National Conference on New Horizons in Material Science
© 2018 IJSRSET | Volume 4 | Issue 3 | Print ISSN: 2395-1990 | Online ISSN : 2394-4099
Themed Section : Engineering and Technology

# Synthesis, Characterization and Antimicrobial Activity of Mn-Fe Tartarate Composites

**N. S. Dhavale[1], C. S. Patil[2], S.M. Vhankate[1], V. B. Tadke[1], R.P. Pawar[2] and S.S. Pawar[1*]**

[1]Department of Chemistry, Fergusson College (Autonomous), Pune, Maharashtra, India
[2]Department of Chemistry, Deogiri College, Aurangabad, Maharashtra, India

## ABSTRACT

The synthesis of Mn-Fe mixed metal tratarate composites of six different proportions is done by co-precipitation method stoichiometrically. The composites of Mn-Fe tartarates formed are characterized by analytical techniques like AAS, IR study, XRD patterns, thermal and elemental analysis. Characterization data of all six complexes reveals that the composites synthesized have polycrystalline nature and bidentate ligand. These composites have antimicrobial activity against micro-organisms like *E.coli, Bacillus subtilis, Staphylococcus aureus, C. albieans, A. niger, P. chrysogenum* ect.

**Keywords:** Mn-Fe Tartarates Composites, Antimicrobial Activity

## I. INTRODUCTION

Dicarboxylates of metals are often exists in natural systems in various conversions and in the process food stuff manufactured [1, 2, 3]. Dicarboxylates of transition and non-transition metals finds important applications in petroleum, paints, cement, PVC and vegetable fat industries [4]. The synthesis and characterization of some polynuclear complexes (i.e. Oxalates) containing Fe, Mn and Zn are useful as precursors by forced hydrolysis [5-9].

This work includes synthesis of Mn-Fe mixed metal tartarates composites using co-precipitation method using various proportions (Sample MFT-1 to MFT-6). The composites of Mn-Fe tartarates are further characterized using analytical methods. The antimicrobial activity of these composites is tested against many micro-organisms such as *E.coli, Bacillus subtilis, Staphylococcus aureus, C. albieans, A. niger and P. chrysogenum.*

## II. METHODS AND MATERIAL

### A) Synthesis of Mn-Fe tartarates:

The Mn-Fe tartarate composites with six different composites such as $[M_xM^1_{(1-x)}(C_4H_4O_6)]\cdot H_2O$, Where M and $M^1$ are Mn and X =0.2, 0.4, 0.6, 0.8 and 1.0, are prepared by co-precipitation method by taking analytical grade $MnSo_4\cdot 2H_2O$ and $FeSO_4\cdot 7H_2O$ in distilled water.

The mixture of metal sulphate solution is prepared with respect to molar ratio of Mn and Fe and placed in a beaker. pH less than 6 is adjusted, so that metal hydroxide does not precipitate. The solution is stirred vigorously and sodium tartarate (10%) solution is added slowly with stirring till a permanent precipitate is obtained. Acetone is added to ensure a high yield of product. The solution is stirred for 30 minutes and the filtered. The product is washed with cold distilled water and then with acetone. The product is dried at ambient temperature.

Such type of six samples of Mn-Fe tartararte composites (MFT-1 to MFT-6) are synthesized [10-13].

### B) Characterization of synthesized Mn-Fe tartarates composites:

The CHN analysis of six synthesized composites (MFT-1 to MFT-6) is carried out using C.E. instrument using K factor calibration method. The metal contents present were estimated by atomic absorption spectroscopy. The Mn-Fe tartarate composites of six different proportions

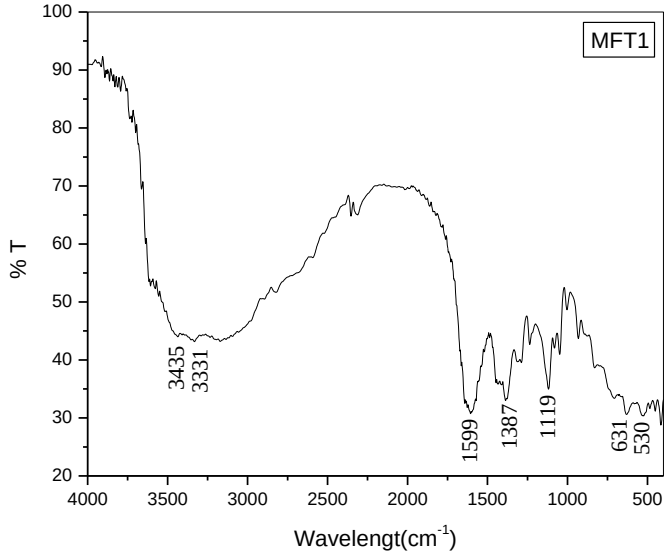
are further characterized using IR study and XRD pattern study of the composites. Thermal decomposition of all Mn-Fe tartarate composites is studied by thermo gravimetric analysis with temperature range $30^0$ to $750^0$C.

