



Synthesis, Characterization and Evaluation of Biological Activity of Mn-Fe tartrate Complexes.

S.S. Pawar¹, N. S. Dhavale², C. S. Patil³,
S.M. Vhankate⁴, R.P. Pawar⁵, V. B. Tadke⁶

^{1,2,4,6}Department of Chemistry, Fergusson College (Autonomous), Pune, Maharashtra, India.

^{3,5}Department of Chemistry, Deogiri College, Aurangabad, Maharashtra, India.

ABSTRACT

The study of metal tartrates is been known. With reference to mixed metal tartrates as their synthesis, characterization and biological activity of various organisms is studied.

The synthesis of pure Manganese tartrate, Ferrous tartrate and its mixed metal complex that is Manganese-Ferrous tartrate monohydrate $[Mn_{(0.5)}Fe_{(0.5)}(C_4H_4O_6)_3H_2O]$ is done by precipitation method stoichiometrically. The composites synthesized are characterized by different techniques such as AAS, IR study, X-Ray diffraction patterns, TGA and Elemental analysis. It has been observed that the composites formed posses bidentate ligand and have polycrystalline nature.

The complexes formed are tested against biological organisms like *Escherichia coli*, *Bacillus subtilis*, *Staphylococcus aureus*, *C.albieans*, *A. niger* and *P. chrysogenum*.

Keywords: Manganese-Ferrous tartrates, bidentate, *C. albieans*.

I. INTRODUCTION

Schiff base ligands are of significant interest not only for their pharmacological properties as antibacterial, antifungal, and anticancer agents [1, 2], but also for their capacity for chemical recognition of anions and metals of biochemical and environmental importance [3, 4, 5, 6]. Schiff base complexes of transition metals are of particular interest to inorganic chemists because their structural, spectral, and chemical properties are often strongly dependent on the nature of the ligand structure [7, 8, 9].

This work deals with the synthesis of manganese tartrate, ferrous tartrate and its mixed metal tartrates.

The coordination chemistry of transition metal complexes with mixed ligands are of current interest because they can provide new materials with useful properties such as magnetic exchange[10, 11], electrical conductivity [12], photoluminescence [13], nonlinear optical property [14], and antimicrobial activity [15].

The synthesized composites (Sample: S1, S2 and S3) are characterized by TGA, IR study, X-Ray patterns, AAS and CHNS analysis.

The biological importance of metal complexes is that they are sometimes more effective than the free ligands [14,15]. Mixed metal complexes containing nitrogen and oxygen donors are important owing to their antifungal, antibacterial, and anticancer activities [1,2].



The biological study of Mn-Fe tartrates is performed against *Escherichia coli*, *Bacillus subtilis*, *Staphylococcus aureus*, *C.albieans*, *A. niger* and *P. chrysogenum*.

II. METHODOLOGY

2.1 Materials:

All the chemicals and metal salts ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and $\text{MnSO}_4 \cdot \text{H}_2\text{O}$) are of analytical grade, and were used as received. Various solutions of metal salts and complexes are prepared using the double distilled water.

2.2 Synthesis of composites:

All the complexes were synthesized using co-precipitation method. The two metals salts aqueous solutions i.e. $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ were mixed in a fixed molar ratio. Dilute H_2SO_4 were added in the solution to maintain pH at slightly acidic condition. In this mixture 10% solution of tartrate in water solution were added slowly with constant stirring to get complete precipitation. Solutions stirred further for some time by adding equal amount of acetone in it. Formation of precipitate observed on cooling the above solution. The complex was, filtered washed with acetone and dried under normal atmospheric conditions and then stored in desiccators. The elemental analysis for percentage of metals, carbon and hydrogen were carried out. The results show good agreement with the accepted values. The Infrared absorption spectra of the ligand and metal complexes were recorded on a Shimadzu spectrophotometer from 4000 to 400 cm^{-1} using KBr pellets. The Powdered diffraction patterns of metal complexes were recorded using Regaku Mini flex diffractometer $\text{CuK}\alpha$ ($\lambda=1.5405 \text{ \AA}$).

