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Research Article

SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL ACTIVITY OF Ni-Mn TARTARATE-MIXED METAL COMPLEXES

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Tartarates ligand based some novel mixed transition metal complexes of type $[M_xM^I_{1-x}(C_4H_4O_6)].XH_2O$ have been synthesized. Further, these complexes were characterized by elemental analysis, FT-IR, TGA and XRD. Analytical data revealed that all the complexes exhibited 1:1 (metal: ligand) ratio. IR data shows that the ligand co-ordinates with the metal ions in a bidentate manner through two oxygen atoms. The thermal analysis showed the degradation pattern of the complexes.

The metal complexes were also screened for in-vitro antimicrobial properties against *Bacillus Subfilis, Staphylococcus Aureus, Escherichia Coli* and *Proteus vulgaris* to assess their antimicrobial effect.

INTRODUCTION

In recent years, there has been renewed interest in the development and study of mixed ligand transition metal complexes [1-11]. These complexes have received an attention due to their applications in diverse field [12-15]. Some metal ligand complexes are found to be used as catalysts in various reactions such as oxidative cleavage, decomposition of H_2O_2 etc. [16,17]. Binary and ternary metal complexes have been shown wide range of biological activity. Metal complexes of N- or O- donor ligands have attracted considerable attention because of their higher antifungal and antibacterial activities than those of parent ligands [18-31].

The present work describes the synthesis and characterization of Ni-Mn Tartarate complexes $[Ni_xMn_{1-x}(C_4H_4O_6)]\ H_2O$ of six different proportions. These six different proportions of Ni-Mn Tartarate complexes are synthesized and characterized on the basis of elemental analysis, spectral and thermal studies. Probable structure has been suggested for Ni-Mn Tartarate complexes on the basis of elemental analysis and various physico-chemical studies.

These Ni-Mn tartarate complexes have been further screened for antimicrobial activity against micro-organisms like *Escherichia Coli, Staphylococcus Aureaus, Bacillus Subtills, Proteus vulgaris* etc.

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MATERIALS AND METHODS

Materials

All chemicals used in the synthesis were of either Analar BDH grade or E. Merck grade. Quantities of reactants were calculated according to the stoichimetry in the final product.

Synthesis of Nickel–Manganese tartarate complexes $[Ni_xMn_{(1-x)}(C_4H_4O_6)].H_2O$

The Nickel-Manganese taratrates with six different compositions $[M_xM1_{(1-x)}(C_4H_4O_6)]\ .H_2O$ where M and M1 are Ni and Mn, and x=0.2, 0.4, 0.6, 0.8 and 1.0 have been successfully prepared by co-precipitation method from high purity $NiSO_4.6H_2O$ and $MnSO_4.H_2O$ in distilled water. The mixture of metal sulphate solution was prepared with respect to molar ratio of $NiSO_4$ and $MnSO_4$ and placed in a beaker. pH of the medium was adjusted to low enough (pH<6); so that the hydroxide does not precipitate. The solution was stirred vigorously and sodium tartarate (10%) solution was added with stirring till a permanent precipitate occurred. Further, an acetone was added in equal amount of metal salts to ensure a high yield of product. The resultant precipitate was light bluish-green. The solution was filtered after stirring for 30 minutes. The filtrate was checked for free Mn^{+2} and Ni^{+2} whose absence ensured completion of co-precipitation process. The residue was washed with cold distilled water and then with acetone to speed up drying. The solid was dried at ambient temperature.

Such types of six samples of Ni – Mn tartarate complexes of different proportions were synthesized (Table-1).