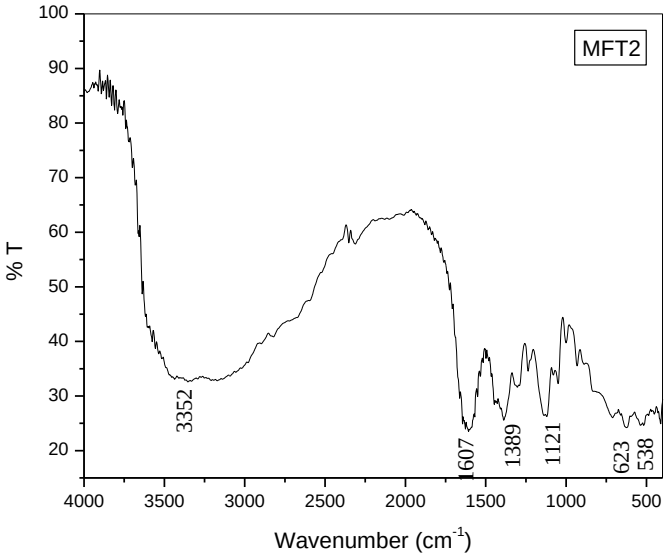
**C) The study of antimicrobial activity of synthesized Mn-Fe tartarate composites:**

Initially 1% solution of six composites is prepared in distilled water. Agar-plates were used and labeled for bacterial culture. Antimicrobial activity of synthesized composites is studied using organisms such as. *i) E.coli, ii) Bacillus subtilis, iii) Staphylococcus aureus, iv) C. albieans, v) A. niger, vi) P. chrysogenum* 0.5 ml of bacterial cultures were spread, inoculated and incubated at $37^0$C for 30 minutes. A well was bored at the center of medium in each plate aseptically. 0.1 ml of each tartarate complex solution is poured aseptically in each respective well and incubated for diffusion at $40^0$C for 1 hr. All those plates were incubated at $37^0$C for 48 hrs and the results are studied.

**Table I** : Elemental Analysis

| Complex | Formula weight (gm) | C wt. % | | H wt. % | | Mn wt. % | | Fe wt. % | |
|---|---|---|---|---|---|---|---|---|---|
| | | Obs | Cal | Obs | Cal | Obs | Cal | Obs | Cal |
| MFT-1 $Mn_{0.2}Fe_{0.8}(C_4H_4O_6)_3.\ 2H_2O$ | 535.66 | 24.92 | 26.88 | 2.79 | 2.99 | 1.85 | 2.051 | 8.12 | 8.34 |
| MFT-2 $Mn_{0.4}Fe_{0.6}\,C_4H_4O_6)_3.\ 2H_2O$ | 557.456 | 24.68 | 25.83 | 2.68 | 2.87 | 3.75 | 3.853 | 9.81 | 9.95 |
| MFT-3 $Mn_{0.6}Fe_{0.4}\,(C_4H_4O_6)_3.\ 2H_2O$ | 535.3 | 26.77 | 26.90 | 2.94 | 2.99 | 5.98 | 6.158 | 4.014 | 4.173 |
| MFT-4 $Mn_{0.8}Fe_{0.2}(\,C_4H_4O_6)_3.2\ H_2O$ | 535.048 | 26.84 | 26.91 | 2.74 | 2.99 | 8.068 | 8.215 | 1.99 | 2.087 |
| MFT-5 $Mn(C_4H_4O_6)_3.\ 2H_2O$ | 5534.94 | 26.84 | 26.92 | 2.81 | 2.7082 | 10.066 | 10.27 | 0.00 | 0.00 |
| MFT-6 $Fe(C_4H_4O_6)_3.\ 2H_2O$ | 535.84 | 26.76 | 26.87 | 2.94 | 2.986 | 0.00 | 0.00 | 10.31 | 1o.42 |



