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PHYSICO-CHEMICAL CHARACTERIZATION AND BIOLOGICAL SCREENING OF METAL COMPLEXES WITH CYANEX 301

TARUN KUMAR PAL*, MD. ASHRAFUL ALAM[1] AND SUCHITRA RANI PAUL[2]

Department of Chemistry, Rajshahi University of Engineering & Technology, Rajshahi, Bangladesh

ABSTRACT

New metal complexes of Mg(II), VO(II), Mn(II), Fe(III), Co(II), Ni(II), Cu(II) and Pd(II) with cyanex 301(L) i.e., bis(2,4,4-trimethylpentyl) dithiophosphinic acid were synthesized. The complexes have been characterized by elemental analysis, molar conductivity, molecular mass determination, magnetic measurements, infrared and electronic spectral studies. The prepared metal complexes have the compositions: 2. $[MgL_2].H_2O$, 3. $[VOL_2]$, 4. $K[MnL_3].H_2O$, 5. $[FeL_3]$, 6. $K[CoL_3].H_2O$, 7. $K[NiL_3]$, 8. $K[CuL_3]$ and 9. $[PdL_2]$. The complexes 2, 3 and 9 are assumed to have tetrahedral, square pyramidal and square planar geometries, respectively but the complexes 4 - 8 are octahedral based on experimental data. From magnetic measurements the complexes 2 and 9 are found to be diamagnetic and others are paramagnetic. Measured molar conductance showed that the complexes 2, 3, 5 and 9 are non-electrolytes and rest are electrolytes. Besides, some complexes have shown good antibacterial and antifungal activities.

Key words: Cyanex 301, Antibacterial, Antifungal, DMSO, Bis (2, 4, 4-trimethylpentyl) dithiophosphinic acid

INTRODUCTION

The metal complexes are now the most active research field of inorganic chemistry. Today coordination chemistry stands as landmark in the area of scientific advancement embracing most diverse branches of science, engineering and technology. In the rapidly developing field of hydrometallurgy, the metal complexes have large utility in the metallurgical operations, is of great practical importance in dying, textile industries, analytical chemistry, engineering technology, and also have important functional values in biological, biomedical, environmental, industrial transformations Islam *et al.* (2007). So, authors become interested to extend their studies and reported herein the synthesis, characterization and biological screening of some new complexes of Mg(II), VO(II), Mn(II), Fe(III), Co(II), Ni(II), Cu(II) and Pd(II) with cyanex 301.

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* Corresponding author: <tpal7@yahoo.com>.

[1] Department of Chemistry, Rajshahi University of Engineering & Technology, Rajshahi Bangladesh.

[2] Department of Botany, Rajshahi University, Rajshahi Bangladesh.

MATERIALS AND METHODS

IR spectra (4000 - 400/cm) were recorded on a Nicolet 310 FTIR (Belgium) spectrometer with a KBr disk and UV-visible spectra were recorded on a 1650 PC, Shimadzu spectrophotometer in DMSO. Magnetic susceptibility measurements were obtained with a Sherwood Scientific Magnetic Susceptibility Balance at room temperature. The electrical conductivities of 10−3 M solution in DMSO were carried out on a heavy-duty conductivity /temperature meter, Extech Instruments, model No. 407303 (USA). Elemental analysis (C, H) was carried out with a Perkin Elmer 2400 II, organic elemental analyzer, Japan. The antibacterial and antifungal activities also were carried out (at Botany Department, Rajshahi University) against the bacteria *viz. Escherihia coli*, *Staphyloccocus aureus* and *Salmonella typhi* and the fungi *viz*. *Aspergillus niger*, *Fusarium oxysporum* and *Trichophyton* sp. by disc diffusion technique Pal *et al.* (2009), Gnanamanickam *et al.* (1980).