Table 1 Various proportion Ni:Mn composites with their Molecular Weights

	B_1	B_2	B_3	B_4	B_5	B_6
Complex	$Ni_{(0.6)}.Mn_{(0.4)}(C_4H_4O_6).H_2O$	$Ni_{(0.6)}.Mn_{(0.4)}(C_4H_4O_6).H_2O$	$Ni_{(0.4)}.Mn_{(0.6)}(C_4H_4O_6).H_2O$	$Ni_{(0.2)}.Mn_{(0.8)}(C_4H_4O_6).H_2O$	$Ni_{(1.0)}.Mn_{(0.0)}(C_4H_4O_6).H_2O$	$Ni_{(0.0)}.Mn_{(1.0)}(C_4H_4O_6).H_2O$
Mol. Weight	223.972	223.214	222.456	221.698	224.69	220.94

RESULTS AND DISCUSSION

Characterization of Ni–Mn Tartarate Complexes

Elemental Analysis

All the six complexes synthesized were subjected to elemental analysis. All the six Ni-Mn Tartarate precursors are found to be in good agreement with the calculated values. (Table-2)

Table 2 Elemental Analysis - Ni-Mn Tartarate

		C %		H %		Ni %		Mn %	
Sr. No.	**Complex**	**Calcd**	**Found**	**Calcd**	**Found**	**Calcd**	**Found**	**Calcd**	**Found**
1	B_1 $Ni_{(0.8)}.Mn_{(0.2)}(C_4H_4O_6).H_2O$	21.43	22.54	2.688	2.96	20.98	18.54	4.91	4.42
2	B_2 $Ni_{(0.6)}.Mn_{(0.4)}(C_4H_4O_6).H_2O$	21.50	18.60	2.688	2.386	15.786	13.16	9.845	8.64
3	B_3 $Ni_{(0.4)}.Mn_{(0.6)}(C_4H_4O_6).H_2O$	21.577	19.17	2.697	3.03	10.556	12.13	14.818	13.21
4	B_4 $Ni_{(0.2)}.Mn_{(0.8)}(C_4H_4O_6).H_2O$	21.651	20.42	2.706	3.11	5.298	6.05	19.825	17.94
5	B_5 $Ni_{(1.0)}.Mn_{(0.0)}(C_4H_4O_6).H_2O$	21.363	21.31	2.67	2.85	26.138	25.942	0	0
6	B_6 $Ni_{(0.0)}.Mn_{(1.0)}(C_4H_4O_6).H_2O$	21.725	19.85	2.716	2.92	0	0	24.866	22.67

IR Spectra

All the six complexes (B_1 to B_6) were scanned on Nicollet NEXUS 7000C spectrometer. The Infrared spectra of these complexes showed frequencies corresponding to hydroxyl group, metal-oxygen group, carbon–hydrogen etc. The bidentate linkage of Tartarate group with metal was confirmed on the basis of difference between antisymmetric and symmetric stretching frequencies (Table-3).

Particle Size

The observed particle size of all six Tartarate complexes (B_1 to B_6) was estimated using an expression $D = [\frac{0.89\lambda}{\beta.cos\theta}]$ in angstrom unit and is presented in (Table-5). The observed particle size of tartarate complexes found to be in between 283 to 830 A^0.

Table 3 Infra Red Spectral Analysis

	B_1	B_2	B_3	B_4	B_5	B_6	
Sr. No.	**$Ni_{(0.8)}.Mn_{(0.2)}(C_4H_4O_6).H_2O$**	**$Ni_{(0.6)}.Mn_{(0.4)}(C_4H_4O_6).H_2O$**	**$Ni_{(0.4)}.Mn_{(0.6)}(C_4H_4O_6).H_2O$**	**$Ni_{(0.2)}.Mn_{(0.8)}(C_4H_4O_6).H_2O$**	**$Ni_{(1.0)}.Mn_{(0.0)}(C_4H_4O_6).H_2O$**	**$Ni_{(0.0)}.Mn_{(1.0)}(C_4H_4O_6).H_2O$**	**Remarks**
1	3126	3340, 3117	3146	3160	3169	3354	ν(O-H)- stretch
2.	2354	2356	2337	2362	-	-	ν (O-H) carboxylic acid
3.	1589	1594	1594	1575	1607	1587	ν(C-O)
4.		1458		1408			ν(C=O)
5.	1386, 1295	1377, 1297	1385, 1294	1297	1387	1396	ν
6.	1168	1123	1085	1117	1115	1126	ν C-O (alcohol)
7.	1084	1043	--	1037	--	1051	ν C-O(sym)
8.	937	938	--	944	--	--	--
9.	712	721	713	709	631	637	νsym(C-C)

XRD Analysis

The X-ray powder diffraction patterns of Tartarate complexes of six different proportions showed a broad as well as sharp lines. This suggests that Tartarate complexes are polycrystalline in nature. The d-spacing values calculated for respective samples and presented in (Table-4).

