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Research Article

SYNTHESIS, STRUCTURE AND ANTIMICROBIAL STUDIES OF FOUR NEW N_2O_4 SCHIFF BASE CONTAINING COMPLEXES OF Ag(I), Cr(III), Fe(III) AND Sb(III) METAL IONS

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ABSTRACT

Ag(I), Cr(III), Fe(III) and Sb(III) complexes of bis(indoline-2-one)triethyelenetetraminewhich is madefrom isatinandtriethylenetetraminewere prepared and characterized by physical properties, spectral and magnetic methods. Based on the analytical results, electronic and infrared spectral data and magnetic susceptibility measurements, suitable structures have been proposed. The antimicrobial results indicate that the cadmium complex exhibit more activity than the palladium (II), mercury and zirconium (II) complexes.All complexes were found to have strong to moderate antibacterial activity against the tested bacteria with no cytotoxic/antifungal effect.

INTRODUCTION

Isatin and its derivatives are special members in the Schiff base family. The simple isatin based Schiff base compounds having, acyl, aroyl and heteroacroyl Schiff bases have addi- tional donor sites >C=O, >C=N-, etc. These donor sites make them more flexible and versatile. This versatility has made them good chelating agents that can form a variety of complexes with various transition and inner transition metals and has attracted the attention of many researchers (Medvedev *et al.,* 1996). Moreover, Schiff bases are regarded as privileged ligands, due to their capability to form complexes with different transition metals can act as catalysts for many different reactions (Yamada *et al*., 2006; Cai *et al*. 2004). Recently, complexes of type, [ML]Cl(2) [M=Cd(II), Pd(II), Hg(II) and Zr(IV)] were reported (Anarul Islam et al. 2014). Where, the Schiff base ligand bis (indoline-2-one) triethylenetetramine (L) obtained from condensation of triethylenetetramine and isatin. Furthermore,Isatin based complexes show variety of biological activities such as potentiation of pentabarbitone induce nercosis (Watkins *et al*., 1990), anti-inflammatory (Glover *et al*., 1998), anti-convulsant (Popp *et al*., 1980), analgesic (Chinnasamy *et al*., 2010), anti-bacterial (Pandeya and Sriram, 1998), anti-fungal (Varma RS and Nobles WL 1975), and anti-HIV (Pandeya *et al*., 2000). Earlier, we studied electronic properties of nickel(II) complexes in solution (Shiraj-U-Ddaula *et al*., 2014; Sakiyama and Kudrat-E-Zahan, 2011; Kudrat-E-Zahan *et al*., 2010). In this study, we are motivated to undertake a systematic study of preparation, characterization and Antimicrobial propertiesof transition metal complexes formed with bis (indoline-2-one) triethyelenetetramine(L) and Ag(I), Cr(III), Fe(III) and Sb(III) ions.

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Experimental

Measurements and materials

Electronic spectra were recorded on a Thermoelectron Nicolet evolution 300 UV-Vis spectrophotometer. All chemicals were commercial products and were used as supplied.

Synthesisofbis(indoline-2-one)triethylenetetramine

To a stirring solution of isatin (0.294g, 2 mmol) dissolved in 25ml of ethanol, a solution of triethylenetetramine (0.16ml,1mmol) in 10ml ethanol was added drop wise. This has resulted a dark orange solution, which was refluxed for 6h.The reaction mixture was cooled and kept for evaporation at room temperature leading to isolation of solid product. The product thus formed was filtered

washed several times with ethanol and finally with diethyl ether. The product was found to be soluble in DMF and DMSO.

Preparation of Complexes

1 mmol solution of a metal chloride/ nitrate (where, M =Ag(I), Cr(III), Fe(III) and Sb(III) metal ions) dissolved in ethanol (15 ml) was taken in a two necked round bottom flask and kept on magnetic stirring. To this solutions of isatin (0.294 g, 2 mmol) dissolved in 25 ml of ethanol and triethylenetetramine (0.16 ml, 1 mmol) in ethanol (10 ml) were simultaneously added Drop wise followed by refluxing for about (6-10 h) leading to isolation of microcrystalline product. The complexes thus formed were filtered and washed several times with ethanol to remove any traces of unreacted starting materials and were further washed with diethyl ether and dried in vacuum over anhydrous $CaCl_2$. The complexes were soluble in DMF and DMSO.

RESULTS AND DISCUSSION

During the course of the reactions Schiff base ligand bis(indoline-2-one)triethyelenetetramine were first formed which the coordinated with Ag(I), Cr(III), Fe(III) and Sb(III) metal ions. The reactions of metal complexes may be represented as follows:

$MX_2 + L \rightarrow [M(L)](X)_{1/3}$

Where,

$L = H_{24}C_{22}N_6O_2$ [bis(indoline-2-one)triethyelenetriamine and
$X = Cl/(NO)_3$

Physical properties of the complexes

Some physical properties of the complexes are shown in the (Table 1). The molar conductance values are in the region range 7 to 13 $\Omega^{-1}{}_{cm}{}^{2}{}_{mol}{}^{-1}$. These values are lower than expected for an electrolyte. So molar conductance values indicate that the complexes are non-electrolyte in nature. The melting point ranges from 245-300.