General procedure for the synthesis of metal complexes: Cyanex 301 (1 or 1.5 mmol) was dissolved in absolute ethanol (25 ml) in a beaker and KOH (1 or 1.5 mmol) was dissolved in absolute ethanol (35 ml) in another beaker. These two solutions were mixed together in a 250 ml beaker and shaken well. The resulting solution was heated on a water bath for 30 minutes and allowed to stand at room temperature. A solution of metal chloride (0.5 mmol) in absolute ethanol (20 ml) was added to the above solution of K-salt of Cyanex 301 (Metal: Cyanex 301 of 1 : 2 or 1 : 3). The solution was stirred well with a glass rod, warmed in the water bath and then allowed to stand at room temperature for 10 minutes. The precipitate of metal complex was obtained, which was filtered off, washed with cooled absolute ethanol for several times and then with distilled water (to remove KCl formed) and finally washed with hot absolute ethanol. The compound was dried in a vacuum desiccator over anhydrous $CaCl_2$.

RESULTS AND DISCUSSION

The reaction of Cyanex 301 with various metal ions [M = Mg(II), VO(II), Mn(II), Fe(III), Co(II), Ni(II), Cu(II) and Pd(II)) led to the formation of the complexes 2 - 9, in the presence of alcoholic potassium hydroxide. Cyanex 301 can act as uninegative bidentate ligand by loosing a proton from the > P (= S)SH moiety in presence of base. All the complexes were obtained via the formation of a potassium salt of the ligand. Among these complexes 2, 3 and 9 were formed in a ligand to metal ion molar ratio of 2 : 1 whereas complexes 4 - 8 were formed in 3 : 1 molar ratio. All the complexes were more air stable and insoluble in most common solvents except benzene and dimethylsulphoxide. The elemental analyses and metal estimation data were in good agreement with their proposed formulae (Table 1). The molecular mass determination of the complexes also supported their proposed molecular formulae (Table 1). The room temperature molar conductance values of the complexes suggested that the all complexes 2, 3, 5 and 9 are non-electrolyte (El-Tabl *et al.* (2008), Maurya *et al.* (2003)), while complexes 4 and 6 - 8 are electrolyte in nature (Tavman 2005). The nature of complexes also suggested that, the ligand was coordinated to metal ions as anion.

Infrared spectra: The IR spectra provide valuable information regarding the nature of functional group attached to the metal atom Raman *et al.* (2007). In order to study the binding mode of the ligand to metal in the complexes, the IR spectrum of the free ligand was compared with the corresponding metal complexes. Selected vibrational bands of the ligand and its metal complexes and their assignments are listed in Table 2. The infrared spectrum of the ligand showed two bands at 2868 and 2957/cm, which are assigned to ν(C-H) symmetric and asymmetric stretching respectively, which observed in the 2867 - 2876 and 2952 – 2954/cm region in the case of the all complexes. It can be observed that there are no considerable shifts in the ν(C-H) vibrations in the case of metal complexes compared to the ligand. It also indicated non-involvement of aliphatic C-H function in the coordination. A medium band observed at 2638/cm in the IR spectrum of the ligand assigned to ν(S-H) vibration Leka *et al.* (2004), which was found to have disappeared in all respective complexes, thereby indicating the involvement of the P-SH moiety's sulphur in bonding with metal ions through deprotonation (Leka 2004). A medium intensity band due to ν(P = S) was observed at 637/cm Rockett 1962, Shagidullin and Lipatoka (1971) in the case of ligand. This band was shifted to lower frequency (49 - 66/cm) after complexation, which suggested the coordination of thiophosphoryl group's sulphur atom to metal ions. This can be explained by the reduction of double bond character of the P = S moiety. The low frequency skeletal vibrations due to M-S stretching provided direct evidence for the complexation. Furthermore, in the present investigation the absence of ν(S-H) band as well as the presence of new weak bands in the 405 - 415/cm region for ν(M-S) vibrations (Leka 2004, Swamy *et al*. 2006) further suggested the coordination of the ligand to metal ions via sulphur atoms. The spectra of complexes 2, 4 and 6 showed a broad and medium intensity band in the 3431 - 3449/cm region, which is assigned to ν(O-H) vibrations of hydrated water molecule (Sonmez 2001). Besides, the water containing complexes did not show any weak band at nearly 860/cm indicating the absence of coordinated water molecule in the complexes Sonmez 2001, Modi *et al.* (2006). In addition, the complex 3 also showed a medium band at 950/cm attributed to V = O frequency Raman *et al.* (2007).

