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Synthesis of Manganese doped Bi_2S_3 Crystal in Gel method and its thermal Characterisations

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ABSTRACT

Manganese doped Bismuth tri sulphide crystals are synthesising using simple gel technique at ambient temperature. X Ray Diffraction analysis was done to determine the structure. Orthorhombic crystal structure was found of grown crystal. FTIR method was utilized for the analysis of various functional groups present in the complex Thermal properties of the crystal was studied by TGA was Discussed .Effect of Doping, and concentration of reactants.

Keywords: Gel method, orthorhombic, XRD, FTIR, TGA

I. INTRODUCTION

The gel method is simple technique for growing perfect and strain free crystals. The principle involved in this method is very simple. The solution of two appropriate compounds, which give rise to the essential insoluble crystalline material by simple reaction, two compounds are chemically reacted in set gel medium . In the gel method, gel prevents turbulence and helps to formation of good crystal by providing a framework of nucleation site. In addition, the gel media has a single place due to gel media control of nucleation centre characteristics. In gel method, convection is not present in gel media. It indicates that, for substances, which are insoluble in water, it is option for growth method.

Many of researchers have been grown undoped and doped crystals with the aim of to study and to identify grown materials for practical and industrial applications. The influence of doping in growth media has been recognized for a long time as a relevant matter in the process of crystallization [1-3]. As for as transition metal doped Bismuth tri sulphide crystals are concerned, information about their growth by gel method, characterization. Therefore, it was felt worthwhile to grow Mn-doped Bismuth tri sulphide crystals and study their properties, which may have potential application in photovoltaic energy. The TGA curve of Manganese doped Bismuth tri sulphide crystal was subjected to study the weight loss measured using TGA method.

The purpose of present paper is to report the growth and influence of various parameters such as concentration of reactants, growth of mechanism of Bismuth tri sulphide crystals in gel with effect of

dopant and molar concentration, its optimum conditions and properties

II. METHODS AND MATERIAL

To grow the Manganese-doped Bi_2S_3 crystal samples, 5cc CH_3COOH solution was taken in beaker. With the help of burette, Na_2SiO_3 Solution was added drop by drop in beaker slowly with continuous stirring. Measure pH values of solution until it acquired the proper pH value. If pH value indicate 4.4 then. The 5 ml H_2S inner a reagent was added in gel solution. Then this mixture was poured in the test tubes of length 15 cm and 2.5 cm diameter. To take a precaution, open test tubes were closed by cotton plug. It would take 3 days for the gel to set. It kept as it is for two more days for ageing of gel and then poured the second reagent solution of $BiCl_3$ of 0.5M and same volume of 0.05M $MnCl_2$ on to the upper surface of set gel medium. The second reagent mixture solution was poured gradually alongside of test tube wall. When diffused outer reactant solution chemically reacts with inner reactant solution then growing procedure of crystals was start. After addition of the second chemical reactant, nucleation was observed within twenty-four hours. Shown in figure1.different shape and sized crystals were obtained as shown in figure 2.The colours of grown crystal were observed as creamy yellow colour. All experiments, Growth of crystals process obtain at room temperature. In gel medium, due the chemical reaction between Bismuth Chloride, Manganese Chloride and H_2S water solution we get different shape Manganese -doped Bismuth tri sulphide crystals

