.0849 (13) - 2.110 (2) Å [10]. Furthermore the crystallographic studies revealed a different packing pattern with chiral channels runing parallel to the c-axis relative to that of a similar compund previously reported. This packing in **1** differs notably as evident in **Figure 2** which could be attributed to the presence of four moelcules of water of crystallisation in the compound that facilitate hydrogen bonding formation.

3.3. Thermal Stability

The thermogravimetric analysis (TGA) of the complex was recorded with the TG curve shown in **Figure 3**. The first step decomposition at 25˚C [7.88% weight loss (calculated, 11.92%)] is attributed to the loss of three molecules of lattice water and the second decomposition at 110˚C [2.50% weight loss (calculated, 3.60%)] which suggests the final loss of one molecule of lattice water, making a total loss of four water molecules of crystallization associated to the nickel (II) complex. The third decomposition at 270˚C corresponding to 29.23% weight loss (calculated, 36.38%) and the fourth decomposition at 330˚C corresponding to 27.50% weight loss (calculated, 29.05%), suggests the decomposition of the ligands. The final residue of 32.89% (calculated, 30.58) is attributed to the mixture of nickel oxides.

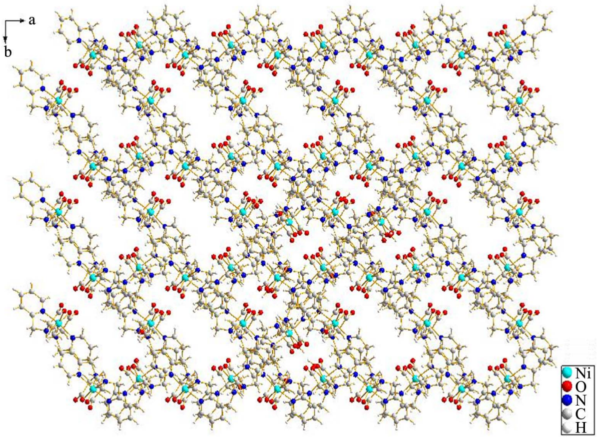

Figure 2. Packing of 1 along c-axis, showing a zigzag Ni(II) array of chiral channels.

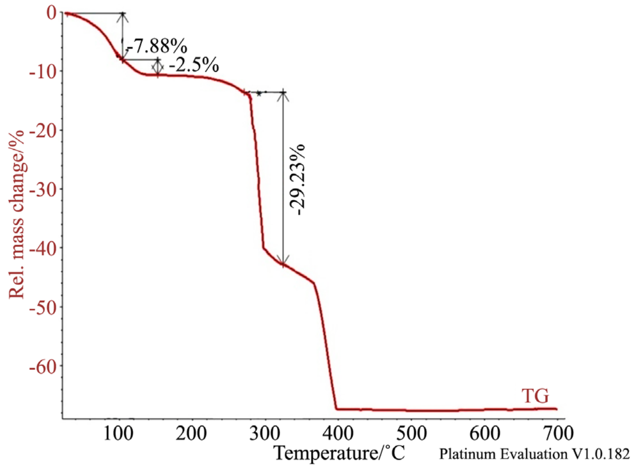

Figure 3. TGA thermogram for 1.

3.4. UV-Visible Spectra

The UV-visible spectra of **1** shows a small energy transition band at 320 - 331 nm and a weak broad absorption band in the range of 488 - 614 nm as shown in **Figure 4**. The small energy transition band at 320 - 331 nm in the nickel (II) spectra is attributed to the metal to ligand charge transfer (MLCT) transition in the nickel (II) complex and the weak broad band observed in the range of 488 - 614 nm is presumably due to the spin allowed d-d transition ($^3A_{2g} \rightarrow {}^3T_{2g}$ (F) which is characteristic of an octahedral geometry around the nickel (II) atom [29].

3.5. Photoluminescent Behavior of Compound

The photoluminescent (PL) property of the complex (1) was investigated in the solid state at room temperature. The PL curve shown in **Figure 5** showed strong fluorescent emission at 442 nm. This was obtained when the complex was excited at 325 nm. In order to understand the nature of the emission, we examined the PL property of the free ligand found that the strongest emission peak is at 391 and 432 nm. Therefore the luminescence of the complexes May be attributed to metal to ligand charge transfer (MLCT) or ligand to metal charge transfer (LMCT) [10] [30] [31]. These observations suggest that the compound may be good a candidates for potential photoactive and in the preparation of optical.

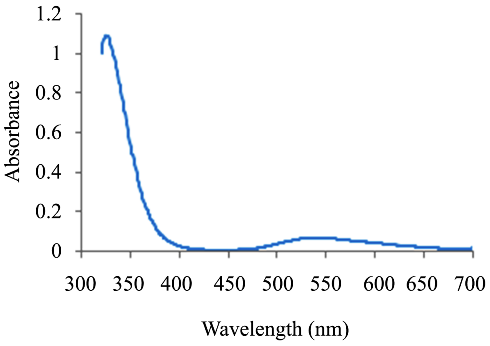

Figure 4. UV-visible spectra of Ni(II) complex.

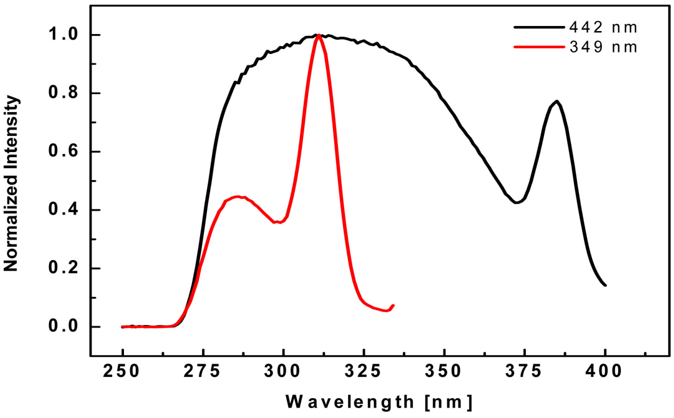

Figure 5. Solid state emission spectra of complex 1.

4. Conclusion

A novel Ni(II) coordination polymer of N-(2-pyridylmethyl)-(L)-alanine (Hpyala) has been obtained and the structure compared with that of a similar compound previously reported. The structural diversity of the compound compared with that previously reported is as a result of a difference in crystal water due to the modification of the experimental conditions such as pH in directing the formation of supramolecular structures in the solid state. Compound (1) crystallizes in the orthorhombic system with chiral space group, $P2_12_12_1$. The diversity in the structure is seen in its packing along various axes.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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