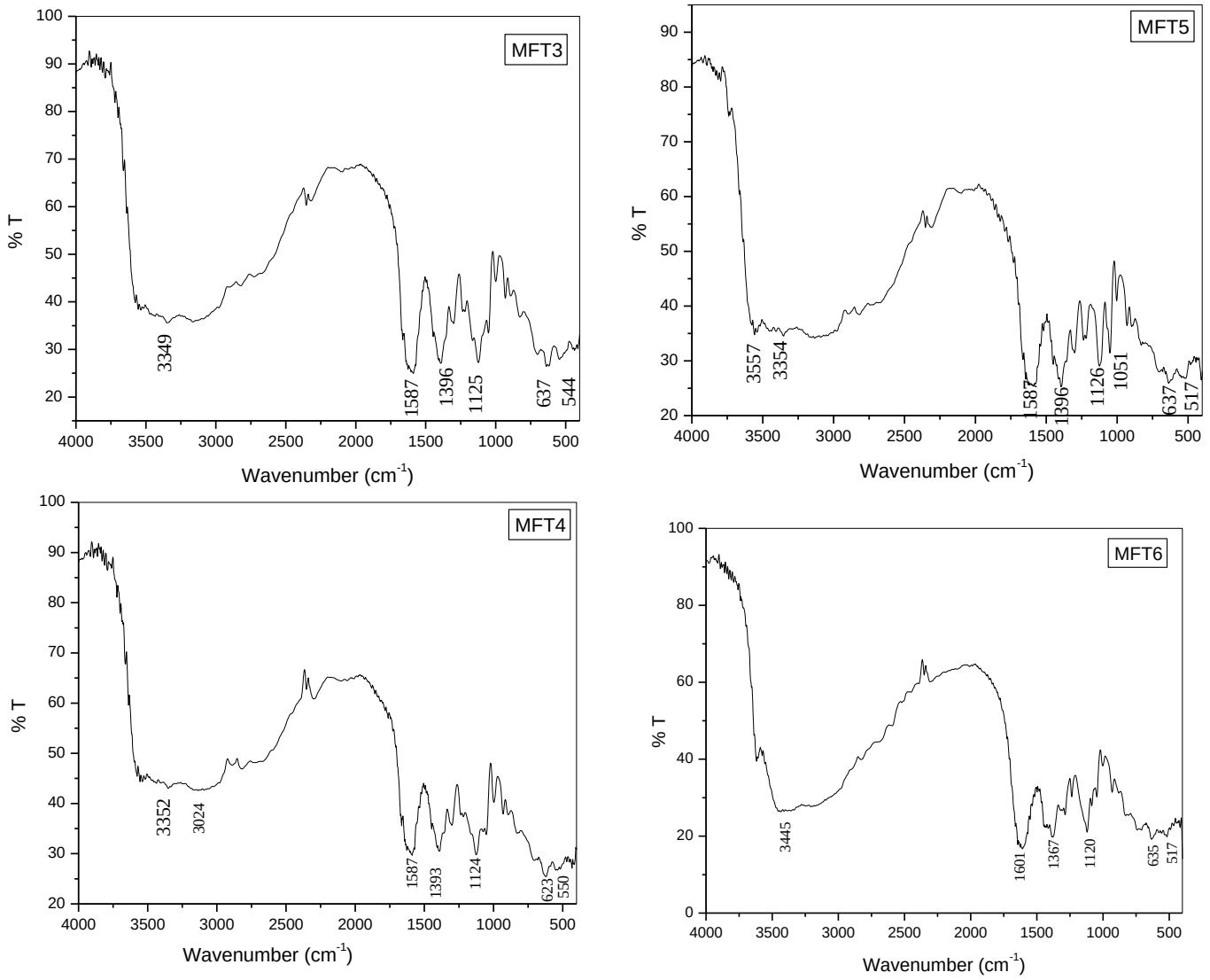



**Figure 1:** IR Spectra of tartarate complexes

**Table II :** IR Spectral Data of Mn-Fe tartarate composites

| MFT-1 $Mn_{0.2}Fe_{0.8}(C_4H_4O_6)_3 \cdot 2H_2O$ | MFT-2 $Mn_{0.4}Fe_{0.6}(C_4H_4O_6)_3 \cdot 2H_2O$ | MFT-3 $Mn_{0.6}Fe_{0.4}(C_4H_4O_6)_3 \cdot 2H_2O$ | MFT-4 $Mn_{0.8}Fe_{0.2}(C_4H_4O_6)_3 \cdot 2H_2O$ | MFT-5 $Mn(C_4H_4O_6)_3 \cdot 2H_2O$ | MFT-6 $Fe(C_4H_4O_6)_3 \cdot 2H_2O$ |
|---|---|---|---|---|---|
| 3435 | 3352 | 3349 | 3352 | 3557 | 3445 |
| 3331 | - | - | 3024 | 3354 | 1601 |
| 1599 | 1607 | 1587 | 1587 | 1587 | 1367 |
| 1387 | 1389 | 1396 | 1393 | 1396 | 1120 |
| 1119 | 1121 | 1125 | 1124 | 1126 | 635 |
| 631 | 623 | 637 | 623 | 1051 | 517 |
| 530 | 538 | 544 | 550 | 637 | |
| | | | | 517 | |

