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Crystallization, FT-IR and powder XRD study of gel grown iron-manganese-nickel ternary dextro-tartrate crystals in hydro silica gel

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ABSTRACT

In the present investigation, iron-manganese-nickel ternary dextro-tartrate crystals have been grown by single diffusion reaction gel growth technique in hydro silica gel.Crystals having green colourand spheruliticmorphology were grown within the gel column. The grown crystals were subjected to powder X-ray diffraction technique to study cell parameters and crystalline nature of iron-manganese-nickel ternary dextro-tartrate crystals. FTIR spectroscopic analysis was performed to identify the presence of various functional groups in the iron-manganese-nickel ternary dextro-tartrate crystals.

Key words: gel growth, crystal growth, spheruliticmorphology,iron-manganese-nickel ternary dextro-tartrate crystals, powder XRD, FT-IR.

INTRODUCTION

Compounds of different tartrates draw attention of many researchers because of its various applications in science and technology as well as in the field of pharmaceutical and even medical sciences, in addition to industrial purpose. For example, various ferrous tartrate crystals are used to prevent anaemia in animals [1] and use of manganese tartrate crystals in chemical temperature indicators [2]. Different types of tartrate crystals can be grown in hydro silica gel. Growth and characterization of different crystals of pure and mixed tartrate compounds were reported by many researchers [3-18].A novel method of growing calcium tartrate single crystals was developed,in which the crystals were hanging freely in supernatant solution assisted by the fine filaments of one type of fungi, viz., *Aspergillums niger*[19].Some tartrate crystals of ternary system were reported earlier, viz., cadmium-zirconium-sodium[20] and iron-manganese-cobalt[21].

In the present investigation, growth of iron-manganese-nickel dextro tartrate ternary crystals and their characterization by FT-IR spectroscopy and powder X-ray diffractiontechniqueswere reported. Cell parameters and crystal structure were evaluated by powder X-ray diffraction and the FT-IR spectroscopic analysisconfirmed the presence of different functional groups and metal-oxygen bonds in the grown crystals.

MATERIALS AND METHODS

Crystal Growth:
Single diffusion gel growth technique was used to grow iron-manganese-nickel dextro tartrate crystals. The glass test tubes were used as crystallization apparatus. Dimensions of glass test tubes were 25 mm diameter and 140 mm length. Hydro silica gel was used as a gel media to grow iron-manganese-nickel dextro tartrate crystals. The hydro

silica gel was prepared by aqueous sodium metasilicate solution having 1.06 specific gravity was acidified by 1 M tartaric acid in such a manner that pH 5.0 could be set for gel mixture. This gelmixture was transferred to different glass test tubes and allowed to set into gel form. The gel was set within forty eight hours duration. After setting gel, 10 ml of supernatant solution wasgently poured over a set gel without break it. The supernatant solution was prepared by mixing of equal amount of 1 M $FeSO_4$, 1 M $NiCl_2$and 1 M $MnSO_4$.

The crystals weregrown after three weeks. After three weeks, it was noticed that supernatant solution was absorbed by the gel and green colour spread within gel as well as spheruliticcrystals having green colour were grown within the gel column. The morphology of grown crystals was spherulitic.

Characterization:

FTIR Spectroscopic analysis:

Infrared spectroscopy (IR Spectroscopy) is the spectroscopy,which deals with infrared region of the electromagnetic spectrum.Basically, IR Spectroscopic technique covers a range of techniques, mostly based on absorption spectroscopy.

FTIR is useful technique to identify organic and/or inorganic substances. It can also be useful to quantitate some components of an unknown mixture. This technique isequally applicable to the analysis of solids, liquids and gases. FTIR is powerful tool for identifying types of chemical bonds hence functional groups. FTIR spectroscopic analyses of some crystals comprising of pure and mixed tartrate compounds arereported byearlier authors [22-25].

The FTIR spectrum of powdered sample of grown iron-manganese-nickel ternary dextro tartrate crystals was recorded in the range of 400 $cm^{-1}$to 4000 cm^{-1} using a Perkin Elmer Fourier transform infrared spectrometer Model: Spectrum GX.

Powder XRD Analysis:

Crystalline nature of materials can be studied by powder X-ray diffraction (XRD) technique. The crystal structure of micro-clusters and the approach of their structure to that of the bulk phase has been a subject of detailed study for many years [26]. In micro-clusters the surface atomsrepresent a large fraction of the material and they areexpected to play a fundamental role in determining thecrystallographic and electronic properties of the clusters [27].Powder XRD analysis of crystals containing pure and mixed tartrate compounds are previously reported by some authors [8,14,23,28].

Powder X-ray diffraction pattern of iron-manganese-nickel ternary dextro tartrate crystals was recorded using a Philips Xpert X-ray diffractometer with CuKα radiation.