Magnetic susceptibility and electronic spectra: The magnetic moment values give information on their probable geometries and the electronic spectral measurements were used to assign the stereochemistries of the metal ions in the complexes based on the positions and number of d-d transition peaks (Raman *et al.* (2004). The magnetic moment values and the electronic absorption spectra of the complexes are presented in the Table 3. The electronic spectrum of the ligand showed a band at 340 nm which is assigned to the $n \rightarrow \pi^*$ transition of P = S group (Islam 2007). On complexation, this band was shifted to longer wavelength, along with an increase in its intensity. which may be due to shift by the lone pairs of the sulphur atoms of the ligand to the metal ions Liu et al. (2006). The electronic spectrum of complex 2 displayed a characteristic band at 325 nm. Since, the complex is spin paired so charge transfer band obscures its ligand field band. Therefore, Mg(II) complex does not show d-d transitions Islam *et al.* (2007). The magnetic measurements at room temperature indicated that the complex 2 is diamagnetic, consistent with tetrahedral geometry around metal ion (Islam 2007). The complex 3 displayed three bands at 769, 610 and 465 nm. On the basis of available reports Revanasiddappa *et al.* (2008,), the vanadium complex assigned the square pyramidal geometry around the VO(II) ion. The three bands may be assigned to ${}^2B_2 \rightarrow {}^2E$, ${}^2B_2 \rightarrow {}^2B_1$ and ${}^2B_2 \rightarrow {}^2A_1$ transitions, respectively. The observed magnetic moment value of the complex 3 is 1.65 B.M., which suggested square pyramidal geometry (Revanasiddappa 2008, Sakurai 1998). The electronic spectrum of the complex 4 showed bands at 667, 500 and 415 nm assignable to ${}^6A_{1g}\rightarrow{}^4T_{1g}$, ${}^6A_{1g}\rightarrow{}^4T_{2g}$ and ${}^6A_{1g}\rightarrow{}^4A_{2g}$, ${}^4Eg$ transitions, respectively, and is consistent with an octahedral environment around the manganese(II) ion Modi *et al.* (2006), Ratan *et al.* (2003), Patel *et al.* (2005). The magnetic moment value of the Mn(II) complex is 5.59 B.M. due to a high-spin d^5–system with an octahedral geometry El-Tabl *et al.* (2007), Murugan and Mohanan (2006). The absorption spectrum of high spin iron(III) complex showed three bonds at 740, 645 and 480 nm due to ${}^6A_{1g}\rightarrow{}^4A_{1g}$, ${}^6A_{1g}\rightarrow{}^4Eg(G)$ and ${}^6A_{1g}\rightarrow{}^4Eg(D)$ transitions, respectively, which are characteristic of octahedral geometry Revanasiddappa *et al.* (2008). The Fe(III) complex 5 showed the magnetic moments of 5.72 B.M., suggesting octahedral geometry around metal ion El-Tabl *et al.* (2007), Murugan *et al.* (2006). The electronic spectrum of cobalt(II) complex exhibited bands at 710, 530 and 450 nm, which assigned to ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$, ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$ and ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(P)$ transitions, respectively. These transitions suggested octahedral geometry for the cobalt(II) complex Modi *et al.* (2006), Cukurovali *et al.* (2006). The complex 6 exhibited the magnetic moment of 4.78 B.M., indicating an octahedral El-Tabl *et al.* (2007), Siddappa *et al.* (2008) arrangement of the ligand around Co (II) ion in the complex. Electronic spectrum of the complex 7 exhibited three bands in the region 720, 550 and 455 nm assignable to ${}^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$, ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ and ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ transitions, respectively, in an octahedral environment around the Ni (II) ion El-Tabl *et al.* (2007), Gudasi *et al.* (2006). The nickel (II) complex