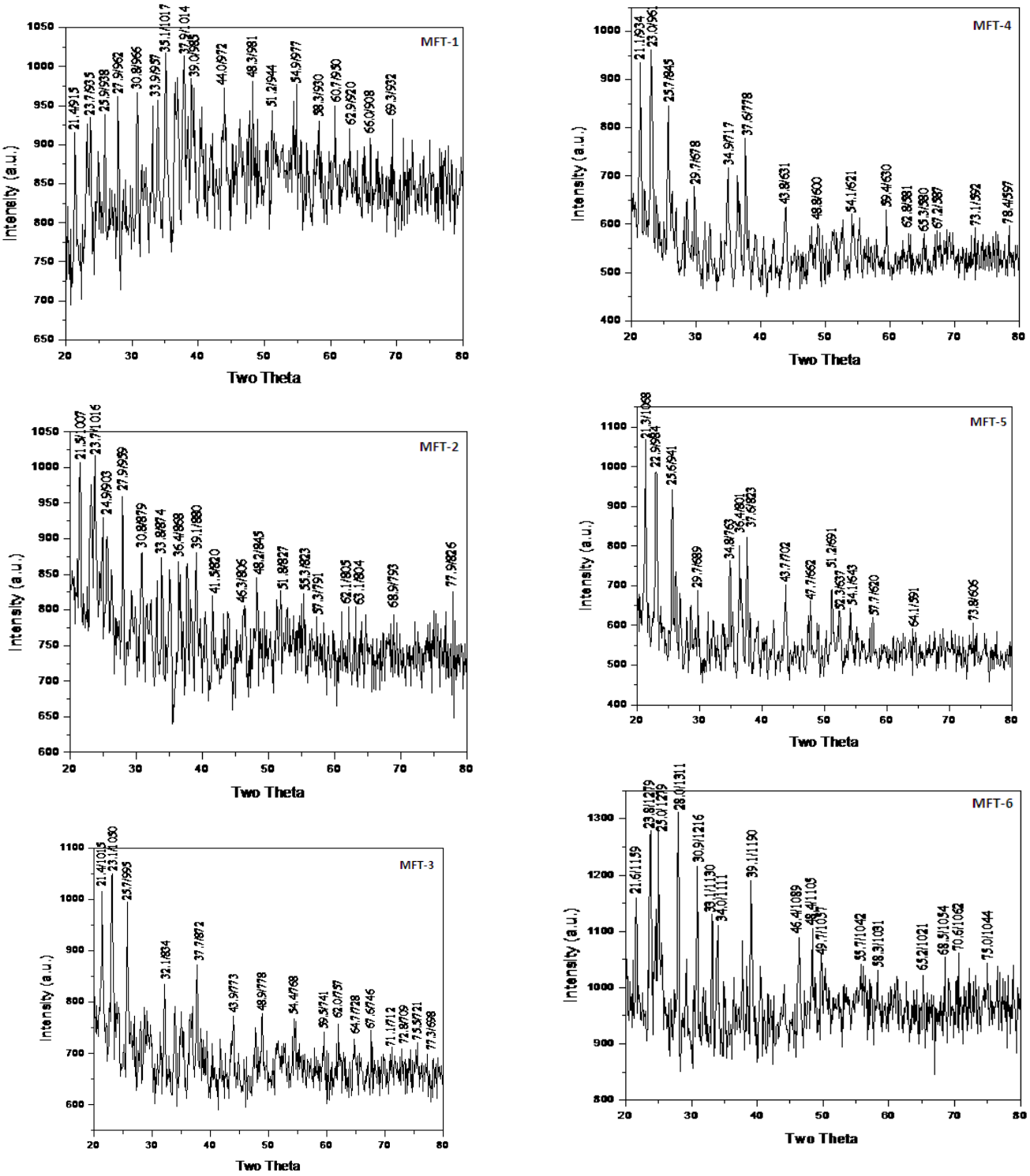
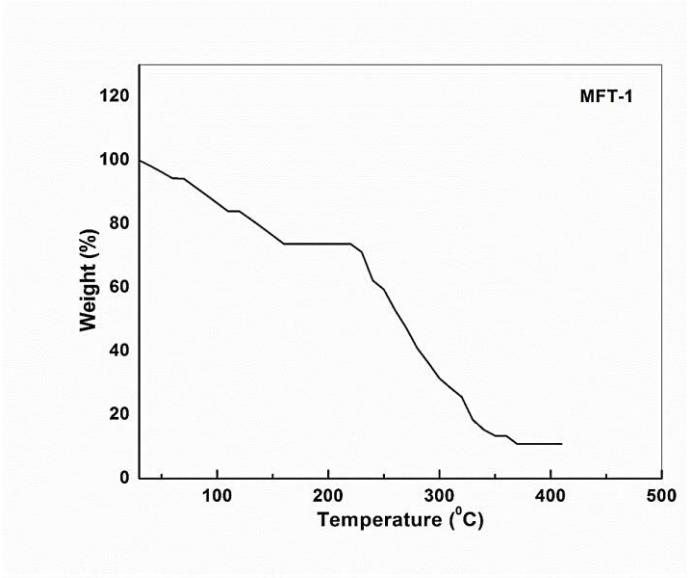