VIS spectrum of the complex, $[SbL](NO_3)_3$exhibited band at 350 nm, arising from charge transfer transition. The magnetic susceptibility measurement showed that the complex was paramagnetic in nature. The complexes were octahedral in geometry. The observed magnetic moment values of Fe(III) (5.91 B.M.) indicated that the complex $[FeL]Cl_3$ is paramagnetic with five unpaired electrons and hence, have Octahedral structure with sp^3d^2 hybridization. The electronic spectra of Fe(III) complex gave four bands at 536,480,410 and 390nm corresponding to the transitions [$^6A_{1g} \rightarrow {}^4T_{1g}(G)$; $^6A_{1g} \rightarrow {}^4T_{2g}(G)$; $^6A_{1g} \rightarrow {}^4E_g(G)$;$^6A_{1g} \rightarrow {}^4A_{1g}(G)$], respectively. These spectra are very much consistent with the octahedral stereochemistry of Fe(III) compound. The observed magnetic moment of all Cr(III) complexes are 3.81 B.M. which corresponds to three unpaired electron and indicated the paramagnetic and octahedral geometry. The reflectance spectra of Cr(III) complexes consist of transition band 265 to 550 nm assignable to the transitions $^4A_{2g} \rightarrow {}^4T_{2g}$, $^4A_{2g} \rightarrow {}^4T_{1g}$ (F) and $^4A_{2g} \rightarrow {}^4T_{1g}$ (P) respectively.

Infrared spectra: IR spectral data are shown in (Table 3). The strong bands obtained at around 3450, 1700 and 1620 cm^{-1} due to υ(-NH), υ(C=O) and υ(C=N) respectively. The presence of metal ligand bonding is evident from the appearance of υ(M-O), υ(M-N) and υ(M-N)(-NH) at around 700, 600 and 500 cm^{-1} respectively in the spectra of the complexes. Based on these facts a probable following structure for all complexes have been proposed

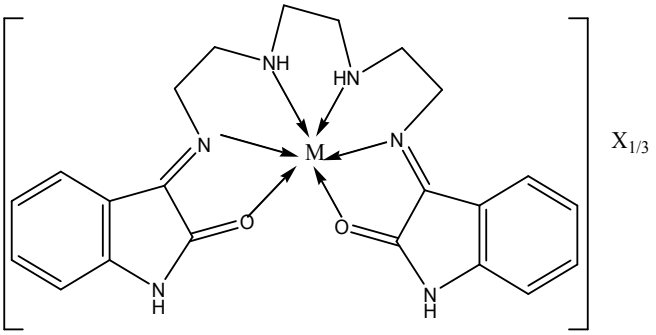

Figure 1. Proposed structure of the complexes

Table 1. Physical properties of complexes

Complexes	Molar conductance ($ohm^{-1}cm^2mol^{-1}$)	Magnetic moment (B.M)	Melting point (±0.5°c)	Colour	d→d (λ/nm)
$[AgL]NO_3$	12.42	1.40	251	Black	---
$[CrL]Cl_3$	7.93	3.81	245	Black	550
$[FeL]Cl_3$	13.00	5.91	260	Brown	536
$[SbL](NO_3)_3$	7.16	1.99	300	White	---

Where, $L=H_{24}C_{22}N_6O_2$

Table 2. Major IR spectral data (cm^{-1}) with their assignment and electronic spectral data of the Schiff base complexes

Complexes	υ(-NH) cm^{-1}	υ(C=O) cm^{-1}	υ(C=N) cm^{-1}	υ(M-O) cm^{-1}	υ(M-N)(-NH) cm^{-1}	υ(M-N) cm^{-1}
$[AgL]NO_3$	3409	1699	1615	617	567	497
$[CrL]Cl_3$	3413	1706	1621	758	651	488
$[FeL]Cl_3$	3468	1706	1627	674	647	579
$[SbL](NO_3)_3$	3435	1706	1631	685	541	494

Where, $L=H_{24}C_{22}N_6O_2$

Magnetic moment and Electronic spectral studies

The magnetic moment (μ_{eff}) and d→d (λ/nm) transitions of the complexes at room temperature are given in Table 1. The UV-

Where, M= Sb(III), Cr(III), and Fe(III) and $X = Cl/(NO)_3$

Antibacterial screening: This antibacterial activity test was carried out at microbiology laboratory, Pharmacy Department,

Table 3. Antibacterial activity of complexes and standard Kanamycin

Complexes	Zone of inhibition, diameter in mm				
	Escherichia coli	*Pseudomonas aeruginosa*	*Staphylococcus Aureus*	*Salmonella typhi*	*Salmonella choleresuls*
$[AgL]NO_3$	09	11	08	11	10
$[CrL]Cl_3$	06	08	11	10	09
$[FeL]Cl_3$	13	11	09	08	07
$[SbL](NO_3)_3$	0	0	0	0	0
DMSO(control)	-	-	-	-	-
Kanamycine(30 μg)	20	22	21	21	20

