Research Article

Synthesis, Characterization, and Antimicrobial Activity Studies of Ni(II) Complex with Pyridine as a Ligand

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Received 4 January 2015; Accepted 20 February 2015

Academic Editor: Jae Ryang Hahn

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We represent a metal complex which has been synthesized by the simple reaction with Ni(II) chloride and pyridine (as a ligand) affording a complex having the molecular formula $[\text{Ni}(\text{C}_5\text{H}_5\text{N})_2\text{Cl}_2]$, characterized on the basis of elemental analyses, electronic, infrared, ^1H NMR, ^{13}C NMR spectra, magnetic susceptibility, and also aid of molar conductivity measurement. Conductivity measurement reveals nonelectrolytic nature of the complex. IR and ^{13}C NMR spectra reveal the presence of *cis*- and *trans*-structure. On the basis of above analyses the square planar *cis*- and *trans*-structures are proposed for the prepared complex.

1. Introduction

Coordination complexes have persist an important and popular area of research due to their simple synthesis, adaptability, and different range of applications. From the existing literature, it seems that transition metal complexes played a vital role in the development of coordination chemistry [1, 2]. It also plays an important role in biological process as exemplified in many instances in which enzymes are known to be activated by metal ions [3]. These complexes have been occupied in the strongest and transport of active substances through membrane [4]. Many metal complexes are resulting application in the microelectronic industry, chemical vapour deposition of metals, and drugs [5]. Coordination compounds display diverse characteristic properties which depend on the metal ion to which they are bound. On the basis of nature of the metal as well as the type of ligand, these metal complexes have wide applications in different fields of human curiosity [6, 7].

Pyridine is a heterocyclic organic compound with the chemical formula $\text{C}_5\text{H}_5\text{N}$ and is structurally related to benzene, with one CH group replaced by a nitrogen atom [8]. It is also a six-membered cyclic aromatic molecule with σ and π binding capabilities through its nitrogen electron lone pair and π system, respectively, which has inspired considerable interests in its bonding with metals over the last decade. A number of experimental and theoretical investigations have established that most metal-pyridine complexes are σ complexes [9–12]. It has importance in industrial organic chemistry, both as a fundamental building block and as a solvent and reagent in organic synthesis [13]. Pyridine derivatives also play significant role in many biological systems as the component of several vitamins, nucleic acids, enzymes, and proteins [14]. Pyridine derivatives have occupied a unique position in the field of medicinal chemistry. Some of them constitute an important class of antitumor compounds [15]. 2-Amino-3-cyanopyridines have been identified to possess antibacterial, [16] antimicrobial,

TABLE 1: Analytical and physical data for the complex.

Complex	Color	M. P. (°C)	Calculated data % (found)					ΔM Ohm ⁻¹ cm ² mol ⁻¹	μ_{eff} B. M.
			C	H	N	Cl	Ni		
[Ni(C ₅ H ₅ N) ₂ Cl ₂]	Yellow		41.21	3.46	9.62	25.56	20.15	0.00 (in CHCl ₃)	
<i>Cis</i> -[Ni(C ₅ H ₅ N) ₂ Cl ₂]	Reddish yellow	319	(40.98)	(3.41)	(9.57)	(25.16)	(19.95)	315 (in H ₂ O)	0.00
<i>Trans</i> -[Ni(C ₅ H ₅ N) ₂ Cl ₂]	Deep yellow	360							

[17, 18] antifungal, [19] cardiogenic, [20] analgesic [21], anti-inflammatory [22], and antitumor [23] activities.

Nickel is an important transition metal normally stable in aqueous solution in the +2 oxidation state [24]. It is important in biological system, which is a key factor affecting the production of secondary plant metabolites, thus influencing plant resistance to disease [25]. It interacts with iron found in the hemoglobin and helps in oxygen transport, stimulates the metabolism, and is regarded as a key metal in several plants and animal enzyme systems [26]. Nickel is involved in the transmission of genetic code (DNA, RNA) and it is also present in certain enzyme systems that metabolize sugars [27]. This metal is more attracted by the researchers in recent years. Many nickel complexes have been reported with their importance [28–31]. Nickel sulfate hexahydrate is used in nickel electroplating, Nickelocene is used as a catalyst and complexing agent, and nickel titanate is used as a pigment [32].