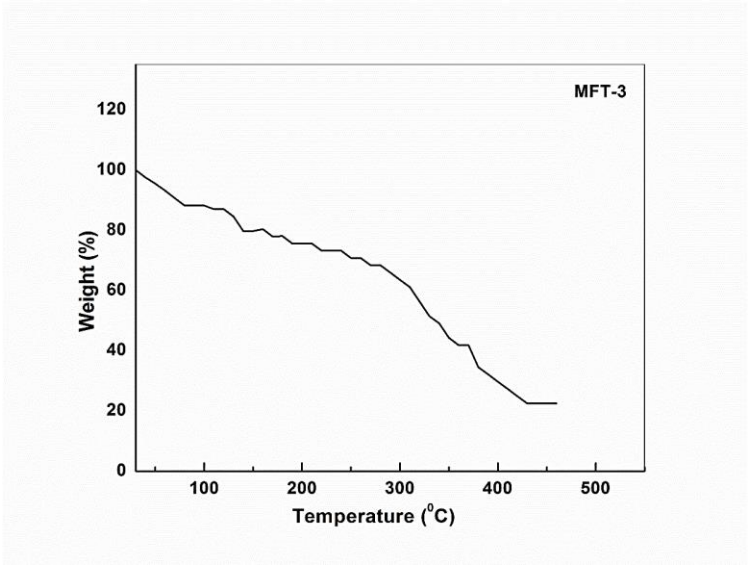

**Figure 2 :** XRD patterns of Mn-Fe tartarate comosites

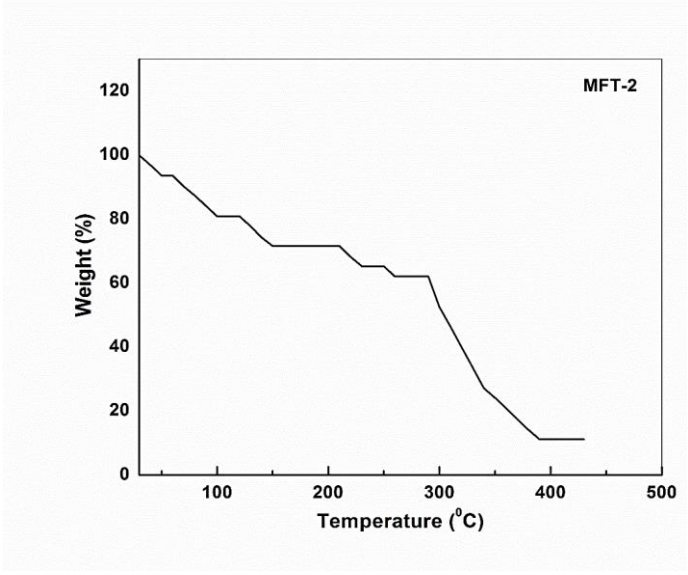
**Table III :** Observed d-Spacing Values ($A^O$) of Mn-Fe tartarate composites

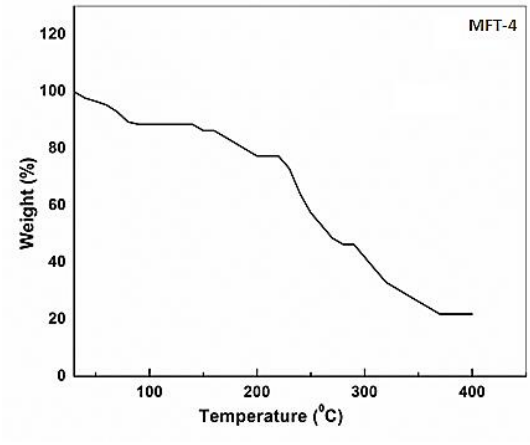
| MFT-1<br><br>$Mn_{0.2}Fe_{0.8}$ $(C_4H_4O_6)_3.2H_2O$ | MFT-2<br><br>$Mn_{0.4}Fe_{0.6}$ $C_4H_4O_6)_3.2H_2O$ | MFT-3<br><br>$Mn_{0.6}Fe_{0.4}$ $C_4H_4O_6)_3.2H_2O$ | MFT-4<br><br>$Mn_{0.8}Fe_{0.2}$ $C_4H_4O_6)_3.2H_2O$ | MFT-5<br><br>Mn $(C_4H_4O_6)_3$ $2H_2O$ | MFT-6<br><br>$Fe(C_4H_4O_6)_3$ $2H_2O$ |
|---|---|---|---|---|---|
| 4.1325 | 4.1281 | 4.1481 | 4.1887 | 4.1660 | 4.1083 |
| 3.7375 | 3.7502 | 3.8457 | 3.8496 | 3.8650 | 3.7339 |