The thermoanalytical measurements were obtained with Perkin-Elmer (Delta series-TGA7) instrument in temperature range 30 to 600 $^{\circ}\text{C}$. Thermo gravimetric analysis data conforms the presence of water of crystallization in newly synthesized complexes. XRD pattern suggest polycrystalline nature of the complexes. The collective results obtained through all above instrumental analysis indicate the formation of binuclear metal complexes with adequate degree of purity.

Above synthesized complexes were for studied for their antibacterial action against some gram positive and gram negative bacteria. The obtained results were summarized and discussed below.

III. RESULT AND DISCUSSION

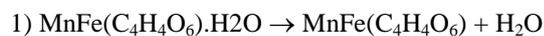
The elemental analysis made in weight % of Mn-Fe Tartrate complexes synthesized (S_1 to S_3) in good agreement with calculated values (Table 1).

The X-ray powder diffraction patterns of these composites showed broad as well as certain sharp lines (Fig.2). This indicates that these complexes are polycrystalline in nature. The d-spacing values calculated for respective samples are given in (Table3).

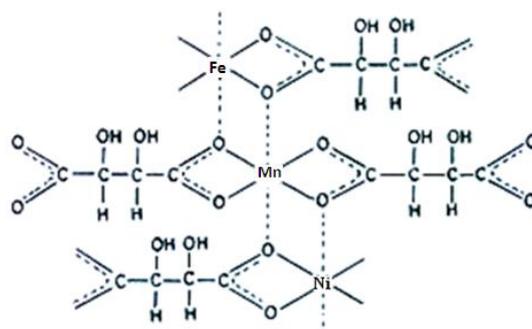
Infrared spectra of these precursors (S_1 to S_3) showed frequencies corresponding to hydroxyl group, metal-oxygen group, carbon-hydrogen group, carbon-oxygen group etc. The bidentate linkage of tartrate group with metal was confirmed on the basis of difference between antisymmetric and symmetric stretching frequencies (Table 2).

The TGA describe the loss of water molecule at about 90 to 100°C. The % loss for one water molecule is well matched with the theoretical loss. The oxidative decomposition of these tartrate ligands is observed in between 180°C to 350°C, which is corresponding to loss of CO, CO₂, C₂H₄ moieties etc.

Thermal study suggest the following probable reactions,



On the basis of several observations like AAS, CHNS analysis, IR study, TGA, XRD etc., the probable structure of complex Mn-Fe tartrate is shown below,



It has been observed that the Mn-Fe tartrate complexes have shown significant antimicrobial activity against selective cultures. These compositions possess antimicrobial activity against gram positive as well as gram negative bacteria (Fig.3).

Complex S₂ (Fe(C₄H₄O₆)).H₂O) showed highest biological activity against almost all organism tested. Complex S₁ (Mn(C₄H₄O₆)).H₂O) showed less activity against certain bacteria like *Escherichia coli*, *Bacillus subtilis* and *C. albicans*. Sample S₃ (MnFe(C₄H₄O₆)).H₂O) showed moderate antimicrobial activity against almost all tested bacteria.

Table 1: Elemental Analysis of Mn-Fe Tartrates

Complex	Formula weight (gm)	C		H		Mn		Fe	
		Obs	Cal	Obs	Cal	Obs	Cal	Obs	Cal
Manganese tartrate. monohydrate Mn (C ₄ H ₄ O ₆) ₃ H ₂ O (S ₁)	516.94	25.38 2	27.85 62	2.685	2.7082	10.186	10.628	0.00	0.00
Ferrous tartrate. monohydrate Fe(C ₄ H ₄ O ₆) ₃ H ₂ O (S ₂)	517.84	27.15 0	27.80 7	2.697	2.7035	0.00	0.00	10.715	10.785
Manganese- Ferrous tartrate.									

