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Synthesis, structure and antibacterial activity of manganese(III) complexes of a Schiff base derived from furfurylamine

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1. Introduction

Coordination chemistry of manganese in various oxidation states has long been investigated as an area of considerable interest in inorganic biochemistry [1]. A large number of Schiff base complexes of manganese(III) find important roles in metalloenzymes, redox and non-redox proteins [2,3]. They possess suitable biometric properties that can mimic the structural features of the active site [4–6]. In recent years, there has been enhanced interest in the synthesis and characterization of such complexes not only due to their biological importance, but also for their interesting catalytic [7] and magnetic properties [8]. Schiff bases derived from salicylaldehyde and various amines have been extensively used to synthesize many complexes of manganese(III). However, complexes with furfurylamine as the amine counterpart of the Schiff base have received very scanty attention. A report on the preparation and characterization of such complexes of Cu(II), Ni(II) and Co(II) has only appeared very recently [9]. However, report on synthesis of complexes of manganese(III) by preparation of the Schiff base in situ or along with any pseudohalide is lacking. Again, the investigation of biological properties of Schiff base complexes of Mn(III) have received less attention in comparison to the corresponding Mn(II) complexes. A few reports on the antimicrobial activity of Mn(III) complexes have been reported recently [10-12].

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ABSTRACT

Two new mononuclear complexes of manganese(III) viz. $[MnL_2(LH)_2]ClO_4$ (1) and $[MnL_2(N_3)]$ -0.5CH₃OH (2) have been synthesized by reacting manganese perchlorate with furfurylamine and salicylaldehyde (plus sodium azide in 2) where L = (2-hydroxybenzyl-2-furylmethyl)imine, an asymmetric bidentate Schiff base formed *in situ* to bind the Mn(III) ion. The complexes have been characterized by elemental analysis, IR spectroscopy, TGA and single crystal X-ray diffraction studies. Structural studies reveal that the complexes 1 and 2 adopt an octahedral and a square pyramidal geometry, respectively. The antibacterial activity of the complexes has been tested against Gram(+) and Gram(-) bacteria.

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We describe here the synthesis of two new mononuclear complexes of manganese(III) containing a Schiff base ligand (HL) obtained *in situ* from furfurylamine and salicylaldehyde. The complexes have been characterized by microanalytical, spectroscopic, thermogravimetric and single crystal X-ray structural studies and tested *in vitro* to assess their antibacterial activities against some common reference bacteria and compared with commercial antibiotic biodiscs of Nalidixic acid and Gattifloxacin.



2. Experimental

2.1. Physical measurements

Elemental analyses were carried out using a Perkin–Elmer 2400 elemental analyzer. The infrared spectra were recorded on a Perkin–Elmer FT-IR spectrophotometer with KBr discs (4000– 300 cm⁻¹). Molar conductances of the complexes in dry methanol were measured using a direct reading conductivity meter of Systronics (Type 304). Room temperature solid phase magnetic susceptibilities were measured at 298 K with a PAR 155 vibrating sample magnetometer with Hg[Co(NCS)₄] as the calibrant.



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Diamagnetic corrections were taken from standard sources. Thermogravimetric analyses were carried out using a Netzsch STA409PC instrument from 30 °C to 700 °C in an atmosphere of dinitrogen at the heating rate of 10 °C min⁻¹.

2.2. Materials

Reagent grade salicylaldehyde, furfurylamine, manganese(II) perchlorate hexahydrate and sodium azide were purchased from reputed manufacturers and used as received. All other chemicals and solvents were of analytical grade.

Caution! Compounds containing perchlorate and azide are potentially explosive. Therefore, only a small amount of the materials should be used at a time and handled with proper care. However, no problems were encountered during our studies including the thermogravimetric analyses of compounds **1** and **2** within the experimental range of temperature in inert atmosphere.