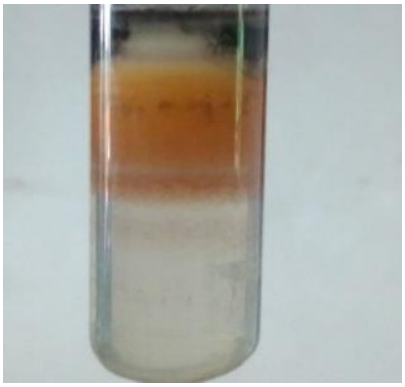

Figure 1 Mn doped crystals of Bismuth tri sulphide inside the test tubes

Figure 2 Manganese doped crystals of Bismuth tri sulphide outside the test tubes

III. RESULTS AND DISCUSSION

A. X-ray diffraction (XRD)

The XRD pattern of grown crystal power is as shown in figure 3. The observed X-ray diffraction patterns for Bismuth tri sulphide crystals doped by 0.05M Mn ions and the calculated (h k l) and d values, which were found to be in good agreement with the JCPDS data (Card no17- 0320). The crystal structures of Mn doped Bismuth tri sulphide is determined to be orthorhombic structure. Dopant has not changed the structure of the parent crystals. The grain size of the grown crystals was derived using x-ray diffraction line broadening analysis based on the Scherer formula. The breadth of the diffraction line was measured by the method of full width at half maximum. From XRD data for different parameters it was found that grain size of the undoped Bismuth tri sulphide

crystals increased on doping 0.05M of Mn and on subsequent doping shows an increasing tendency in grain size. Variations in lattice parameters, cell volumes, and intensity and diffraction angle of peaks attributes to the dopant in the crystal.

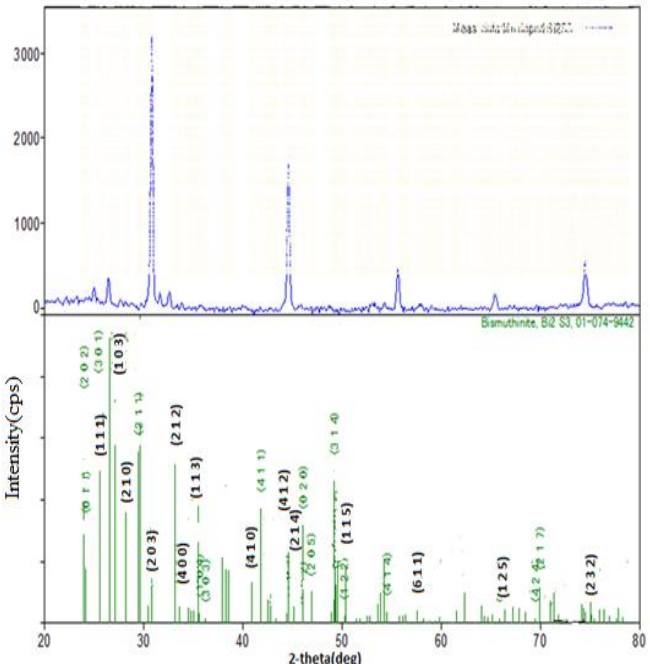

Figure 3 XRD Patterns of the crystals of Bismuth tri-sulphide doped by Mn for 0.05M

. By using Scherer's formula, the crystallite size Calculated as D = 37.63nm

From XRD data and quadratic relation lattice Parameters evaluated as a= 10.86Å, b= 4.06 Å and c = 10.22 Å i.e. a ≠ b ≠ c.

In Orthorhombic crystal structure the length of unit cells are different and $\alpha = \gamma = \beta = 90^0$.

B. Fourier transforms infrared spectroscopy:

Figure 4.Shows The FTIR Spectra of Mn doped Bismuth tri sulphide crystal. The spectrum was obtaining using SHIMADZU IRAffinity-1 cm^{-1} at Department of Chemistry, M.J.College Jalgaon.

The FTIR analysis technique used to obtain the information about the chemical bonds in molecules and provides information based chemical composition of sample. *FTIR absorption peaks and molecular set vibration are related. As per to infrared spectra theory, Molecular set vibration and characteristic absorption bands were assign are related In* FT-IR, light (covering the whole frequency range 4000cm^{-1}-400cm^{-1}) is split into two beams. Either one beam is passed through the sample or both the beam are passed, but one beam is made to travel longer path than the other does. Due to recombination of two beams produces an inference pattern that is the sum of all the inference patterns created by each wavelength in the beam. By systematically changing the differences in the two paths, the inference patterns change to produce a detected signal varying with optical path difference.

However, Fourier transformation of the interfero - gram converts it into plot of % transmittance against wave number.

