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Synthesis, spectroscopic characterization and *In-vitro* antibacterial properties of some metal(II) complexes of schiff bases containing aminoindane moiety

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ABSTRACT

New VO(IV), Mn(II), Co(II) and Ni(II) complexes of the Schiff bases, 2-[(2,3-dihydro-1*H*-inden-4-ylimino)methyl]-5-nitrophenol (HL^1) and 3-[2,4-dihydro-1H-inden-4-ylimino) methyl]napthalen-2-ol (HL^2) have been synthesized and characterized by elemental analysis, molar conductance, infrared and electronic spectral measurements. The purity of the ligands is confirmed by microanalyses and 1HNMR. The IR spectra show that the ligands are bidentate coordinating via azomethine N and napthalenol/phenol O atoms, and the metal complexes of HL^1 exhibit geometric isomerism being in the Cis-form with a lone *ν*C=N band. The metal complexes all assume a 4-coordinate tetrahedral/square planar geometry with exception of the VO(IV) complex which is 5-coordinate square-pyramidal as corroborated by electronic spectra. None is an electrolyte in nitromethane. The Schiff base, HL^2, and its metal complexes have higher molar absorptivities of the ligand bands than HL^1 and its metal complexes; due to a closer metal to ligand overlap, a consequence of extended conjugation in the naphthalene ring. The in-vitro antibacterial activities of these complexes and their Schiff bases against *Staphylococcus aureus, Bacillus cereus, Escherichia coli, Proteus mirabilis, Klebsiella oxytoca* and *Pseudomonas aeruginosa* shows that HL^1 and its metal complexes are more active than HL^2 and its metal complexes, and the latter compounds have selective inactivity against *Bacillus cereus* and *Klebsiella oxytoca*. It is note worthy that $[CoL^1]_2$ exhibited broad-spectrum antibacterial activity like the antibiotic ciprofloxacin (26.0-30.5 mm), although with smaller inhibitory zones in the range 12.5-17.0 mm.

Introduction

Aminoindane and its derivatives are renowned bronchodilators [1], analgesics, protease inhibitors and anticonvulsants [6-9], anti-HIV and anticancer agents [2-5]. Moreover, tridentate aminoindane Schiff base Cr(III) compounds are good catalysts in Diels-Alder reactions of α,β-unsaturated aldehydes, and ring opening of mesoaziridines [6-7]. Furthermore, Schiff bases derived from indane-1,3-dione-2-imine-N-acetic acid, 2-imino-N-2-propionic acid and ninhydrin, glycine/L-alanine and their metal(II) complexes exhibit unique geometries, and good antimicrobial activities against *E. coli, P. mirabilis, S. aureus* and *P. faecalis* [8-9].

Extensive literature search revealed a dearth of information on the Schiff bases, 2-[(2,3-dihydro-1*H*-inden-4-ylimino)methyl]-5-nitrophenol (HL^1) and 3-[2,3-dihydro-1H-inden-4-ylimino)methyl] napthalen-2-ol (HL^2) and their VO(IV), Mn(II), Co(II) and Ni(II) complexes [10-14];except for our recent work on the synthesis and *in-vitro* anticancer properties of Cu(II), Zn(II) and Pd(II) complexes of HL^1 [15]. Thus, our aim is to synthesize, characterize and investigate the effect of changing the phenyl ring and nitro substituent in HL^1 to a naphthalene ring in HL^2 on the electronic and antibacterial properties of their metal(II) complexes, as a continuation of the research activities of our group on the synthesis and physicochemical properties of various Schiff base complexes [16-18].

Experimental

Materials and reagents

Reagent grade 2-hydroxy-1-naphthaldehyde, 2-hydroxy-5-nitrobenzaldehyde, 4-aminoindane, triethylamine, vanadyl sulphate monohydrate, nickel(II) nitrate hexahydrate, manganese(II) nitrate hexahydrate and cobalt(II) nitrate hexahydrate were purchased from Aldrich chemicals and were used as received.