2.3. Synthesis of compounds

2.3.1. Synthesis of $[MnL_2(LH)_2]ClO_4$ (1)

A methanolic solution of manganese(II) perchlorate hexahydrate (0.36 g, 1.0 mmol) was added to a mixture of salicylaldehyde (0.24 g, 2.0 mmol) and furfurylamine (0.19 g, 2.0 mmol) in methanol with constant stirring and the total volume was made up to 50 ml (solution-1) by adding the same solvent. Stirring was continued for half an hour and the solution was left for slow evaporation at room temperature in a beaker open to the atmosphere. After a week, dark brown crystals of compound **1** appeared. The crystals were collected by filtration, washed with methanol and finally dried (0.26 g, 55%). *Anal.* Calc. for C₄₈H₄₂ClMnN₄O₁₂: C, 60.22; H, 4.42; N, 5.85; Mn, 5.74. Found: C, 59.98; H, 4.38; N, 5.82; Mn, 5.69%. FTIR (KBr, cm⁻¹): 1644(s), 1623(s), 1599(m), 1530(w), 1485(m), 1448(m), 1419(w), 1320(m), 1290(s), 1151(m), 1094(s), 1013(w), 898(w), 835(w), 758(m), 623(w). A_M (MeOH, Ω^{-1} cm² mol⁻¹): 130. μ_{eff} (RT, BM): 4.7.

2.3.2. Synthesis of [MnL₂(N₃)]·0.5CH₃OH (**2**)

To a 50 ml of solution-1 in methanol (prepared using the same mole ratio of reactants as in Section 2.3.1. above), 0.065 g (1 mmol) of sodium azide dissolved in minimum volume of water was added. Stirring was continued for further half an hour. Pure dark brown crystals of **2** were collected after two days, washed and dried following the same procedure described in Section 2.3.1. (0.37 g, 72%). *Anal.* Calc. for C_{24.5}H₂₂MnN₅O_{4.5}: C, 57.31; H, 4.32; N, 13.64; Mn, 10.70. Found: C, 57.25; H, 4.27; N, 13.62; Mn, 10.68%. FTIR (KBr, cm⁻¹): 2926(s), 2853(s), 2038(s), 1613(s), 1546(w), 1301(m), 1142(w), 904(w), 738(m). $\Lambda_{\rm M}$ (MeOH, Ω^{-1} cm² mol⁻¹): 5. $\mu_{\rm eff}$ (RT, BM): 4.8.

2.4. Crystal structure determination and refinement

Single crystal X-ray studies of **1** and **2** were carried out in a Nonius Kappa CCD diffractometer using the related analysis software [13]. No absorption corrections were made to the data sets. All structures were solved by direct methods using the sire97 program [14] combined with Fourier difference syntheses and refined against *F* using reflections with $[I/\sigma(I) > 3]$ utilizing the CRYSTALS program [15]. All atomic displacement parameters for non-hydrogen atoms have been refined with anisotropic terms. The hydrogen atoms were theoretically located on the basis of the conformation of the supporting atom or found by Fourier difference. Complex **2** crystallizes apparently with one methanol molecule which shows positional disorder with 50% occupancies. Crystallographic data and refinement details for the compounds are summarized in Table 1.

Table 1

Crystallographic data and structure refinement for 1 and 2.

Parameters	1	2
Formula Formula weight (g mol ⁻¹)	C ₄₈ H ₄₂ Cl ₁ Mn ₁ N ₄ O ₁₂ 957.3	C _{24.5} H ₂₂ Mn ₁ N ₅ O _{4.5} 513.4
Crystal system	triclinic	monoclinic
Space group	PĨ	$P2_1/a$
a (Å)	9.3469 (2)	9.5848 (4)
b (Å)	9.6091 (3)	24.1361 (6)
<i>c</i> (Å)	24.6111 (7)	11.1908 (5)
α (°)	83.409 (2)	90
β (°)	89.692 (2)	113.433 (2)
γ (°)	78.914 (2)	90
$V(Å^3)$	2154.6 (1)	2375.4 (2)
Ζ	2	4
T (K)	150	293
λ (Mo Kα) (Å)	0.71073	0.71073
Number of unique reflections (R_{int})	10218 (0.079)	5196 (0.052)
D (g cm ⁻³)	1.475	1.436
μ (mm ⁻¹)	0.44	0.599
Number of reflections used	4534	3457
Number of parameters refined	598	325
$R(F), I > 3\sigma(F_o)$	0.045	0.0584
$R_{\rm w}$ (F), $I > 3\sigma(F_{\rm o})$	0.0465	0.0679
S	1.11	1.12
$\Delta ho_{ m max}$ (e Å ⁻³)	0.37	0.40
$\Delta ho_{ m min}$ (e Å ⁻³)	-0.38	-0.45

