

Single Crystal: Bisthiourea Doped Manganese Chloride Synthesis, Growth And Characterization

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ABSTRACT: Single crystals of bisthiourea doped manganese chloride were successfully grown from an aqueous solution by slow evaporation method at room temperature. Recrystallization process was used to increase the purity of the grown crystal. The grown crystals were characterized by single crystal XRD, FT-Raman, EDAX, UV and TGA/DTA analysis. Structure and unit cell parameters were determined by single crystal XRD. Functional groups of grown crystal and their modes of vibration were identified using FT-Raman spectral analysis. Absorbance percentage of the grown crystal was studied using UV analysis. Thermo gravimetric analysis and differential thermal analysis reveal that the good thermal stability of the material.

Keywords: Crystal growth: Single Crystal XRD; FT-Raman; UV; TGA and DTA.

I. INTRODUCTION

Optical fibre is playing a major role in the emerging communication and optoelectronic technologies. In optic fibre communication, for proper transmission and reception of laser signal, communication link is very essential. Thiourea is a white crystalline solid both naturally occurring and synthetic, that is soluble in water, ammonium triocyanate and ethanol. Thiourea is an interesting inorganic matrix modifier because of its large dipole moment and its typical polar molecule^[11]. Thiourea belongs to orthorhombic crystal system. In the present work, an attempt has been made to combine bisthiourea doped manganese crystal is grown by slow evaporation technique. The bisthiourea doped manganese chloride single crystals obtained are subjected to various characterization studies such as Single crystal X-ray diffraction, UV-Visible, thermal analysis TGA/DTA and EDAX studies.

II. EXPERIMENTAL PROCEDURE

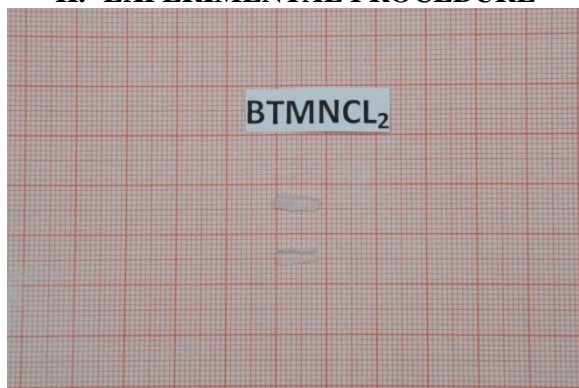
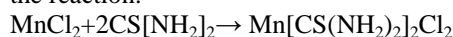


Fig.1. Photograph of BTMNC crystal.

In the present investigation bisthiourea manganese chloride (BTMNC) crystals was grown by slow evaporation solution growth of thiourea in saturated aqueous solution with manganese chloride in the molar ratio 2:1 as per the reaction:



Colourless crystals were harvested in 35days (Fig. 1).

III. RESULTS AND DISCUSSION

3.1. Single Crystal X-ray diffraction analysis

The unit cell parameters of the grown crystal are confirmed using the ENRAF NONIOUS CAD4 X-ray diffractometer equipped with MO K α ($\lambda = 0.701073$) radiation. Bisthiourea doped DSHP belongs to orthorhombic system and its cell parameters are $a = 5.48 \text{ \AA}$, $b = 7.68 \text{ \AA}$, $c = 8.57 \text{ \AA}$, and volume $V = 361 (\text{ \AA})^3$.

3.2. Ultraviolet Visible spectral analysis

The optical absorption spectrum of bisthiourea doped manganese chloride crystal was recorded in the wavelength range from 200 nm to 800 nm using Varian Cary- 5E UV -Vis -NIR Spectrophotometer and shown in following fig 2. The optical absorption spectrum of the grown crystal shows a low absorption in the entire visible region, there is no absorbance in the wavelength range from 286 nm to 800 nm. UV-Visible spectral studies carried out to determine the lower cut-off wavelength of the grown crystal at 268 nm. The direct optical band gap is determined from the optical absorption studies and it is determined to be 4.631 eV. This makes these crystals suitable for UV tunable laser (optoelectronic device) and SHG device applications^[1].

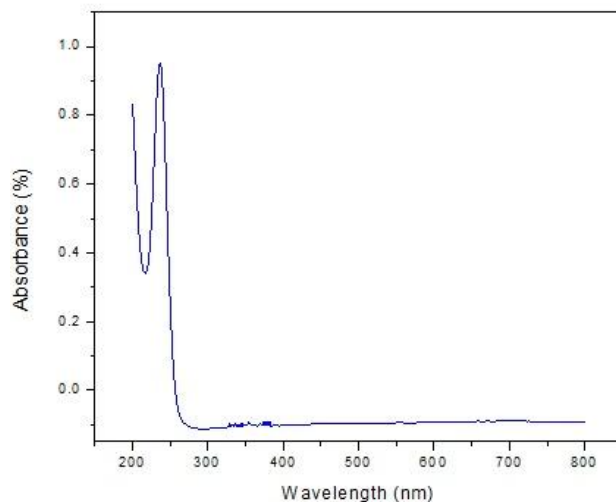


Fig.2. UV-Visible absorbance spectrum of bisthiourea doped manganese chloride crystal.