Syntheses

Preparation of compounds

Preparation of 3-[2,4-dihydro-1H-inden-4-ylimino)methyl]napthalen-2-ol, HL^2, The ligand was prepared by refluxing homogeneous solution of 1.94 g (1.13×10^{-2} moles) 2-hydroxy-1-napthaldeyde and 1.5g (1.13×10^{-2} moles) of 4-aminoindane in 60 mL of hot ethanol to which 4 drops of acetic acid were added for 3 hours. The orange products (Figure 1) formed on cooling in ice were collected by suction and recrystallized from ethanol and dried over silica gel. The yield of the product was 2.59 g(80%). The same procedure was used to prepare 2-[(2,3-dihydro-1*H*-inden-4-ylimino)methyl]-5-nitrophenol, HL^1 [15].

HL^1; ^{1}H NMR (300 MHz, $CDCl_3$, δ in ppm): 15.0(s, 1H, C^2 OH), 8.78(s, 1H, HC^7N), 8.42-8.22 (m, 3H, C^3, C^4, C^6), Indane ring: 7.29-7.06 (m, 3H, $C^{5'}$, $C^{6'}$, $C^{7'}$); 3.02 (t, 2H, $C^{1'}$), 2.15 (q, 2H, $C^{2'}$), 3.05(t, 2H, $C^{3'}$).

HL^2; ^{1}Hnmr (300 MHz, $CDCl_3$, δ in ppm): 15.76 (s, OH), 9.4 (s, HCN), 7.30 - 8.14(m, 6H, $C_{10}H_6$); 7.0-7.27 (m, 3H, {$C^{5'}$

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C $^{6'}$ C $^{7'}$ H_3}); 3.07-3.12(t, 2H, $C^{3'}H_2$); 2.98-3.05 (t, 2H, $C^{1'}H_2$); 2.10-2.22 (q, 2H, $C^{2'}H_2$).

Preparation of the metal complexes

The various complexes were prepared by refluxing a homogeneous solution of 0.30 mmol (0.05-0.09 g) of hydrated M(II) nitrates (M = Co, Ni, Mn,) and 0.60 mmol (0.17 g) of the ligand, to which 0.06 mmol (0.06 g) of triethylamine was added in 30 mL ethanol for 3 h. The product precipitated as solids, which were filtered, washed with ethanol and dried over silica gel. The VO(IV) complex was prepared using the same method from its sulphate salt.

Antibacterial Assay

The assay was carried out on the Schiff bases and their metal(II) complexes. The bacteria used were identified laboratory strains of *E. coli, B. cereus, S. aureus, P. mirabilis, K. oxytoca* and *P. aeruginosa.* The antibacterial susceptibility test was carried out using the agar well diffusion technique [5].

The surface of the agar plate (Muller Hinton) was uniformly inoculated with 0.3 mL of 18 h old test bacteria culture (10^6 CFU/ mL). Using a sterile cork borer, 9 mm wells were bored into the agar. Then 0.06 mL of 10 mg/mL concentration of each metal complex in DMSO was introduced into the wells and the plates were allowed to stand on the bench for 30 minutes before incubation at 37^0C for 24 h, after which inhibitory zones (in mm) were taken as a measure of antibacterial activity. The experiments were conducted in duplicates and ciprofloxacin was used as the reference drug.

Physical measurements

The electronic and infrared spectra were recorded on a Perkin-Elmer λ25 and Thermo Nicolet FTIR 200 spectrophotometers.

The ^{1}H nmr spectrum was recorded on a 300 MHz Brucker DRX-400 NMR instrument in $CDCl_3$ at 295K, and ^{1}H chemical shifts were referenced to the residual signals of the protons of $CDCl_3$ and were quoted in ppm.

The elemental analyses for C, H and N were recorded on Thermo Quest CE Instruments flash EA1112 analyser, while percentage manganese, cobalt, nickel were determined titrimetrically, and percentage vanadium was determined gravimetrically [19]. Electrolytic conductivities in nitromethane and melting points (uncorrected) were determined using a HANNA HI 991300 conductivity meter and Mel-Temp electro thermal machine respectively.

Results and discussion

The reactions for the formation of the metal(II) Schiff base complexes are represented by equations 1 and 2 respectively. The compounds were formed in good yields of 60-70%.