2.5. Antimicrobial activity – minimum inhibitory concentration

Complexes 1, 2 and two reference commercial antibiotics viz. Nalidixic acid and Gattifloxacin (purchased in powder form from Span Diagnostic Limited, Surat, India) were tested in vitro to assess their growth inhibitory activity against two Gram positive bacteria, viz. Staphylococcus aureus MTCC 2940 and Bacillus subtilis MTCC 441 and two gram negative bacteria viz. Pseudomonas aeruginosa MTCC 2453 and Klebsiella pneumoniae MTCC 530 by Kirby Bauer method with necessary modifications [16]. The antibacterial activity of furfurylamine, salicylaldehyde and the Schiff base HL prepared [9] from furfurylamine and salicylaldehyde were also evaluated during the same experiment. The bacterial strains grown on nutrient agar at 37 °C for 18 h were suspended in a saline solution (0.85% NaCl) and adjusted to a turbidity of 0.5 MacFarland standards (10⁸ CFU ml⁻¹). The suspension was used to inoculate 90 mm diameter sterile Petri plates in which the test organisms were grown on nutrient agar medium. All the compounds including the commercial antibiotics were dissolved in dimethylsulphoxide (1 mg ml^{-1}) and soaked in filter paper discs of 5 mm diameter and 1 mm thickness for 12 hours at <40 °C. The discs were placed on the previously seeded plates and incubated at 30 °C for B. subtilis and at 37 °C for other bacteria. Antibacterial activities were evaluated by measuring inhibition zone diameters (IZD). The experiments were repeated thrice along with a control set using dimethylsulphoxide (dmso). The minimum inhibitory concentrations (MIC) were also determined by serial dilution technique (from 1 mg ml⁻¹ to 5 mg ml⁻¹ concentrations) as followed by the National Committee for Clinical Laboratory Standards [17]. MIC was the lowest concentration of a compound extracted in dmso that exhibited no visual growth of the organisms in the culture tubes.

3. Results and discussion

3.1. Synthesis

The mononuclear Mn(III) complexes **1** and **2** have been prepared by adding salicylaldehyde and furfurylamine directly to the reaction mixture and the bidentate Schiff base HL was formed *in situ* in both the cases. Thus the pre-condensation to form the Schiff base



Fig. 1. Molecular structure of 1 with atom labeling. (a) and (b) represent the two independent mononuclear units of Mn2 and Mn1, respectively.

ligand has been avoided and no HL was needed to be isolated. Addition of azide to the reaction mixture of **1** yielded the mixed ligand azido complex 2 with a completely different geometry. The complexes have been characterized by elemental analysis, IR spectroscopy, electrical conductivity and magnetic susceptibility measurements as well as by thermogravimetric and single crystal X- ray structural analysis. The results are consistent with the mononuclear formulae. In methanol solution, 1 behaves as a 1:1 electrolyte while **2** behaves as a non-electrolyte as is evident from their $\Lambda_{\rm M}$ values (ca. 130 and 5 Ω^{-1} cm² mol⁻¹, respectively). Room temperature magnetic susceptibility measurements indicate that the complexes have magnetic moments (4.7-4.8 B.M.) close to the spin-only value of Mn(III) as expected from discrete and magnetically non-coupled mononuclear d⁴ ions. Mn(II) has undergone aerial oxidation to Mn(III) in both the reactions as was observed earlier by our research group and others [18,19] in similar syntheses. This is probably due to the formation of more stable complexes by harder Mn^{III} ion with ligands containing harder donor atoms. As usual, the furyl group is not coordinated to Mn(III) ion and our attempt to synthesize complexes with tridentate Schiff base of furfurylamine remained unsuccessful.