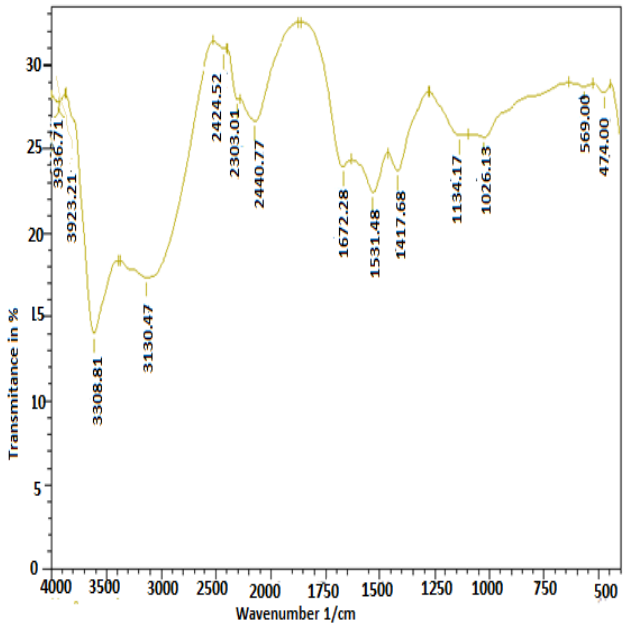

Figure 4 FT-IR spectra of Mn doped Bismuth tri-sulphide crystals

The FTIR Spectrum for a chemical compound is single characteristics, which shows the chemical bonding geometry. An important use of FTIR is to identify unknown compound and identify structure of unknown compound. Figure 4. Shows The FTIR Spectra of Mn-Bismuth tri sulphide

Using Fourier Transform Infrared Spectrometer in the range 500 cm^{-1} to 4000 cm^{-1}. The observed

spectrum of grown crystal is shown in figure 4. The bonds in the range 3500 to 3200cm^{-1} recognized to a symmetric O-H stretching of water molecules. The C-C bond stretching appears in range 2000 to 2300 cm^{-1}. It is seen that the peak at 2140.77 cm^{-1} can be assigned to C≡C bonds stretching. The sharp band present at 2424.52 cm^{-1} can be recognized C–C bond stretching. The band 1672.28 cm^{-1} can be attributed to C=C stretching of Alkyl group. The band appearing at 1531.48 cm^{-1} can be attributed N-O stretching vibration presence in nitro compounds. The band 1417.68 cm^{-1} can be attributed to bending frequency C - C group. The peaks at 1134.17 and 1026.13 cm^{-1} can be assigned to C-O stretching vibration bonding. The peak 569 cm^{-1} can be attributed Halogen compounds bond. The peak 474 cm^{-1} assigned to Bi-S bonding.

The other extra bands observed in the spectra are may be due to inclusion of sodium Meta silicate in the grown crystal.

C. Thermogravimetric analysis (TGA)

The TGA curve of Manganese doped Bismuth tri sulphide crystal was subjected to study the weight loss measured using TGA method. The TGA curve of grown crystals is as shown in figure 5.