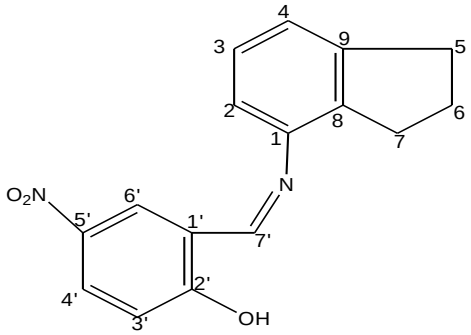
$M(NO_3)_2.6H_2O + 2HL \rightarrow [ML_2]aH_2O + 2HNO_3 + xH_2O$ (1)

(When L = L^1, M = Ni/Co {a = 0, x = 6})

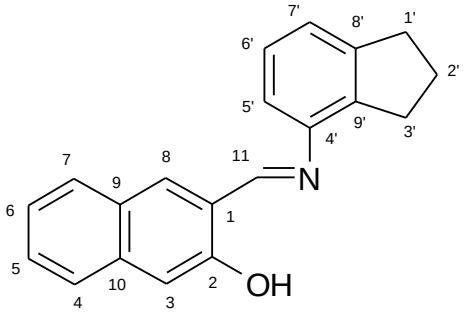
(When L = L^2, M = Mn {a = 4, x = 2}, M = Co {a = 3, x =3}; Ni {a = 1, x =5})

$VOSO_4.2H_2O + 2HL^1 \rightarrow [VOL^1{}_2]0.5H_2O + H_2SO_4 + 1.5H_2O$ (2)

The structural formulas of the ligands are provided in Figure 1 and their formation is confirmed by microanalysis and 1HNMR.



(HL^1)



(HL^2)

Figure 1: The proposed structure for the Schiff bases.

The Schiff bases HL^1 and HL^2 have melting points of 152-153 and158-160 ^{o}C, whereas their metal complexes melt/decompose in the range164-332 and 163-308 ^{o}C respectively, collaborating coordination. The water molecules associated with the complex formation are outside the coordination sphere; because heating the hydrated complexes in programmed oven in the temperature range 80-100^{o}C for 2-3 hours leads to dehydration, with corresponding weight loss of 0.5 - 4 water molecules. It has been documented that water of coordination is usually eliminated in the temperature range 120–250^{o}C [20]. Single crystal of the complexes could not be isolated from any solution. Hence, analytical and spectroscopic data(Table 1) have been used to derive structures, and the complexes are reported here for the first time.

Conductance measurements

The metal(II) complexes are non-electrolytes in nitromethane as shown by their molar conductivities (Λ_m) of 1.2-14.5 (L^1) and 28.0-30.0 (L^2) $ohm^{-1}cm^2mol^{-1}$, which are lower than reported value of 60-118 ohm^{-1} cm^2mol^{-1} for a 1:1 electrolyte [21].

Electronic Spectra

The electronic spectra are presented in Table 2. The ultraviolet spectra of the compounds are characterized by three peaks at 28.7-31.8, 32.6-36.5 and 40.1-42.7 kK for L^1 complexes with molar absorptivities of 10^4 – 10^5 M^{-1} cm^{-1}; while L^2 complexes have three bands which are bathochromic shifted relative to L^1 complexes, but with higher molar absorptivities of 10^5 - 10^6 M^{-1} cm^{-1}, at 24.5-31.1, 31.2-31.7 and 40.8- 42.0 kK respectively. These bands are assigned as charge transfer and π-π* transitions [8-9]. The molar absorptivities of all the complexes in the visible region are in the range 10^2-10^3 M^{-1} cm^{-1} ruling out octahedral geometry since octahedral complexes have molar absorptivities in the range 1-50 M^{-1} cm^{-1} [22]. The electronic spectrum of $[VOL^1{}_2]$ shows two absorption bands at 14.00 and 22.35 kK which indicates a five coordinate, square-pyramidal geometry with the assignment $b_2 \rightarrow e\pi^*$ band(II) and $b_2 \rightarrow a_1$ band (III) respectively [22]. $[MnL^2{}_2]$ has two weak bands at 14.29 and 21.55 kK in the visible region,