3.2. FTIR spectra

The infrared spectra of the compounds **1** and **2** are similar in some respects as expected. They show strong bands at 1613–1623 cm⁻¹ which are assignable to the C=N stretching vibrations [ν_{CN}] indicating the formation of the Schiff base products. However, the spectrum of compound **2** exhibits a very strong and sharp absorption band at 2038 cm⁻¹ corresponding to the asymmetric stretching vibrations of the terminal azide ion [$\nu_{as}(N_3)$]. The asymmetric single and sharp band expected for ClO₄⁻ is also observed at 1094 cm⁻¹ in compound **1**. All other characteristic vibrations including the phenolic C–O stretching of the metal-bound Schiff bases are located in the range 600–1600 cm⁻¹. Thus the IR spectra of the compounds are in good agreement [20] with the respective structural features of Mn(III) Schiff base complexes.

3.3. Thermogravimetric analysis

The thermograms of TGA reveal that the compound **1** is stable up to 160 °C and first stage of weight loss (\sim 21%) occurs between

 $250^{\circ}-360$ °C, corresponding to the removal of one ligand followed by gradual weight loss up to 700°C. In compound **2**, the first stage of weight loss (~3.12%) occurs between 60 °C and 70 °C corresponding to the loss of the methanol molecule present in the crys-

Table 2

Selected bond lengths (Å) and bond angles (°) of 1.

Mn1–N126 Mn2–N206 Mn1–O114	2.048(3) 2.068(3) 2.150(3)	Mn1-0134 Mn2-0214 Mn2-0234	1.882(3) 1.875(2) 2.151(3)
O114-Mn1-N126 N126-Mn1-O134 N126-Mn1-O114 O114-Mn1-N126 O134-Mn1-N126 O134-Mn1-O134 O134-Mn1-O134 N206-Mn2-O214 O234-Mn2-O234 N206-Mn2-O234	89.5(1) 89.9(1) 90.5(1) 90.5(1) 90.1(1) 95.1(1) 180 90.0(1) 180 89.4(1)	0114-Mn1-0134 0114-Mn1-0114 0134-Mn1-0114 N126-Mn1-N126 0114-Mn1-N126 N126-Mn1-0134 0114-Mn2-0214 N206-Mn2-0234 0214-Mn2-0234	84.9(1) 180 95.1(1) 180 89.5(1) 90.1(1) 84.9(1) 180 90.6(1) 84.2(1)



Fig. 2. Molecular structure of 2 with atom labeling.

Table 3	
Selected bond lengths (Å) and bond angles (°) of 2	2.

- - - -

Mn1-01 Mn1-N29	1.861(2) 2.053(2)	Mn1-N9 Mn1-N41	2.049(2) 2.100(3)	Mn1-021	1.850(2)
01-Mn1-N9 01-Mn1-N29 01-Mn1-N41 N29-Mn1-N41	89.9(1) 89.3(1) 94.7(1) 95.9(1)	01-Mn1-021 N9-Mn1-N29 N9-Mn1-N41	168.4(1) 162.0(1) 102.1(1)	N9-Mn1-O21 O21- Mn1-N29 O21-Mn1-N41	87.9(1) 89.2(1) 96.9(1)

tal lattice. This is followed by a steep weight loss up to 320 °C, after which it again decreases gradually up to the maximum temperature of study.

3.4. Crystal structure of the compounds

3.4.1. Crystal structure of 1

The molecular structure of **1** is shown in Fig. 1 and a few selected bond distances and angles are listed in Table 2. The X-ray structure reveals that the compound **1** is built up of two independent mononuclear units (Fig. 1a and b with Mn2 and Mn1, respectively as the central metal ions) in the solid state. These two molecules are structurally identical except for a slight variation in bond angles and lengths.

In both the units, the Mn(III) ion adopts a distorted octahedral geometry in which the equatorial plane is formed by the coordination of the bidentate Schiff base ligand i.e., two imino nitrogen (N126 for Mn1 and N206 for Mn2) and two phenolic oxygen atoms (O134 for Mn1 and O214 for Mn2). The apical positions are occupied by two phenolic oxygen atoms (O114 and O234 for Mn1 and Mn2, respectively) of N-protonated Schiff base molecules, each acting as a monodentate ligand. All the axial bonds are slightly longer than the equatorial ones as a result of Jahn-Teller distortion occurred in octahedral Mn(III) complexes which is evident from the average Mn–O, –N and Mn–O bond lengths of 1.968 and 2.150 Å, respectively in the equatorial and the apical positions. The electroneutrality is maintained by the presence of the perchlorate anions.