7 was found to be paramagnetic in nature having μ_{eff} value 3.46 B.M., assuming octahedral geometry around Ni(II) ion (Figgis and Lewis 1964). The copper (II) complex 8 showed only one transition at 721 nm assigned to the $^2E_g \rightarrow ^2T_{2g}$ transition, which is in good agreement with the distorted octahedral configuration around the copper (II) ion Gudasi *et al.* (2006). Though three transitions are expected in this case, but they are very close in energy and often appear in the form of one broad band envelope Raman *et al.* (2004), Gudasi *et al.* (2006). The magnetic moment value of the Cu(II) complex is 1.92 B.M. due to the octahedral geometry Raman *et al.* (2004). The bands observed in the complex 9 at 480 and 440 nm, tentatively assigned to the spin allowed *d* - *d* transitions, i.e., $^1A_{1g} \rightarrow ^1A_{2g}$ and $^1A_{1g} \rightarrow ^1B_{1g}$ transitions, respectively consistent with a square planar geometry Bermejo *et al.* (1999). Moreover, the magnetic measurements of the complex 9 revealed that the complex was diamagnetic, consistent with square planar geometry around palladium (II) ion Leka 2004, Gudasi *et al.* (2006).

Antimicrobial activity: The biological activities of the free ligand and its metal complexes were determined at a concentration of 25 μg/disc against three pathogenic bacteria and three pathogenic fungi. The standard drugs ciprofloxacin and griseofulvin were also tested for their antibacterial and antifungal activities at the same concentration under the conditions similar to that of the test compounds. The results of biological activity tested for the free ligand and its complexes are given in Tables 4 and 5. Diameter of inhibition zone (mm) including the disc diameter was measured for each treatment. The ligand presented weak or no activity against the all tested microorganisms under identical experiment conditions. The antibacterial activity results revealed that the complex 2 showed maximum activity with the zone of inhibition 20 mm against *Escherichia coli* as compared to standard drug ciprofloxacin, which showed 24 mm inhibition. The complex 4 exhibited highest activity with the zone of inhibition 26 mm against *Staphylococcus aureus*. The complex 6 displayed moderate activity with the zone of inhibition 14 mm against *Salmonella typhi* as compared to the standard drug with 22 mm inhibition. The antifungal activity results also revealed that the complex 2 showed good activity with 20 mm inhibition, while the complexes 3 and 7 displayed moderate activity against *Aspergillus niger*, as compared to the standard drug griseofulvin (24 mm). The complex 7 exhibited almost similar activity with the zone of inhibition 21 mm against *Fusarium oxysporum* when compared to the standard drug which showed 23 mm inhibition. On the other hand, the complex 5 was found to be reasonably active with 20 mm inhibition against *Trichophyton* sp. compared to the standard drug with 26 mm inhibition. Such increased activity of the metal complexes can be explained on the basis of chelation theory. On chelation, the polarity of the metal ion will be reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of the positive charge of the metal ion with donor groups. It enhances the penetration of the complexes into lipid membranes of microorganisms.

Table 1. Analytical and physical data of the ligand and complexes.

No.	Ligand/ Complex	M. P. (ºC)	Colour	Yield (%)	Elemental analyses Expt. (Calc.) C	H	M	Λ_M (Ω^{-1} cm^2 mol^{-1})
1	Cyanex 301	-	-	-	-	-	-	-
2	$[MgL_2].H_2O$	258	White	45	55.68 (55.81)	10.02 (10.00)	03.49 (03.55)	02.42
3	$[VOL_2]$	280(d)	Green	49	54.01 (53.87)	09.68 (09.66)	07.20 (07.18)	03.45
4	$K[MnL_3].H_2O$	324	Brown	38	53.60 (53.57)	09.48 (09.55)	05.06 (05.11)	55.10
5	$[FeL_3]$	260(d)	Reddish brown	40	56.42 (56.52)	10.01 (10.08)	05.40 (05.48)	05.02
6	$K[CoL_3].H_2O$	323	Dark brown	50	53.30 (53.37)	10.00 (09.52)	05.50 (05.46)	50.09
7	$K[NiL_3]$	315	Light green	49	53.40 (53.38)	09.47 (09.52)	05.46 (05.43)	53.41
8	$K[CuL_3]$	330	Dark brown	56	53.12 (53.14)	09.50 (09.48)	05.81 (05.86)	46.27
9	$[PdL_2]$	269	Yellow	62	51.08 (51.03)	09.12 (09.15)	14.16 (14.20)	02.03

L= Cyanex 301 anion.

Table 2. The most important bands in the IR spectra of the ligand and complexes/cm.