RESULTS AND DISCUSSION

Fourier Transform Infrared Spectroscopic (FTIR) Analysis:

The FTIR spectrum was recorded in a region of 400 $cm^{-1}$to 4000 $cm^{-1}$for iron-manganese-nickel ternary dextro tartrate crystalsis shown in Figure 1. The position of peaksand assignments are documented inTable 1. The absorptions at 3618cm^{-1} and 3452 $cm^{-1}$are due to the asymmetric and symmetric stretching of O-H bond, which normally indicates the presence of water of crystallization. The absorption at 1601$cm^{-1}$is due to the carbonyl C=O group. The C-O stretching vibrations give rise to absorptionsat 1447 cm^{-1},1380cm^{-1}, 1305 cm^{-1}, 1288cm^{-1}, 1237cm^{-1}. The absorptions at 1118cm^{-1} and 1082 cm^{-1} are due to the C-O stretching vibrations. The absorptionsat1118cm^{-1}, 1082 cm^{-1},1047$cm^{-1}$and 1006 cm^{-1} are due to out of plane O-H deformation and C-O stretching. The absorptionspositioned between 716$cm^{-1}$to 517 cm^{-1} are due to the Metal-Oxygen stretching vibrations.

Moreover, calculation is done to determine the mechanical stiffness constant for C=O vibrations positioned at 1601$cm^{-1}$by using the following formula [10,29] which is found to be 1035 Nm^{-1}.

$$\bar{\upsilon} = 1303\sqrt{F\left[\frac{1}{m_1}+\frac{1}{m_2}\right]}.$$

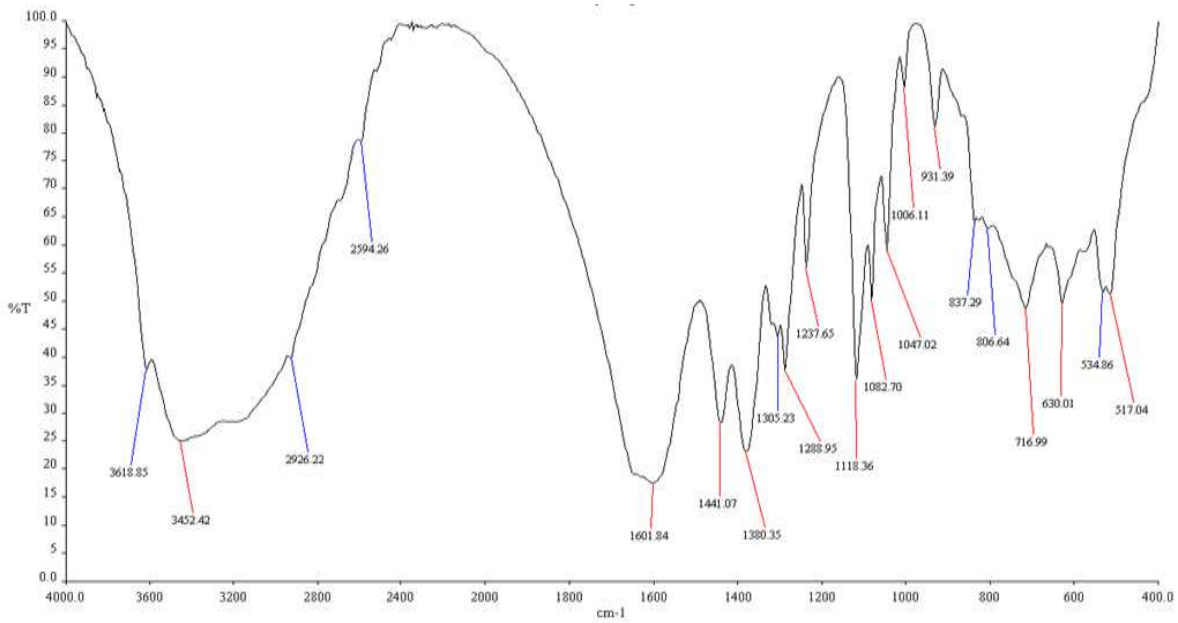

Figure1:FTIR spectrum of iron-manganese-nickel ternarydextro tartrate crystal

Table 1: FTIR peak assignment of iron-manganese-nickel ternarydextro tartrate crystal

Wave numbers in cm^{-1}	Peak assignments
3618, 3452	O-H stretching
1601	C=O stretching
1380, 1305	O-H plane deformation
1477,1380,1305,1288, 1237	C-O stretching
1118, 1082,1047, 1006	O-H deformation out of plane and C-H stretching
716, 630,534, 517	Metal–Oxygen bonding

Powder X-ray diffraction analysis:

X-ray diffraction pattern of powdered sample of iron-manganese-nickel ternary dextro tartrate crystals was recorded by using a Philips Xpert X-ray powder diffractometer with CuKα radiation is shown in Figure 2.