monohydrate $Mn_{(0.5)}Fe_{(0.5)}(C_4H_4O_6)_3 H_2O$ (S ₃)	517.47	26.98	27.83	2.649	2.706	5.178	5.309	5.385	5.3972
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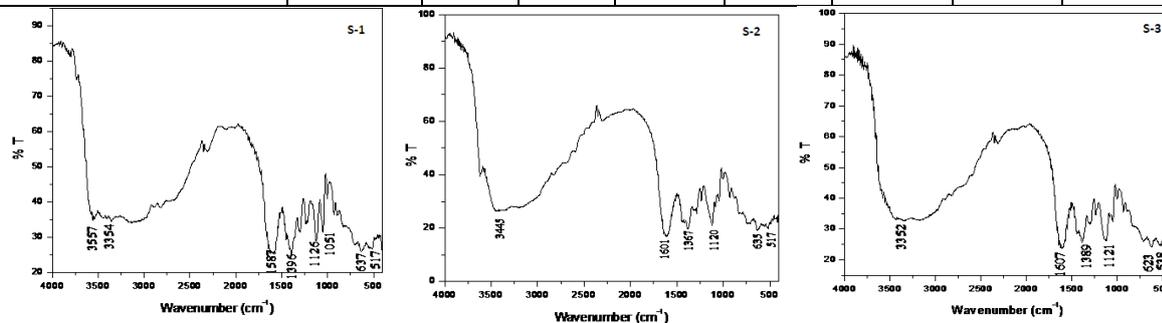


Fig. 1: IR Spectra of Mn-Fe tartrate complexes

Table 2: IR Spectral Data of Mn-Fe tartrate complexes

Manganese tartrate. monohydrate $Mn(C_4H_4O_6)_3 H_2O$ (S ₁)	Ferrous tartrate. monohydrate $Fe(C_4H_4O_6)_3 H_2O$ (S ₂)	Manganese- Ferrous tartrate. monohydrate $Mn_{(0.5)}Fe_{(0.5)}(C_4H_4O_6)_3 H_2O$ (S ₃)	Assignment
3454	3445	3352	Co-ordinate H ₂ O
2850	-	2850	v: C-H (Carbonyl)
2300	2350	2350, 2250	v:O-H (Alcohol)
1587	1601	1607	v _{asym} :C-O
1336	1367	1389	v _{sym} : C-O (Carboxyl)
1126, 1055	1120	1121	v _{sym} : C-O (alcohol)
720	-	720	v _{sym} : C-C
-	635	623	v: H-O-H
517	517	538	v:M-O