No.	Ligand / Complex	ν(O-H)[a]	ν(S-H)	ν(P=S)	ν(M-S)	ν(V=O)
1	Cyanex 301	-	2638 m	637 m	-	
2	$[MgL_2].H_2O$	3449 b		588 m	407 w	
3	$[VOL_2]$	-		586 m	410 w	950 m
4	$K[MnL_3].H_2O$	3431 b		580 m	405 w	
5	$[FeL_3]$	-		582 m	410 w	
6	$K[CoL_3].H_2O$	3440 b		571 m	415 w	
7	$K[NiL_3]$	-		581 m	406 w	
8	$K[CuL_3]$	-		574 m	410 w	
9	$[PdL_2]$	-		580 m	407 w	

[a] Water.

Table 3. The molecular mass, magnetic moment and electronic spectra of the ligand and complexes.

No.	Ligand/ complex	Molecular mass in g/mol		μ_{eff} (B. M.)	Band observed (nm)	Band assignment
		Expt.	Calc.			
1	Cyanex 301	-	322	-	340	$n \rightarrow \pi^*$
2	$[MgL_2].H_2O$	684.15	685.07	Diam.	325	$n \rightarrow \pi^*$
3	$[VOL_2]$	709.98	709.70	1.65	769, 610, 465	$^2B_2 \rightarrow {}^2E, {}^2B_1, {}^2A_1$
4	$K[MnL_3].H_2O$	1075.80	1076.20	5.59	667, 500, 415	$^6A_{1g} \rightarrow {}^4T_{1g}, {}^4T_{2g}, {}^4A_{2g}$
5	$[FeL_3]$	1020.25	1020.00	5.72	740, 645, 480	$^6A_{1g} \rightarrow {}^4T_{1g}, {}^4E_g(G), {}^4E_g(D)$
6	$K[CoL_3].H_2O$	1080.40	1080.20	4.78	710, 530, 450	$^1T_{1g} \rightarrow {}^4T_{2g}(F), {}^4A_{2g}(F), {}^4T_{2g}(P)$
7	$K[NiL_3]$	1080.02	1079.90	3.46	720, 550, 455	$^3A_{2g}(F) \rightarrow {}^3T_{2g}(F), {}^3T_{1g}(F), {}^3T_{1g}(P)$
8	$K[CuL_3]$	1084.68	1084.80	1.92	721	$^2E_g \rightarrow {}^2T_{2g}$
9	$[PdL_2]$	750.04	749.20	Diam.	480, 440	$^1A_{1g} \rightarrow {}^1A_{2g}, {}^1B_{1g}$

Table 4. Antibacterial activity of the ligand and its complexes.

No.	Ligand/ complex	Antibacterial activity (zone of inhibition in mm)		
		Esherihia coli (25 µg/disc)	*Staphyloccocus aureus* (25 µg/disc)	*Salmonella typhi* (25 µg/disc)
1	Cyanex 301	07	00	08
2	$[MgL_2].H_2O$	20	10	09
3	$[VOL_2]$	00	00	00
4	$K[MnL_3].H_2O$	00	26	00
5	$[FeL_3]$	00	00	00
6	$K[CoL_3].H_2O$	00	00	14
7	$K[NiL_3]$	10	00	00
8	$K[CuL_3]$	10	10	00
9	$[PdL_2]$	00	00	00
	Ciprofloxacin	24	22	22
	DMSO (Control)	00	00	00

Table 5. Antifungal activity of the ligand and its complexes.

No.	Ligand/ complex	Antifungal activity (zone of inhibition in mm)		
		Aspergillus niger (25 μg/disc)	*Fusarium oxysporum* (25 μg/disc)	*Trichophyton* sp. (25 μg/disc)
1	Cyanex 301	00	07	00
2	$[MgL_2].H_2O$	20	10	12
3	$[VOL_2]$	12	10	08
4	$K[MnL_3].H_2O$	07	00	00
5	$[FeL_3]$	10	09	20
6	$K[CoL_3].H_2O$	07	00	00
7	$K[NiL_3]$	12	21	00
8	$K[CuL_3]$	00	00	10
9	$[PdL_2]$	10	10	09
	Griseofulvin	24	23	26
	DMSO (Control)	00	00	00

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