| | | | | | |
|---|---|---|---|---|---|
| 3.4251 | 3.5613 | 3.4512 | 3.4527 | 3.4651 | 3.5580 |
| 3.1857 | 3.1857 | 2.7799 | 2.9996 | 2.9996 | 3.1831 |
| 2.8926 | 2.8937 | 2.3797 | 2.5642 | 2.5711 | 2.8904 |
| 2.6299 | 2.6434 | 2.0574 | 2.3863 | 2.4610 | 2.7038 |
| 2.5540 | 2.4610 | 1.8588 | 2.0808 | 2.3856 | 2.6344 |
| 2.3665 | 2.2980 | 1.6833 | 1.8624 | 2.0668 | 2.3015 |
| 2.3028 | 2.1705 | 1.5508 | 1.6922 | 1.9029 | 1.9551 |
| 2.0525 | 1.9561 | 1.4940 | 1.5533 | 1.7810 | 1.8788 |
| 1.8797 | 1.8834 | 1.4382 | 1.4774 | 1.7459 | 1.8327 |
| 1.7798 | 1.7611 | 1.3834 | 1.4267 | 1.6922 | 1.6488 |
| 1.6684 | 1.6576 | 1.3238 | 1.3912 | 1.5948 | 1.5811 |
| 1.5791 | 1.6048 | 1.2970 | 1.2927 | 1.4507 | 1.4297 |
| 1.5226 | 1.4917 | 1.2572 | 1.2181 | 1.2821 | 1.3684 |
| 1.4745 | 1.4706 | 1.2327 | | | 1.3329 |
| 1.4126 | 1.3605 | | | | 1.2653 |



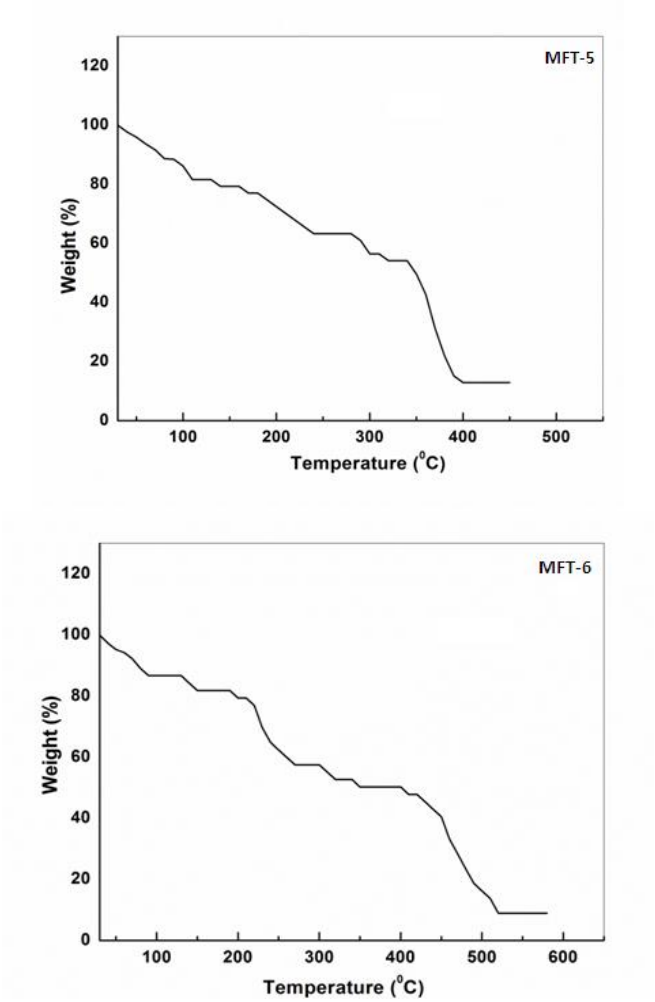



**Figure 3 :** TGA curves of Mn-Fe tartarate composites

**Table IV :** TGA Results of Mn-Fe tartarate complexes

| **Complex** | **Observed % Mass loss** | **Temp Range $^{o}$C** |
|---|---|---|
| MFT-1 $Mn_{0.2}Fe_{0.8}(C_4H_4O_6)_3.2H_2O$ | 15.82 | 109 |
| | 89.30 | 380 |
| MFT-2 $Mn_{0.4}Fe_{0.6}C_4H_4O_6)_3.2H_2O$ | 18.67 | 105 |
| | 88.69 | 400 |
| MFT-3 $Mn_{0.6}Fe_{0.4}C_4H_4O_6)_3.2H_2O$ | 11.59 | 90 |
| | 77.33 | 435 |
| MFT-4 $Mn_{0.8}Fe_{0.2}C_4H_4O_6)_3.2H_2O$ | 11.45 | 90 |
| | 78.13 | 370 |
| MFT-5 $MnC_4H_4O_6)_3.2H_2O$ | 13.26 | 115 |
| | 90.59 | 525 |
| MFT-6 $FeC_4H_4O_6)_3.2H_2O$ | 12.96 | 112 |
| | 90.59 | 530 |