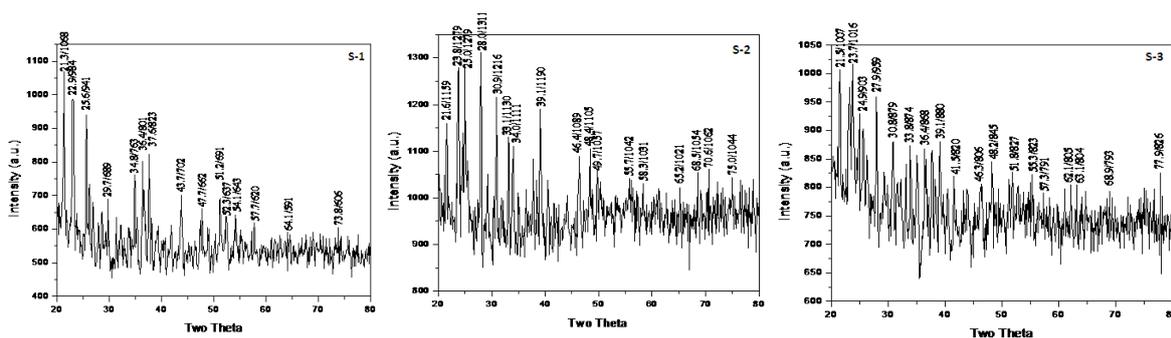


Fig. 2: XRD patterns of Mn-Fe tartrate complexes

Table 3: Observed d-Spacing Values (A^o) of Mn-Fe Tartrate complexes

Manganese tartrate. monohydrate Mn (C ₄ H ₄ O ₆) ₃ H ₂ O (S ₁)	Ferrous tartrate. monohydrate Fe(C ₄ H ₄ O ₆) ₃ H ₂ O (S ₂)	Manganese- Ferrous tartrate. monohydrate Mn _(0.5) Fe _(0.5) (C ₄ H ₄ O ₆) ₃ H ₂ O (S ₃)
4.1660	4.1083	4.1281
3.8650	3.7339	3.7502
3.4651	3.5580	3.5613
2.9996	3.1831	3.1857
2.5711	2.8904	2.8937
2.4610	2.7038	2.6434
2.3856	2.6344	2.4610
2.0668	2.3015	2.2980
1.9029	1.9551	2.1705
1.7810	1.8788	1.9561
1.7459	1.8327	1.8834
1.6922	1.6488	1.7611
1.5948	1.5811	1.6576
1.4507	1.4297	1.6048
1.2821	1.3684	1.4917
	1.3329	1.4706
	1.2653	1.3605

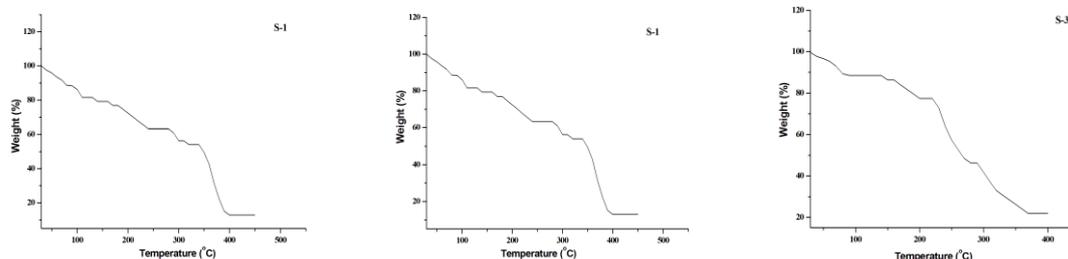


Fig. 3: TGA of Mn-Fe Tartrates

Table 4: TGA data of Mn-Fe Tartrates

Complex	% Mass loss	Temp. Range °C
Manganese tartrate. monohydrate Mn (C ₄ H ₄ O ₆) ₃ H ₂ O (S ₁)	18.54	110
	87.22	400
Ferrous tartrate. monohydrate Fe(C ₄ H ₄ O ₆) ₃ H ₂ O (S ₂)	12.94	100
	81.24	420
Manganese- Ferrous tartrate. monohydrate Mn _(0.5) Fe _(0.5) (C ₄ H ₄ O ₆) ₃ H ₂ O (S ₃)	11.45	90
	78.13	370

Table 5: Antibacterial Activity of Mn-Fe Tartrates (zone of inhibition in mm)

No.	Chemical	Escherichia coli	Bacillus subtilis	Staphylococcus aureus	C. albicans	A. niger	P. chrysogenum
1	Mn (C ₄ H ₄ O ₆) ₃ H ₂ O [S1]	01	3	4	4	7	14
2	Fe(C ₄ H ₄ O ₆) ₃ H ₂ O [S2]	16	8	9	16	12	1
3	MnFe(C ₄ H ₄ O ₆) ₃ H ₂ O [S3]	8	4	12	3	1	5
4	Control [Sterile distilled water]	0	0	0	0	0	0



Fig 3: Antibacterial Activity Plates of synthesized complexes (S1, S2 and S3)

IV. CONCLUSIONS

- i) Result of element analysis of synthesized Mn-Fe tartrate complexes are well matched with calculated ones.
- ii) Presence of water of crystallization for these complexes is confirmed on the basis of TGA curves.
- iii) IR spectra of these complexes are found to have bidentate linkage of carboxylate group with metal ion.
- iv) The study of X-ray powder patterns observed and d-spacing values of these precursors shows that the complexes are polycrystalline with octahedral arrangement.
- v) Evaluation of microbial activity of these complexes is seemed to possess antimicrobial activity against Escherichia coli, Bacillus subtilis, Staphylococcus aureus, C. albicans, A. niger and P. chrysogenum.
- vi) Biological activity of sample S₂ i.e. Fe(C₄H₄O₆).H₂O have showed highest activity against Escherichia coli, C. albicans and A. niger.

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