Where, $L=H_{24}C_{22}N_6O_2$

Rajshahi University.As the test bacteria are pathogenic, all steps of the work were done with high precaution and aseptic conditions. The investigations of antibacterial activity were performed by disc diffusion method and isolated antibiotics were used for this purpose. The antibiotic was dissolved in DMSO and concentration (100 μ/disc) was used make a better correleation of the antibacterial activity. Kanamycin (30 μ/disc) was used as a standard. The antibacterial activity of the tested sample was determined by measuring the zone of inhibition in term of mm and was compared to that of the standard, kanamycin.All the complexes except $[SbL](NO_3)_3$showed moderate to strong antibacterial activity (Table 3).

Cytotoxicity

All the complexes did not showed any toxicity against *Asalina*nauplii and *T. castaneum*. But the triethylenetetramine showed toxicity against salinanauplii. Trien is a toxic ligand. When it form Schiff base with isatin and behaves non toxic because isatin blocks amino group of trien. So, Schiff base metal complex also behaves as non toxic. So these complexes may be used as chelation therapy. Specially, these non toxic complexes will be a very interesting topics for further study.

REFERENCES

Cai, Y.P., C.Y. Su, A.W. Xu, B.S. Kang, H.Q. Liu, S. Jie, 2001. Syntheses and characterization of copper(II) complexes of bis(acetylacetone)trimethylenediimine, *Polyhedron,* 20, 657-661

Chinnasamy, R.P., R. Sundararajan, S. Govindaraj, 2010. Synthesis, characterization, and analgesic activity of novel schiff base of isatin derivatives,*J. Adv. Pharm. Tech. Res.* 1,342-347

Glover, V., Bhattacharya, SK., Charkrabarti, A. and Sandler, M. 1998. The pharmacology of isatin: A brief review. *Stress Med*., 14, 225-9

Md. Anarul Islam, Md. Anarul Islam, Roksanakhatun, M.Monirul Islam and Md.Kudrat-E-Zahan, 2014. Synthesis, spectroscopic monitoring and biological activities of N_2O_4schiff base ligand containing metal complexes of Cd(II), Pd(II), Hg(II) and Zr(IV). *Asian Journal of Research in Chemistry*, 7(2); 163-165

Md. Shiraj-U-Ddaula, Md. Anarul Islam, Shejutyaktar, Md. Khairul Islam, Md. Abdul Alim Al-Bari, Md. MasuqulHaque and Md.Kudrat-E-Zahan, 2014. Synthesis, Characterization and Antimicrobial activity of Cd(II), Ni(II), Co(II) and Zr(IV) metal complexes of Schiff base ligand derived from diethylenetriamine and isatin. *Asian Journal of Research in Chemistry*, 7(7), 619-621

Md.Kudrat-E-Zahan, Yuzo Nishida, Hiroshi Sakiyama, 2010. Identification of cis/trans isomers of bis(acetylacetonato) nickel(II) complexes in solution based on electronic spectra. *Inorganica. Chimica. Acta*, 363(1), 168-172

Medvedev, AE., Clow, A., Sandler, M. and Glover, V.,1996. Isatin – a link between natriuretic peptides and monoamines? *BiochemPharmacol*, 52, 385-91

Pandeya, SN. and Sriram, D. 1998. Synthesis and screening for antibacterial activity of Schiff's and Mannich bases of Isatin and its derivatives. Acta Pharm Turc., 40, 33-8

Pandeya, SN., Sriram, D., Nath, G. and De Clercq, E. 2000. Synthesis, antibacterial, antifungal and anti-HIV activities of norfloxacinMannich bases. *Eur J Med Chem*., 35, 249-55

Popp, F.D., R. Parson, B.E. Donigan, 1980. Potential anticonvulsants. III. The condensation of isatin with cyclic ketones, *J. Heterocycl. Chem.* 17, 1329-1330

Sakiyama, H. and M. Kudrat-E-Zahan, 2011. Identification of metal complexes in solution based on electronic spectra – equilibrium ratio between cis/trans isomers of bis(acetylacetonato)nickel(II) complex, *New Trends in Coordination, Bioinorganic, and Applied Inorganic Chemistry*, 474-476

Varma, RS. and Nobles, WL. 1975. Antiviral, antibacterial, and antifungal activities of isatin N-Mannich bases. *J Pharm Sci*., 64, 881-2

Watkins, P., Clow, A., Glover, V., Halket, J., Przyborowska, A. and Sandler, M.,1990. Isatin, regional distribution in rat brain and tissues. *Neurochem Int*., 17, 321-3

Yamada, T., T. Ikeno, Y. Ohtsuka, S. Kezuka, M. Sato, I. Iwakura, 2006. Manganese and cobalt 3-oxobutylideneaminato complexes: Design and application for enantioselective reactions, *Sci. Technol. Adv. Mater.* 7,184-196

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