**Table VI :** Antibacterial Activity (zone of inhibition in mm) of Mn-Fe tartarate Composites Zones of Inhibition [in mm]

| No. | Chemical | Escherichia coli | Bacillus subtilis | Staphylococcus aureus | C. albieans | A. niger | P. chrysogenum |
|---|---|---|---|---|---|---|---|
| 1 | MFT-1 $Mn_{0.2}Fe_{0.8}(C_4H_4O_6)_3.2H_2O$ | 04 | 02 | 06 | 11 | 07 | 05 |
| 2 | MFT-2 $Mn_{0.4}Fe_{0.6}C_4H_4O_6)_3.2H_2O$ | 08 | 04 | 14 | 03 | 02 | 05 |
| 3 | MFT-3 $Mn_{0.6}Fe_{0.4}C_4H_4O_6)_3.2H_2O$ | 13 | 07 | 09 | 07 | 06 | 02 |
| 4 | MFT-4 $Mn_{0.8}Fe_{0.2}C_4H_4O_6)_3.2H_2O$ | 12 | 11 | 02 | 02 | 05 | 01 |
| 5 | MFT-5 $MnC_4H_4O_6)_3.2H_2O$ | 01 | 03 | 04 | 04 | 07 | 11 |
| 6 | MFT-6 $Fe(C_4H_4O_6)_3.2H_2O$ | 11 | 08 | 09 | 11 | 10 | 01 |
| 7 | Control [Sterile distilled water] | 00 | 00 | 00 | 00 | 00 | 00 |

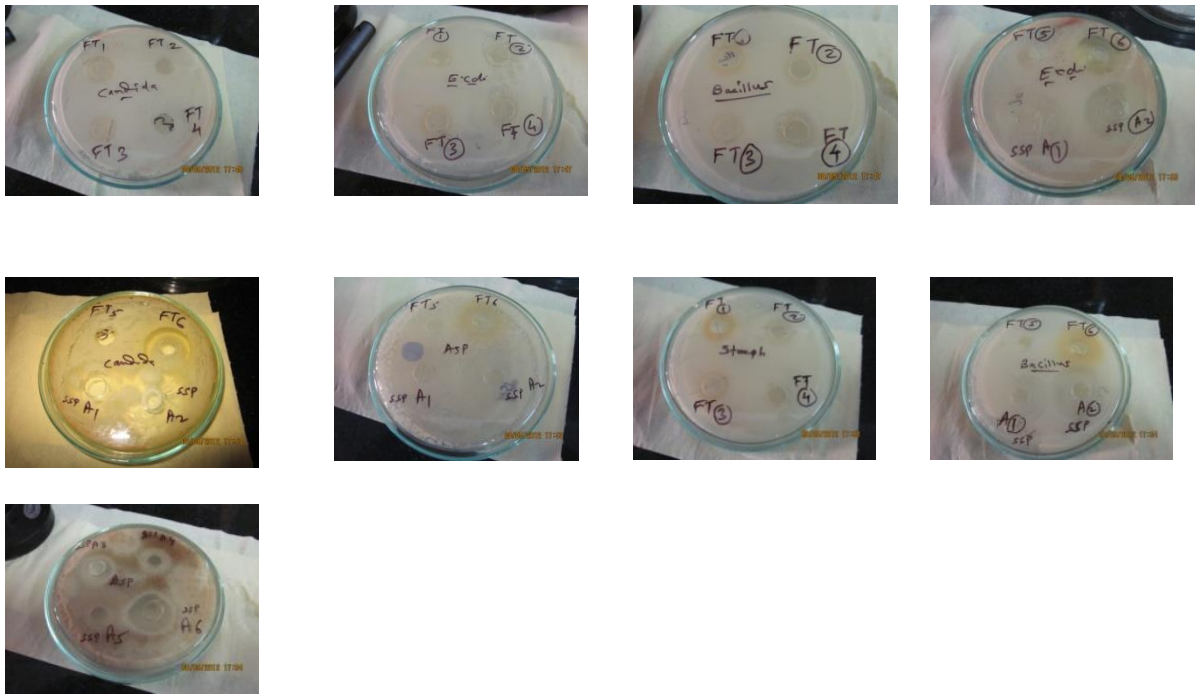


**Figure 4** : Antibacterial Activity Plates of synthesized metal composites

## III. RESULT AND DISCUSSION

The Mn-Fe tartarate composites of six different proportions (MFT-1 to MFT-6) are synthesized by co-precipitation method and their elemental analysis reveals that the observed weight % of element is in good agreement with calculated values (Table 1).

The study of IR spectra of these six samples (MFT-1 to MFT-6) showed characteristic frequencies corresponding to many groups like carbon-oxygen, metal-oxygen, carbon-hydrogen, -OH etc. on the basis of symmetric and antisymmetric starching frequencies observed confirms the tetradentate linkage of tartarate group attached to Mn and Fe metal atoms (Table 2). The presence of bands such as $v_{asy}$(OCO) suggests the co-ordination of both $COO^-$ groups present in the tartaric acid molecule to metal ion. The infrared suggests that the probable structure of Mn-Fe tartarate complexes is octahedral.