As a part of continuing interest in this area of research, we have investigated coordination behavior of square planar Ni(II) complex of pyridine. The complex has been characterized by spectral technique and thin-layer chromatography method. The new nickel(II) complex has also been screened for antibacterial and antifungal activities against various pathogenic bacteria and fungi.

2. Experiment

2.1. Materials and Methods. All the chemicals used were of analytical reagent grade. These were obtained from M/S E. Merck and used without further purification. The analysis of C, H, and N was performed on a vario MICRO Vi. 6.1 GmbH, Japan CHNS analyzer. Molar conductance of the complex was measured in water and chloroform at room temperature using a HANNA instrument with HI 8820N conductivity cell. Magnetic susceptibility of the complex was performed on a Sherwood Scientific magnetic susceptibility balance. IR spectra were recorded as KBr disc in the range of 400–4000 cm⁻¹ on a Perkin-Elmer 883 and Shimadzu infrared spectrophotometer. Electronic spectra were run between 250 and 500 nm on a Shimadzu UV-visible spectrophotometer (model UV-1800) using 1 cm cell. ¹H-NMR and ¹³C-NMR spectra were recorded on a Bruker 400 MHz instrument using TMS as an internal reference. The NMR spectrum of the complexes under study was recorded from the Department of Chemistry and Biotechnology, Osaka University, Tottori, Japan. Melting points were measured with an electrothermal melting point apparatus. The Ni content of the prepared

complex was done by complexometric titration method [33]. The purity of the complex was checked by TLC using silica gel G plates.

2.2. Synthesis of [Ni(C₅H₅N)₂Cl₂] Complex. 23.77 g (0.1 moles) NiCl₂ was dissolved in 100 mL distilled ethanol with continuous stirring. The solution was greenish-blue in color and in this solution 8.05 mL (0.1 moles) pyridine was added dropwise with continuous stirring. The color of the mixture became deep blue instantly. The reaction mixture was stirred on a magnetic stirrer for further 2–3 hours. Then the mixture refluxed for about 10 hours in a water bath. The reaction mixture was then allowed to stand 30 hours. During this period a light yellow coloured precipitation started to form. To ensure the complete formation of the metal complex it was preserved for another 24 hours. Then the light yellow coloured nickel-pyridine complex was separated by vacuum filtration. After separation the complex was washed by redistilled ethanol until the colourless filtrate is coming out. After washing, the complex was dried in a vacuum desiccator over silica gel.

3. Result and Discussion

The complex obtained is yellow colored microcrystalline powder and air stable, but soluble in water and chloroform. The results of elemental analysis (C, H, N, Ni, and Cl) along with molecular formulae and melting points of the complex are presented in Table 1.

3.1. Infrared Spectral Data. The infrared spectrum of [Ni(C₅H₅N)₂Cl₂] complex is shown in Figures 1 and 2 and the absorption bands are listed with the relative intensities along with tentative assignments of the various bands in Table 2. In the spectrum of the complex, absorption bands at 3020–3080 cm⁻¹ and 1145–1220 cm⁻¹ are due to the stretching vibration of C–H and C–C, respectively. According to Ni–N bonding [34], IR spectrum is found with 530–600 cm⁻¹. In our prepared complex a nice IR band is found at 542 cm⁻¹, 549.71 cm⁻¹, and 557.43 cm⁻¹ which are due to the stretching vibration of Ni–N bond, which indicates that the formation of nickel-pyridine complex is a mixture and confirms it by TLC and separated into *cis*- and *trans*-product. The strong absorption bands at 1400–1500 cm⁻¹ and medium intensity absorption peaks at 1550–1598 cm⁻¹ are due to the stretching vibration of C=C bond and C–N bond [34, 35], respectively. The strong intensity absorption peaks at 1600–1650 cm⁻¹ are due to the stretching vibration of C=N. We have found that

TABLE 2: Infrared and electronic spectral data for the complex.