Table 4 Observed d-Spacing values

B_1	B_2	B_3	B_4	B_5	B_6
$Ni_{(0.8)}.Mn_{(0.2)}(C_4H_4O_6).H_2O$	$Ni_{(0.6)}.Mn_{(0.4)}(C_4H_4O_6).H_2O$	$Ni_{(0.4)}.Mn_{(0.6)}(C_4H_4O_6).H_2O$	$Ni_{(0.2)}.Mn_{(0.8)}(C_4H_4O_6).H_2O$	$Ni_{(1.0)}.Mn_{(0.0)}(C_4H_4O_6).H_2O$	$Ni_{(0.0)}.Mn_{(1.0)}(C_4H_4O_6).H_2O$
3.6733	4.7815	4.5025	4.6043	4.3718	4.1660
3.2056	4.5621	4.076	4.2301	0.7719	3.8650
2.5293	4.1448	3.9162	3.8423	4.093	3.4651
2.1436	3.9311	3.7275	3.3543	3.5046	2.9996
2.0679	3.6178	3.5189	2.6492	0.8966	2.5711
3.9171	3.5189	3.2056	2.7462	3.4824	2.4610
3.4466	3.2056	2.9967	3.0622	2.8551	2.3856
4.5211	2.9793	2.5297	2.2350	0.8291	2.0668
2.2543	2.5169	2.1436	1.9017	2.505	1.9029
2.0048	2.1613	2.0679	1.8622	1.9282	1.7810
1.9362	2.0679	2.0052	1.8002	1.3025	1.7459
1.8182	1.9902	2.2543	1.7481	1.9874	1.6922
	2.2543	2.1496			1.5948
	2.3406	2.2099			1.4507
	2.4545	1.7234			1.2821

Table 5 Observed particle size

B_1	B_2	B_3	B_4	B_5	B_6
411.9 A°	282.6 A°	321.5 A°	830 A^0	287.153 A°	337.54 A^0

Thermogravimetric Analysis

The Thermogravimetric analysis of all the six Ni –Mn tartarates complexes has been done.

All samples of Tartarate complexes (B_1 to B_6) showed loss of water molecule at about 90 to 105^{0}C. The % loss for one water molecule is well matched with the theoretical loss. The oxidative decomposition of these ligands is observed between 150 to 350^{0}c, which is corresponding to loss of CO, CO_2, C_2H_4 etc. (Scheme-1). Thermal study suggests the following probable reactions.

Scheme-1: Thermogravimetric Analysis

1. $Ni.Mn(C_4H_4O_6).H_2O \rightarrow Ni.Mn(C_4H_4O_6) + H_2O\uparrow$
2. $Ni.Mn(C_4H_4O_6) \rightarrow Ni.MnO_2 + 2CO_2\uparrow + C_2H_4\uparrow$

On the basis of CHNS analysis, IR study, TGA observations, AAS study and XRD analysis, the probable structure of the complex Ni-Mn Tartarate is shown as in (Figure-1.)

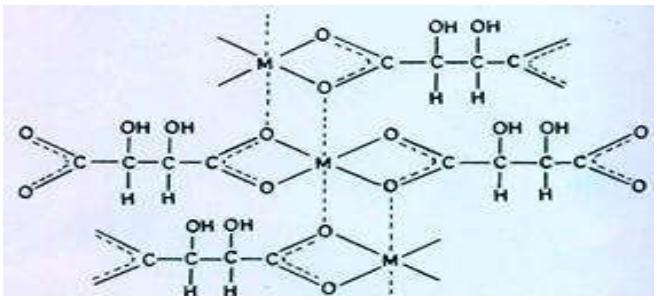

Figure 1 Probable structure of the complex Ni-Mn Tartarate

Antimicrobial Activity of Synthesized Ni-Mn Tartarate Complexes $[Ni_x.Mn_{(1-x)}(C_4H_4O_6)].H_2O$

All the synthesized mixed metal complexes i.e. Ni–Mn Tartarates (sample B_1 to B_6) were screened for antibacterial as well as antifungal activity. Anti-microbial activity against four different organisms such as, *Bacillus Subtilis, Staphylococcus Aureus, Escherichia Coli* and *Proteus Vulgaris* were studied.