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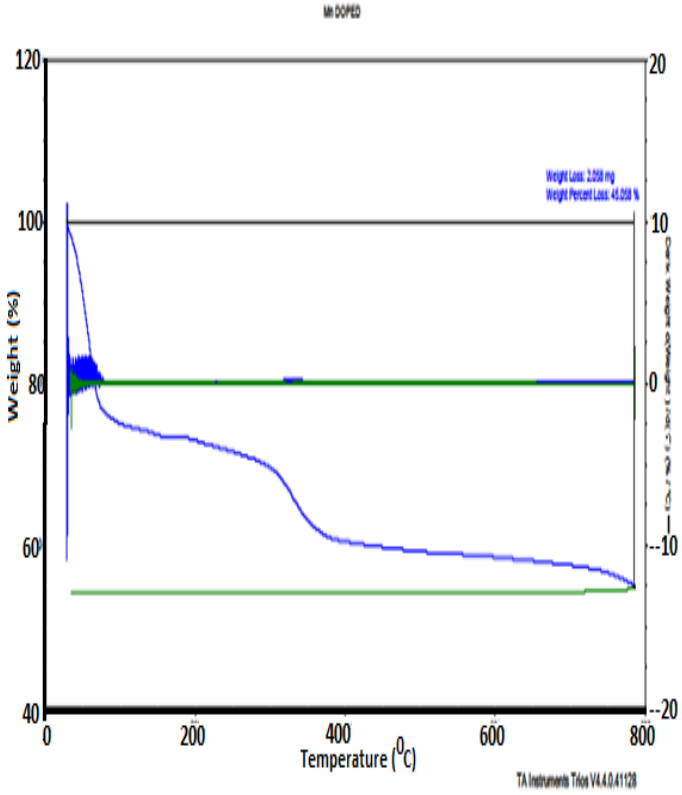

Figure 5. TGA curve of Mn doped Bi_2S_3 crystals

Decomposition of the first stage occurs in 28.32 to 52.20 ^{0}C temperature range; observe weight loss of 10.82 % in this range agreed with calculated weight loss 10.94 %. This weight loss is attributed to loss of [$2H_2O$ +$2SO_2$] and decomposition is in continuous manner.

Decomposition of the second stage occurs in 52.20 to 221.69 ^{0}C temperature range in which observed weight loss of 16.91 % nearly agree with calculated weight loss 17.08 %. Here observed weight loss appear less as compared with calculated. It can be attributed to incomplete decomposition of Bi_2S_3. This weight loss is attributed to loss of [$4SO_2$] and decomposition is in continuous manner. The remaining product finally turns into residue $MnBi_2O_3$ (Mn Bismuth Oxide) is conformed at 790.112^{0}C.The observed residue weight is 72.27 %. This is nearly agreement with calculated residual weight 69.55%.This confirms presents of manganese and Bismuth in the grown crystals.

Observed Weight Loss: -

1. 28.32 to 52.20 ⟶ 100.117 – 89.297 = 10.82 %
2. 5220 to 221.69 ⟶ 89.297 - 72.387= 16.910 %

$[MnBi_2S_3.H_2O] + 9x(O_2) \longrightarrow (H_2O) + 2SO_2 \uparrow$

$+ 2\ MnBi_2O_3 + 4SO_2$

Residue ⟶ $[2MnBi_2O_3]$

Calculated Weight Loss

$2\ [MnBi_2S_3.H_2O] + 9\ x(O_2) \longrightarrow [2(SO_2) + 2(H_2O)]$

2 x 587 +18x2+ 18X16 18 x 2 + 2 x32+4x16

= 1174 +36+ 288 ⟶ = 36 + 64 + 64

=1498 [164 x 100/1498 %]

⟶ $[4SO_2]\uparrow$

= 4 x 32 + 16 x 8

= 128 + 128 = 256

256 x 100/1498 = 17.089%

Residue ⟶ $(2MnBi_2O_3)$

$(2MnBi_2O_3)$ = 2 x 55+ 4 x 209 + 6 x 16

= 110 + 836+96 = 1042

Residue weight = 1042 x 100/1498 = 69.55%

IV. CONCLUSION

By using silica gel method Manganese doped Bi_2S_3 crystals successfully grown. Single diffusion method is convenient for the growth of the Manganese-doped Bi_2S_3crystals. The Period of gel-setting period depends on the pH value of mixture acetic acid and sodiummeta- silicate solution and of sodium meta-silicate density. Grain size of the doped crystals increases with the concentration of Mn dopant. FTIR spectrum clearly indicates that the functional groups of undoped Bismuth tri Sulphides were not altered by the addition of the dopant. Presence of C=C, C-O-C, non-bonded O-H, C-H bond. TGA analysis suggest that the thermal stability of Bismuth tri- Sulphide crystal decreases due to Manganese doping

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