Complex	Infrared spectral data (cm ⁻¹)						Electronic spectral bands (nm)	
	$\nu_{\text{C-H}}$	$\nu_{\text{C-C}}$	$\nu_{\text{C=C}}$	$\nu_{\text{C-N}}$	$\nu_{\text{C=N}}$	$\nu_{\text{Ni-N}}$	$\nu_{\text{Ni-Cl}}$	
[Ni(C ₅ H ₅ N) ₂ Cl ₂]	3028.24, 3070.68	1155.36	1448.54, 1489.05	1573.91	1606.70	542.00, 549.71, 557.43	688.59, 700.16	395, 400, 300
<i>Cis</i> -[Ni(C ₅ H ₅ N) ₂ Cl ₂]	—	1149.57	1485.19	1598.99	1600.92	511.14	692.44 694.37	—

FIGURE 1: Infrared spectrum of [Ni(C₅H₅N)₂Cl₂].

FIGURE 2: Infrared spectrum of [Ni(C₅H₅N)₂Cl₂].

the strong intensity absorption peaks at 680–700 cm⁻¹ are due to the stretching vibration of Ni–Cl bond. According to [36, 37] Ni–N bonding, strong IR spectrum is found at 511.14 cm⁻¹ and 588.29 cm⁻¹ which indicates the formation of *cis*- and *trans*-configuration shown in Figures 3 and 4 due to the stretching vibration of Ni–N bond. From IR spectrum

position the *cis*- and *trans*-configuration of square planar [Ni(C₅H₅N)₂Cl₂] complex are confirmed.

3.2. Electronic and NMR Studies. The electronic spectrum of the complex displays Table 2 absorption band at 300 nm

FIGURE 3: Infrared spectrum of *cis*-[Ni(C₅H₅N)₂Cl₂]. FIGURE 4: Infrared spectrum of *trans*-[Ni(C₅H₅N)₂Cl₂]. FIGURE 5: ¹H-NMR spectral data for the complex [Ni(C₅H₅N)₂Cl₂].

FIGURE 6: ^{13}C -NMR spectrum of *cis*- $[\text{Ni}(\text{C}_5\text{H}_5\text{N})_2\text{Cl}_2]$. FIGURE 7: ^{13}C -NMR spectrum of *trans*- $[\text{Ni}(\text{C}_5\text{H}_5\text{N})_2\text{Cl}_2]$.

in the ultraviolet region which indicates that nickel-ligand charge transfer transition and absorption bands at 390–405 nm in water show the square planar geometry.

The ^1H -NMR spectrum of Ni(II) complex shows in Figure 5 three triplet signals at 7.6625–7.6693 ppm being for the proton of C_3 carbon. The second 7.6773–7.6854 ppm and third 7.6934–7.7002 ppm show for the protons of C_2 , C_4 and C_1 , C_5 carbons which are pairwise equivalent. But ^{13}C -NMR spectra give a separate resonance for each stereochemically distinct carbon atom. The ^{13}C -NMR spectra give additional evidences on the assignment of structures on the basis of ^1H -NMR spectrum analysis.

The spectrum in Figure 6 exhibits four peaks at 102.698 ppm, 139.282 ppm, 163.472 ppm, and 208.894 ppm which correspond to four nonequivalent carbon atoms. Among the four peaks, the second one at 139.282 ppm is for the C_2 and C_4 carbon being pairwise equivalent. The first peak shows that the C_3 carbon atom is comparatively more shielded, but the last two indicate that C_1 and C_5 carbons are highly deshielded compared to those of other ring carbons. According to the spectral position, the *cis*-conformation is fixed. Figure 7 shows three signals; 124 ppm indicates that the C_3 atom is comparatively more shielded than other ring carbons. The second (136 ppm) and third (150 ppm) signal exhibit the C_2 , C_4 carbons and C_1 , C_5 carbons, respectively, which are pairwise equivalent. However the last peak also indicates that C_1 and C_5 carbons are highly deshielded

compared to those of other ring carbons. According to all NMR spectral positions the *cis*- and *trans*-conformation are confirmed.