3.4.2. Crystal structure of 2

The molecular structure of **2** is displayed in Fig. 2. and a few selected bond dimensions are collected in Table 3. The coordination geometry around the Mn(III) ion is a distorted square pyramid. The two imino nitrogen (N9, N29) and two phenolic O-atoms (O1, O21) are located in the trans positions to form the square base.

As expected, the Mn–N bond distances are greater than the Mn–O bonds in the basal plane. The apical position is occupied by one N-atom of the azide ion with a Mn(1)-N(41) bond length of 2.100(3) Å. Distortion from an ideal square pyramidal geometry

Table 4

Antibacterial activities of **1**, **2** and related compounds compared to control (dmso) and standard antibiotics (concentration = 1 mg ml⁻¹ and n = 3 trials/compound).

Compounds	Inhibition zone diameter (mean ± standard error)			
	Gram positive bacteria		Gram negative bacteria	
	S. aureus	B. subtilis	P. aeruginosa	K. pneumoniae
1 2 Furfurylamine Salicylaldehyde HL ^a dmso	13.66 ± 0.33	12.66 ± 0.88	15 ± 1.00	13.00±1.00 14.66 ± 0.88
Nalidixic acid Gattifloxacin	26.66 ± 1.33 31.33 ± 0.76	28.33 ± 1.66 32.66 ± 1.00	22.66 ± 1.20 29.33 ± 1.20	29.33 ± 0.88 30.66 ± 1.00

^a Schiff base of furfurylamine and salicylaldehyde [9].

Table 5

Minimum inhibitory concentrations (mg ml^{-1}) of **1**, **2** and related compounds.

Compounds	S. aureus	B. subtilis	P. aeruginosa	K. pneumoniae
1	<2	>5	<1.5	<2
2	>5	<2	>5	<2
Furfurylamine	>5	>5	>5	>5
Salicylaldehyde	>5	>5	>5	>5
HL	>5	>5	>5	>5
Nalidixic acid	<1	<1	<1	<1
Gattifloxacin	<1	<1	<1	<1

is due to the asymmetric nature of the bidentate Schiff base ligand. However, the distortion is much less as evidenced from the cisoid and transoid angles, all being closer to 90° and 180°, respectively. The observed bond angles indicate that the Mn(III) ion is slightly above the square base.

3.5. Antibacterial activity

The antibacterial activity of all of the tested compounds and their MIC values are presented in Tables 4 and 5, respectively. The results of the antibacterial screening indicate that although furfurylamine, salicylaldehyde and the corresponding Schiff base lack any bacterial growth retardation activity, the complexes 1 and 2 exhibit antibacterial activity to some extent. However, both the complexes possess rather higher MIC and lower IZD values. The IZD data shows that compound **1** is active against *S. aureus* but is inactive against B. subtilis whether the reverse is seen in compound **2**. Again, complex **1** shows activity against both the Gram(-) bacteria, but 2 is active only against K. pneumoniae. Thus the antibacterial study reveals that K. pneumoniae is susceptible against both the compounds and the MIC values of the two are of the same order. However, the antibacterial activities of both 1 and 2 are much lower than the tested commercial antibiotics at similar concentrations.

4. Conclusion

Two new mononuclear Mn(III) complexes could be synthesized with a less familiar asymmetric bidentate Schiff base ligand obtained *in situ* from the respective aldehyde and amine. Both the complexes possess rather unusual ligand coordination and geometry. The results of antibacterial screening of the compounds indicate mild bactericidal activities. In addition to the synthetic and structural investigations, this study helps to evaluate the potentiality and effectiveness of newer Schiff base complexes of Mn(III) to use as antibacterial agents.

Supplementary data

CCDC 714205 and 714206 contains the supplementary crystallographic data for **1** and **2**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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