typical of a tetrahedral geometry and are assigned to the forbidden transitions 6A1 → $^4E_1$ (ν_1) and $^6A_1$ → $^4A_1$ (ν_2) respectively [11]. The visible spectra of the [CoL^1_2] and [CoL^2_2] have two absorption bands each in the range 15.87-17.54 and 21.0-22.42 kK assigned to $^4A_2 \rightarrow {}^4T_1(F)$, ($\nu_2$), and $^4A_2 \rightarrow {}^4T_1(P)$, ($\nu_3$) transitions of a tetrahedral geometry [5]. The electronic spectra of [NiL^1_2] and [NiL^2_2] have two absorption bands in the range 16.35-16.40 and 22.22-22.37 kK assigned to $^3T_1(F) \rightarrow {}^3T_2$ ($\square_1$) and $^3T_1(F) \rightarrow {}^3A_2(\nu_2)$ transitions, of a 4-coordinate tetrahedral geometry [18, 23-24].

The higher molar absorptivities of the ligand bands of HL^2 and its metal complexes are attributed to more intimate overlap of the naphthalene ligand with the metal ions, a consequence of extended conjugation [9].

Infrared Spectra

The assignments of the infrared bands are made by comparing the spectra of the compounds with reported literature on similar systems [8-9]. The metal-free ligand, HL^1, has the uncoordinated C=N stretching vibrations as a lone band at 1650 cm^{-1}. This band still remains one, and mostly hypsochromic shifted to 1670-1655 cm^{-1} in its metal complexes, indicative of geometric isomerism with the complexes being in the Cis-isomeric form. On the contrary, the metal-free ligand, HL^2, has its uncoordinated C=N stretching vibrations as three bands at 1620-1540 cm^{-1}, and are mostly bathochromic shifted to 1617-1505 cm^{-1} in its metal complexes as three to four bands. These shifts confirm coordination through the imine nitrogen atom [15-16]. The strong bands at 3427 and 3424 cm^{-1} in HL^1 and HL^2 are assigned as □OH stretching frequency, and are conspicuously absent in the spectra of the complexes, indicative of the deprotonation and involvement of the napthalenol /phenol O atoms in chelation [14]. The new broad bands, at 3500 cm–1, in the hydrated complexes are assigned to □ (OH) crystallization water. The *v*(V=O) band of the vanadyl complex is strong at 976 cm^{-1}, which confirms its monomeric nature since polymeric complexes have *v*(V=O) in the range 848-860 [22]. The presence of bands due to □(M—O) and □(M—N) at 465-425 and 560-520 cm^{-1}; and 484-414 and 582-500 cm^{-1} in L^1 and L^2 complexes respectively, is further evidence of coordination [25-26].

Antibacterial activities

The antimicrobial data are presented in Table 3 and shown in Figure 2. HL^1 is active against all the bacteria used with exceptions of *K. oxytoca and P. aeruginosa* with inhibitory zones range of 12.5-15.0 mm. [VOL^1_2] has the least activity being active against just two organisms .i.e. *B. cereus* and *P. mirabilis* with inhibitory zones of 14.0 and 18.5 mm.

Furthermore, [NiL^1_2] is active against all the bacteria with the exception of *P. aeruginosa* with inhibitory zones in the range of 14.5-16.5 mm. [CoL^1_2] has the best activity against all the bacteria with inhibitory zones range of 12.5-17.0 mm, thus, proving its potential usefulness as a broad-spectrum antibacterial agent. It is interesting to note that HL^1 and its metal complexes are active against *B. cereus* and *P. mirabilis* while HL^2 and its complexes are inactive against both *B. cereus* and *K. oxytoca*. Additionally, HL^2 and its complexes are less active in comparison with their L^1 analogues e.g. HL^2 showed activity against three organisms namely; *P. aeruginosa*, *S. aureus*, and *P. mirabilis* with inhibitory zones range of 13.0-16.5 mm, while [CoL^2_2] has the least activity being active against two organisms *E. coli* and *P. mirabilis* with inhibitory zones of 15.0 and 16.5 mm respectively.