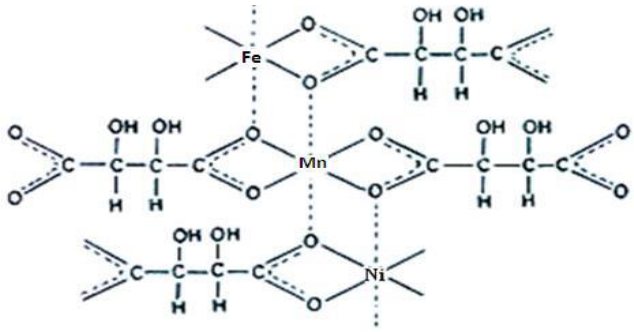
The XRD patterns of Mn-Fe tartarate composites showed certain sharp lines with many broad lines (fig. 2). Therefore the composites synthesized are polycrystalline. The d-spacing values are given in table 3.

The thermo gravimetric analysis indicates the loss of water molecules at about 100 to 110 $^0$C. The % loss for water molecule is matched with theoretical loss. There is a loss of CO, $CO_2$ and $C_2H_4$ molecules within 180$^0$C to 350$^0$C. Thermal study suggests probable reactions.

1) $Mn.Fe(C_4H_4O_6).2H_2O$ -------→ $Mn.Fe(C_4H_4O_6)$ + $2H_2O$

2)| $Mn.Fe(C_4H_4O_6)$ ------→ $Mn.Fe.O_2$ + $2CO_2$ + $C_2H_4$

The characterization of all six Mn-Fe tartarate composites using techniques like CHNS analysis, IR study, XRD study, AAS analysis and TGA suggest the following type of structure.




These synthesized Mn-Fe tartarate composites show antimicrobial activity against micro-organisms such as *E.coli, Bacillus subtilis, Staphylococcus aureus, C. albieans, A. niger and P. chrysogenum.* Sample MFT-2 shows highest activity against *Staphylococcus aureus,*

and sample MFT-3 is more active against *E.coli.*(fig. 3). All these complexes (A1 to A6) possess potential to inhibit the growth of gram positive as well as gram negative bacteria selected indicating their possible to use as bacterial agent.

## IV. CONCLUSION

Six newly mixed metal composites of Mn-Fe tartarates (MFT-1 to MFT-6) were synthesized and characterized by different techniques. By thermogravimetric analysis percentage of water of crystallization in the complexes were confirmed. Elemental compositions of complexes are in good agreement to the calculated one. From XRD patterns polycrystalline nature of the complexes were revealed. Antimicrobial activity of the complexes was carried c These complexes have shown significant antimicrobial activity against studied micro-organisms. Sample MFT-2 showed highest activity against bacteria *Staphylococcus aureus*. Sample MFT-4 is moderately active against bacteria *P. chrysogenum.*

## V. REFERENCES


[1]. The Calender of food Chemistry, WPL is , Warszawa (1954)
[2]. Z. Inzuk, The progress of Microbiology, Part- IV, PWN Warszawg (1996).
[3]. J. Janicek, J. Pokorny and J.Dawidek, Food Chemistry, (WNT, Warszawa,(1977).
[4]. S.B. Elliott " The alkaline earth and heavy Metal Soaps" Reinhold, New York (1946).
[5]. S.Hamada, S. Nuseki and Y. Kudo, Bull. Chem. Soc. Japan
[6]. 59 (1986) 3443.
[7]. S.Hamada and E. Matijevic, J. Colloid. Interface Sci., 84 (1981) 274.
[8]. E.Motijevic, Acc. Chem. Res., 14 (1981) 22.
[9]. L. Bo and Z. Leyi, IEEE Trans. Mag., MAG-17 (1981) 3144.
[10]. M.Brezeanu, L.Patron, E. Cristurean, D. Marinescu, A. Antoniu M. Andruh, O. Carp, M. Andruh, A. Gheorghe and N. Stanica, Rev. Roumaine Chim. 38 (1993) 1291.
[11]. S. S. Pawar, C. S. Patil , V. B. Tadke, S. M. Vhankate, S. A. Dhanmane, G. R. Pathade and R. P. Pawar, Int.Journal of Pharm. Study and Res., 5(4) (2014), 1557 – 1565.
[12]. S. M. Vhankate, S. S. Pawar S, S. A. Dhanmane, N. S. Dhawale, K. Fulzele, C. S. Patil, R. P. Pawar and V. B. Tadke, Shri Ramanand Tirth Marath. Univ. Nanded, Res. J. Sci., 2(1) (2013), 88 -100.
[13]. A. K. Nikumbh, A.V. Nagawade, V. B. Tadke, P. P. Bakare, J. Mater. Sci. 36(2001)1.
[14]. A. K. Nikumbh and V. B. Tadke, `Thermochim Acta., (2001).