MATERIALS AND METHODS

1. Ni -Mn tartarate complexes of six different proportions, (Set A: samples B_1 to B_6 and Set B: samples B_1 to B_6.)
2. Diluents: Sterile distilled water [10 ml in each 7 tubes.
3. Nutrient agar medium [Cruickshank *et al* 1975] plates-18 in numbers.
4. Fresh 24 Hrs old nutrient broth cultures of test bacterial organism. *a) Bacillus Subtilis– Gram positive in nature. b) Staphylococcus aureus- Gram positive in nature. c) Escherichia Coli- Gram negative in nature. d) Proteus Vulgaris- Gram negative in nature.*
5. Well borer and glass spreader.
6. Sterile1 ml. capacity glass pipette/micropipette.

Procedure

Using sterile distilled water diluent 1% solution of each chemical [Ni-Mn tartarate samples, B_1 to B_6] was prepared. For every chemical solution [Ni-Mn tartarate samples, B_1 to B_6], three nutrient agar plates were used and labeled for above three bacterial cultures. In total 12 sets of plates [3 plates in each set] were prepared. In each set of plates 0.5 ml. of above bacterial cultures were spread, inoculated and incubated at 37^0 C for 30 minutes to adsorb the culture on medium surface. Using well borer, a well was bored at center of medium in each plate, aseptically. 0.1 ml. of each chemical solution [sample B_1 to B_2] was poured aseptically in each respective well and incubated for diffusion at 40^0 C for 1 hrs. All the plates were incubated at 37^{0}C for 48 hrs and results were recorded.

Ni-Mn Tartarate composites with all six compositions (B_1 to B_6) possess antimicrobial activity against gram positive as well as gram negative bacteria. Overall activity with all composites (B_1 to B_6) is more effective against *Staphylococcus Aureus* and gave pronounced antimicrobial activity.

Complex B_6, have shown highest biological activity (3.1cm) against *Proteus Vulgaris* organism, where as complex B_5 have shown lowest biological activity against *Staphylococcus Aureus* as compare to statndard Gentamycin (Table-6).

Table 6 Biological Activity of Ni-Mn Tartarates against different organism

Samples of Ni-Mn tartarate composites	E. Coli (mm)	Bascillus S. (mm)	Proteous V. (mm)	S. Aureus (mm)
B_1 $Ni_{(0.8)}.Mn_{(0.2)}(C_4H_4O_6).H_2O$	23	22	16	24
B_2 $Ni_{(0.6)}.Mn_{(0.4)}(C_4H_4O_6).H_2O$	22	21	20	25
B_3 $Ni_{(0.4)}.Mn_{(0.6)}(C_4H_4O_6).H_2O$	21	24	19	25
B_4 $Ni_{(0.2)}.Mn_{(0.8)}(C_4H_4O_6).H_2O$	15	20	21	26
B_5 $Ni_{(1.0)}.Mn_{(0.0)}(C_4H_4O_6).H_2O$	12	14	14	10
B_6 $Ni_{(0.0)}.Mn_{(1.0)}(C_4H_4O_6).H_2O$	20	29	31	25
Standard Gentamycin	20	25	22	25

CONCLUSION

Herein we report the synthesis of Tartarates ligand based some novel mixed transition metal complexes of type $[M_xM^I_{1-x}(C_4H_4O_6)].XH_2O$. The synthesized complexes on screening for antimicrobial activities against *Bacillus Subfilis, Staphylococcus Aureus, Escherichia Coli* and *Proteus vulgaris* showed excellent to moderate results.

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