3.3. Magnetic and Conductance Studies. The molar conductivity value of $0.00 \text{ ohm}^{-1} \text{ cm}^2 \text{ mole}^{-1}$ (Table 1) in chloroform strongly supports the nonelectrolytic nature of the complex; that is, all the anions are in the coordination sphere as expected for four coordinated square planar Ni(II) complexes. On the other hand, the molar conductivity value of $315 \text{ ohm}^{-1} \text{ cm}^2 \text{ mole}^{-1}$ of the yellow coloured aqueous solution of this complex corresponding to 1:2 electrolytes provides evidence that di-aqua complex is formed by the complete replacement of Cl^- ion by H_2O molecules in aqueous solution. The magnetic moment value of the complex is 0.00 B.M. which corresponds to no unpaired electron as expected for square planar nickel (II) complex.

3.4. Antimicrobial Studies. Transition metal coordination complexes play vital role in biological study; some of these have now been widely studied for their antimicrobial and anticancer properties [38] and extensive investigations in the field of metal complexes have been reported [39, 40]. A novel Ni(II) complex has also been studied with its antimicrobial activities [41]. In the continuation of this discovery present studies synthesized a new Ni(II) pyridine complex and

FIGURE 8: Inhibition of fungi mycelial growth by the tested ligand and complex.

FIGURE 9: Bacterial growth inhibition by the tested ligand and complex.

the antifungal and antibacterial effect were observed. So it appeared interesting to see whether the compounds involved in this study exhibit any such activity or not.

In the present investigation both ligand and complex have been evaluated against human pathogenic bacteria such as *Salmonella typhi* (B1), *Shigella dysenteriae* (B2), *Escherichia coli* (B3), *Bacillus cereus* (B4), and phytopathogenic fungi such as *Macrophomina phaseolina* (F1), *Alternaria alternata* (F2), *Fusarium equiseti* (F3), *Colletotrichum corcolei* (F4), and *Botryodiplodia theobromae* (F5).

The results of the inhibition zones of the selected bacteria due to the effect of ligand and complex are graphically presented in Figure 8. The present work reveals that the complex was more effective against bacteria than its ligand and the data show that *E. coli* was inhibited to the greatest degree by the prepared complex. The results of the percentage inhibition of mycelia growth of the plant pathogenic fungi due to the effect of study are given graphically in Figure 9. The overall results indicated that the present complex has various

amounts of effects on the inhibition of mycelial growth. Further (in most of cases) the ligand and complexes are found to show higher inhibition on the growth of *Macrophomina phaseolina* in comparison with other fungi.

From the above discussion, it can be concluded that nature of ligands and metals plays a significant role in the inhibition of mycelial growth. However, in order to understand the functions responsible for antifungal activities of pyridine and its metal complexes, more studies are needed to be carried out with a series of analogous ligands and their complexes against a series of phytopathogenic fungi and bacteria.

4. Conclusion

From analytical and physical some spectral data at four coordinated *cis*- and *trans*-structures of the complex were proposed. Measurements of inhibition zones of ligand and complex show that the prepared complex has enhanced antibacterial activity more than ligand. In conclusion, this complex could reasonably be used for the treatment of some common diseases caused by *E. coli*.

Conflict of Interests

The authors declare that they have no conflict of interests.

Acknowledgments

The authors thank the Authority of Bangladesh Council of Scientific and Industrial Research (BCSIR) for providing all support, heartfelt gratitude is due to Osaka University, Osaka, Japan, for recording 1H -NMR and ^{13}C -NMR spectra of the complexes, and also special thanks are due to Chittagong College for assisting them by the support.

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