3.3. Thermal analysis

The thermal stability of bisthiourea doped manganese chloride was studied by thermogravimetric analysis (TGA) and differential thermal analysis (DTA) using CNST thermal analyser in the temperature range -50°C to 500°C heating rate $20^{\circ}\text{C}/\text{min}$ in the nitrogen atmosphere. Fig.3. shows the TGA and DTA curves of the grown crystal. From the TGA curves two transitions are observed. The first and second transitions occur over the range from 187.87°C to 251.13°C and 251.13°C to 316.82°C due to melting of the material. This shows that the material is stable upto 187.87°C . From the DTA curve two endothermic peaks were obtained. The first endothermic peak at 183.40°C in the DTA curve due to liberation of thiourea and second endothermic peak at 228.17°C in the DTA curve is due to the liberation of manganese chloride. It found that at different stages, various gaseous fractions like CO_2 , nitrogen are liberated, leading to the bulk decomposition of the compound before 453°C ^[2].

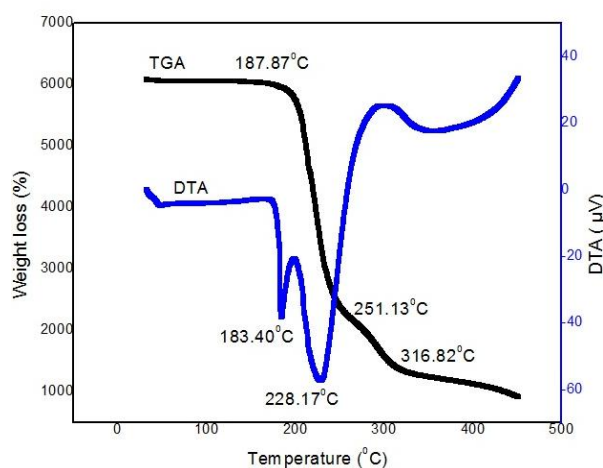


Fig.3. The TGA/DTA curve of grown crystal.

3.4. EDAX analysis

The energy dispersive analysis carried out on BTMNC crystal was used to detect the real concentration percent in the in the sample. The EDAX spectrum of bisthiourea doped manganese chloride crystal has the peaks attributed to the C, S, N, O and Mn at different energies is depicted in fig.4. Table 1 shows the weight percentage of the elements present in the sample.

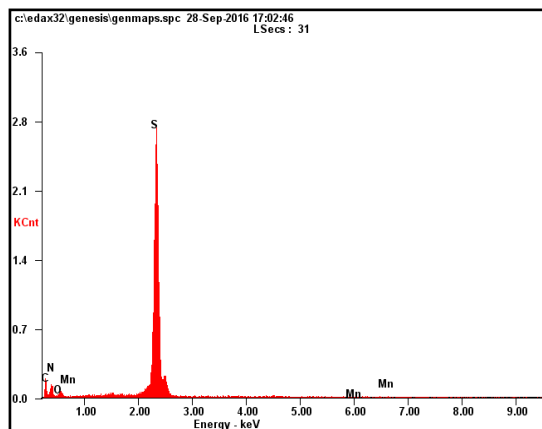


Fig.4. EDAX spectrum.

<i>Element</i>	<i>Wt%</i>	<i>At%</i>
<i>CK</i>	33.11	46.06
<i>NK</i>	25.22	30.08
<i>OK</i>	04.20	04.39
<i>SK</i>	37.26	19.41
<i>MnK</i>	00.21	00.06
<i>Matrix</i>	Correction	ZAF

TABLE.1.

IV. CONCLUSION

Single crystals of bisthiourea doped manganese chloride were grown from the aqueous solution by slow evaporation method at room temperature. The cell parameters of the grown crystals are $a = 5.48 \text{ \AA}$, $b = 7.68 \text{ \AA}$, $c = 8.57 \text{ \AA}$, and volume $V = 361 (\text{ \AA})^3$. The grown crystal belongs to orthorhombic structure. The elemental composition was determined by EDAX studies, which show that the grown crystal is of good quality. UV-Visible absorption spectrum carried out that lower cut-off wavelength of the grown crystal at 268 nm. Bisthiourea doped manganese chloride crystal is found to be thermally stable upto 187.87 °C.

REFERENCES

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