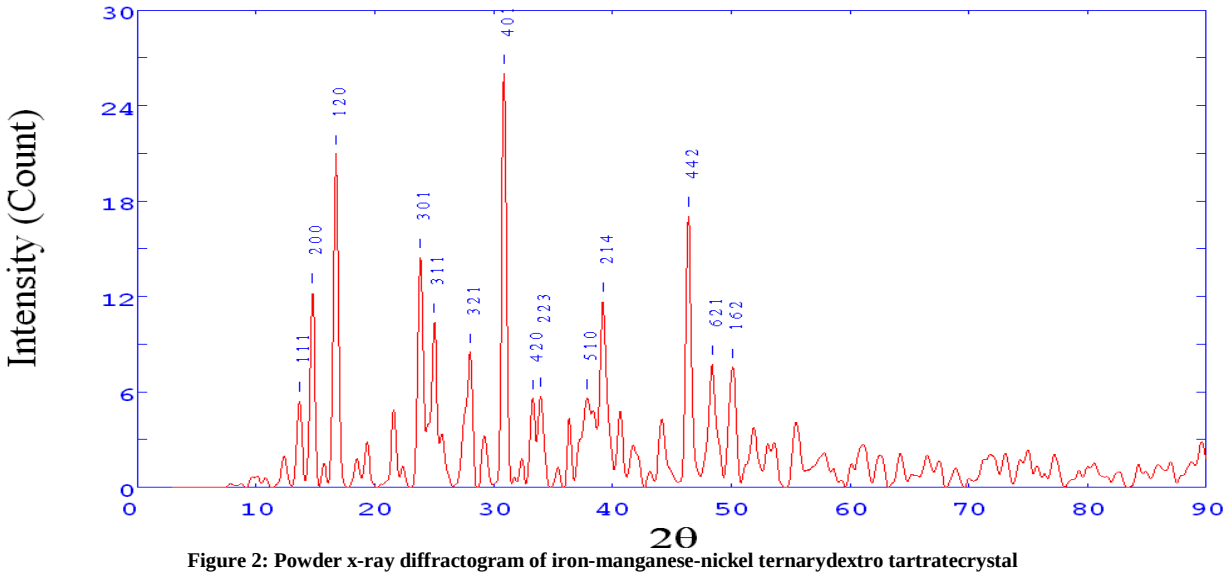

Figure 2: Powder x-ray diffractogram of iron-manganese-nickel ternarydextro tartratecrystal

The crystal structure and unit cell parameters of the sample werecomputed by using powder X computer assisted software. It is observed that the grown crystals were orthorhombic and p-type of lattice. The unit lattice cell parameters is obtained from XRD pattern using powder X software are a = 12.1087 Å, b = 11.8888 Å and c = 10.1098 Å. The calculated and observed values of 2θ and hkl parameters are shown in Table (2).

Table (2): Powder diffraction data of iron-manganese-nickel ternary dextro tartrate crystal

hkl	2 Θ		d		Intensity
	(exp.)	(cal.)	(exp.)	(cal.)	(exp.)
1 1 1	13.648	13.615	6.48307	6.49854	5.45
2 0 0	14.749	14.619	6.00122	6.05435	12.27
1 2 0	16.727	16.600	5.29581	5.33607	20.99
3 0 0	23.833	23.717	3.73057	3.74853	14.47
3 1 1	25.006	24.886	3.55809	3.57504	10.40
3 2 1	28.029	28.120	3.18086	3.17074	8.51
4 0 1	30.881	30.808	2.89332	2.89996	26.05
4 2 0	33.285	33.184	2.68958	2.69753	5.60
2 2 3	33.958	33.948	2.63785	2.63856	5.76
5 1 0	37.871	37.884	2.37380	2.37301	5.61
2 1 4	39.197	39.335	2.29647	2.28874	11.71
4 4 2	46.393	46.392	1.95565	1.95568	17.09
6 2 1	48.390	48.438	1.87950	1.87774	7.76
1 6 2	50.115	49.969	1.81876	1.82375	7.56

CONCLUSION

Iron-manganese-nickel dextro tartrate crystals were grown successfully by single diffusion technique in hydro silica gel. The grown crystals have spherulitic morphology and green colour. With the help of powder X-ray diffraction analysis,it is found that powdered sampleis crystalline in nature and confirms the unit cell parameters and d-values. The presence of O=H, C=O, C-O, C-H and metal- oxygen bonds were confirmed from the FTIR spectroscopy.

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