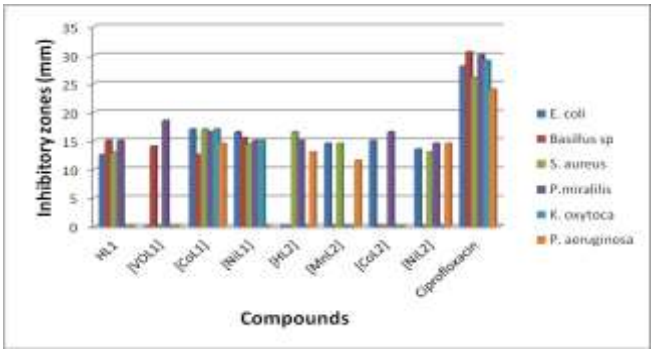


Figure 2: The inhibitory effect of the compounds against various bacterial isolates.

[MnL^2_2] is active against three bacteria, *E. coli, S. aureus* and *P. aeruginosa* with inhibitory zones range of 11.5-14.5 mm, and [NiL^2_2] has the best activity, being active against four organisms, *E. coli*, *S. aureus*, *P. mirabilis* and *P. aeruginosa* with inhibitory zones range of 13.0-14.5 mm. The improved antibacterial activities of L^1 compounds may be attributed to the presence of the nitro group which is renowned for its antimicrobial activity, and their better lipophilic nature [24]. In most cases, the metal complexes are generally more active than the metal free ligand due to chelation which increases antimicrobial activity, because of partial sharing of its positive charge with donor groups of the ligand and possible π-electron delocalisation which increased the lipophilic character [25]. The resistance of *B. cereus* and *K. oxytoca* to HL^2 and its metal complexes is attributed to production of extended beta-lactamases which inactivates the compounds [26].

Conclusion

The Schiff bases, 2-[(2,3-dihydro-1*H*-inden-4-ylimino)methyl]-5-nitrophenol (HL^1) and 3-[2,4-dihydro-1H-inden-4-ylimino)methyl]napthalen-2-ol (HL^2) coordinates to VO(IV), Mn(II), Co(II) and Ni(II) ions via azomethine N and napthalenol/phenol O atoms. The complexes assume a four-coordinate tetrahedral/square planar geometry with the exception of the VO(IV) complex which is 5-coordinate square-pyramidal. The Schiff base, HL^2 and its metal complexes have higher molar absorptivities of the ligand bands than HL^1 and its metal complexes; due to a closer metal to ligand overlap, a consequence of extended conjugation in the naphthalene ring. On the contrary, HL^1 and its metal complexes have better in-vitro antibacterial activities than HL^2 and its metal complexes which may be attributed to the presence of the nitro group and better lipophilic nature.

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Table 1 Analytical data for the compounds

Compound (Empirical formula)	Formula mass	Color	% Yield	Λ_m*	M.p (°C)	Analysis (Calculated)			
						%C	%H	%N	% M
HL^1 $C_{16}H_{14}N_2O_3$	282.29	Orange	70	-	152-153	68.4 (68.1)	4.8 (5.0)	9.5 (9.9)	-
$[VOL^1{}_2]0.5H_2O$ ($CoC_{32}H_{26}N_4O_6$)	638.51	Brown	70	1.9	164-166	60.5 (60.2)	4.8 (4.3)	8.6 (8.8)	8.0 (8.0)
$[CoL^1{}_2]$ ($CoC_{32}H_{26}N_4O_6$)	621.49	Peach	70	1.2	280-282	61.8 (61.8)	4.5 (4.2)	8.2 (9.0)	9.8 (9.5)
$[NiL^1{}_2]$ ($CoC_{32}H_{26}N_4O_6$)	621.49	green	70	14.5	320-332	61.5 (61.9)	4.1 (4.2)	8.5 (9.0)	9.2 (9.5)
HL^2 ($C_{20}H_{17}NO$)	287.36	Golden Yellow	70	-	158-160	83.6 (83.6)	5.8 (6.0)	4.7 (4.9)	-
$[MnL^2{}_2]4H_2O$ ($MnC_{40}H_{40}N_2O_6$)	699.72	Brown	60	28.0	163-165	68.7 (68.7)	4.9 (5.8)	3.8 (4.0)	7.9 (7.9)
$[CoL^2{}_2]3H_2O$ ($CoC_{40}H_{38}N_2O_5$)	685.67	Red	60	28.0	210-212	69.6 (70.1)	4.7 (5.6)	3.6 (4.1)	8.5 (8.6)
$[NiL^2{}_2]H_2O$ ($NiC_{40}H_{34}N_2O_3$)	649.43	Green	70	30.0	308[b]	74.1 (74.0)	4.8 (5.3)	3.8 (4.3)	9.1 (9.0)

*Ω^{-1} cm^2 mol^{-1}, b = decomposition temperature

Table 2 Infrared (cm^{-1}) and electronic (kK) spectral data for compounds

Compound	*v*OH	*v* (C=N)	*v* (M-N)	*v* (M-O)	λ_{max} (ε)
HL^1	3427s	1650s	-	-	28.7 (1x 10^4), 36.5(1x 10^4), 42.7(1x 10^4)
$[VOL^1{}_2]0.5H_2O$	3500b	1611 s	540s 523m	456s 434s	14.0(100), 22.4(2000), 28.7(3000), 32.6(1x 10^4), 39.3(1x 10^5), 41.5 (1x 10^5)
$[CoL^1{}_2]$	-	1655s	551s 520m	450s 428m	17.5(200), 22.4(2000), 31.8(1x 10^4), 40.2(1x 10^4)
$[NiL^1{}_2]$	-	1670s	560s 530s	465m 425m	16.4(100), 22.4(2000), 33.0(1x 10^4), 40.1(1x 10^5)
$[HL^2]$	3424s	1620s 1580s 1540s	-	-	24.5 (1x 10^5), 31.3(1x 10^5) , 41.3(1x 10^6)
$[MnL^2{}_2]4H_2O$	3500b	1622s 1582s 1542s	514s 500m	443s 414s	14.3(100), 21.6(3000), 31.3(1x 10^5), 41.5 (1x 10^6)
$[CoL^2{}_2]3H_2O$	3500b	1617s 1602s 1568s 1533s	563s 514m	457s 414m	15.9(100), 21.0(3000), 31.7(1x 10^5), 42.0(1x 10^6)
$[NiL^2{}_2]H_2O$	3500b	1600s 1578s 1535s 1505s	582s 550s	484m 428m	16.4(100), 22.2 (1000), 31.1(1x 10^5), 40.8(1x 10^6)

b = broad, m = medium; s = strong; 1kK = 1000 cm^{-1}

Table 3: Antibacterial activities of the Schiff bases and their complexes

Complexes	*E. coli*	*Bacillus cereus*	*S. aureus*	*P. miralilis*	*K. oxytoca*	*P. aeruginosa*
HL^1	12.5±0.7	15.0±0	13.0±0	15.0±0	R	R
$[VOL^1_2]0.5H_2O$	R	14.0±0	R	18.5±0.7	R	R
$[CoL^1_2]$	17.0±0	12.5±0.7	17.0±0.7	16.5±0.7	17.0±0.7	14.5±0.7
$[NiL^1_2]$	16.5±0.7	15.5±0.7	14.5±0.7	15.0±0.7	15.0±0.7	R
$[HL^2]$	R	R	16.5±0.7	15.0±0	R	13.0±0
$[MnL^2_2]4H_2O$	14.5±0.7	R	14.5±0.7	R	R	11.5±0.7
$[CoL^2_2]3H_2O$	15.0±0	R	R	16.5±0.7	R	R
$[NiL^2_2]H_2O$	13.5±0.7	R	13.0±0	14.5±0.7	R	14.5±0.7
Ciprofloxacin	28.0±1.4	30.5±0.1	26.0±1.4	30.0±1.4	29.0±0.0	24.0±1.4

R = resistance