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GROWTH AND CHARACTERIZATION OF DL-NOR LEUCINE MAGNESIUM SULPHATE CRYSTALS

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Abstract :

A new semi organic crystal of DL-nor Leucine Magnesium Sulphate crystal has been grown by slow evaporation technique. The X ray diffraction peaks confirmed the presence of the blend compound in the crystal. FTIR spectrum exposed the functional groups of the grown crystals. The crystal has very good optical absorption and transmission in UV-Vis region. The surface morphology was analyzed by SEM analysis. The elemental analysis showed the presence of atoms in the compound crystal. The thermal analysis revealed the thermal stability of the crystal. The crystal showed non linear property by second harmonic generation analysis.

Keywords:

XRD - X ray Diffractometer, FTIR - Fourier Transform Infrared Spectrometer, MSD – DL-Nor Leucine Magnesium Sulphate, NLO – Non Linear Optic, SEM – Scanning Electron Microscope.

1. Introduction:

Semi organic compound crystal exhibits organic based material character in which organic molecule is stoichiometrically bonded with an inorganic ion. The π conjugate network of organic system with non linear optical behavior has good absorption in the entire visible region and is transparent. An interesting class of semi organic crystal with wider application in recent research work is the amino acid family which has molecular chirality and high transparency in UV-Vis spectrum region. There are 20 kinds of amino acid. Among them DL-Nor Leucine has optically active character which combined with inorganic host of magnesium sulphate material showing

very good thermal, optical, non linear efficiency for optoelectronic material, telecommunication and frequency conversion etc., applications[1].

2. Experimental procedure :

2.1 Synthesis:

The DL-Nor Leucine mixed Magnesium Sulphate was synthesized at room temperature by dissolving magnesium sulphate and DL-Nor Leucine in deionized water in 1:1 ratio. The solution was stirred using a magnetic stirrer for 30 minutes. The synthesized salt was purified by re-crystallization process. A saturated solution was taken and filtered in a beaker. After 20 days of time, good quality transparent crystals were harvested as shown in fig.1.

Fig.1 Photograph of MSD crystals

2.2 Characterization:

Single X ray diffraction studies of the grown MSD crystals have been carried out to identify the structure and to estimate the lattice parameters using ENRAF NONIUS CAD 4 diffractometer. The Powder X ray diffraction studies of the crystals were carried out using Rigaku Ultima III XRD equipment. The optical absorption spectrum was recorded in the range 200nm-1100nm using Perkin Elmer lambda 35. The FTIR Spectrum was recorded in the range of 4000-400cm^{-1}using Perkin Elmer spectrum RX I. The surface morphology of the compound crystal was analysed by SEM analysis using TESCAN SEM-VEGA III. The presence of atoms were identified by elemental analysis. TGA and DTA studies of the crystals were carried out using NETZSCH STA 449F3 thermal analyzer . The NLO efficiency of the crystal was evaluated by Kurtz and Perry technique[2] using a Q switched high energy Nd:YAG laser (1064 nm) emitting laser pulses with radiation under 10Hz of repetition rate.

3. Result and discussion:

3.1 Single XRD studies

Single X ray diffraction studies of the grown crystal have been carried out to identify the structure and to estimate the lattice parameters using ENRAFNONIUS CAD 4 diffractometer. The crystallographic data obtained from the analysis revealed that the crystal possesses monoclinic structure. The unit cell dimensions were a=9.85 A˚, b= 4.66 A˚, c= 16.28 A˚, α=90˚, β=104.46˚, γ= 90˚ and cell volume was 724 $A^{\circ 3}$.

3.2 Powder XRD studies:

The measured powder XRD pattern for MSD crystal is compared with standard Joint Committee of Powder Diffraction Standard card. As can be seen four well defined peaks are identified [Fig.2] at the orientations 22.8˚, 34.17˚, 28.4˚, 17.2˚. These peaks of the title compound crystal are clearly observed from the pattern (Fig.2) indicating the presence of magnesium sulphate and DL-Nor Leucine in the compound crystal MSD. The observed peaks are in fair agreement with JCPDS data (74-1364, 05-0072). It can be noticed that highly crystalline material with strong orientation is reflected by the strong peaks at 22.8˚ (101), 34.17˚ (112), 28.4˚ (120), confined the presence of magnesium sulphate. Peak at 17.2˚ shows the presence of amino acid leucine group [3]. The sharp peaks show that the compound crystal MSD has very good purity and crystallinity nature.

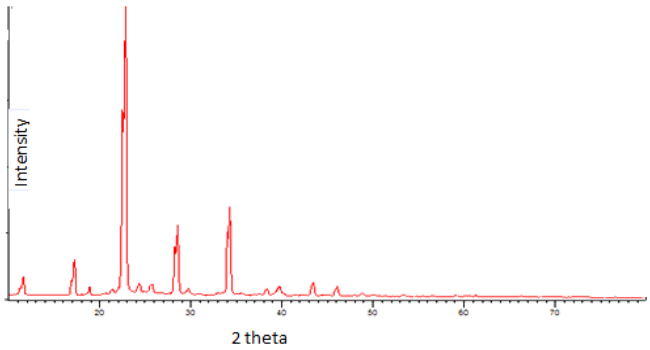

Fig. 2 Powder XRD of MSD crystal

3.3 FTIR studies:

The infrared spectrum of the grown compound crystal has been taken in the range of 4000-400cm^{-1}. The sample is made as a pellet by using KBr. The FTIR spectrum of DL Nor Leucine magnesium sulphate crystal (MSD) is shown in fig.3. The spectrum shows the presence of all the functional groups in MSD crystal. In the FT-IR spectrum [Fig.3] of grown crystal, the lower peaks are observed according to the wave number ranging from 400 - 4000 cm^{-1}. The presence of functional group in the crystal for the corresponding assignment is clear. The OH and NH stretching vibrational bands are observed at 3937.8 cm^{-1} and 3760.75 cm^{-1}. The CH stretching band is observed at 2938.78 cm^{-1} and 2864.70 cm^{-1} showing medium absorption. The overtone of

CH bending due to Fermi resonance is observed at 2671.89cm^{-1}, whereas peak at 2316.54cm^{-1} shows the stretching between halide group. Peak at 2095.47 cm^{-1} shows the anti symmetric stretching among nitrogen atoms. The c=c stretching shows the medium absorption at 1655.02 cm^{-1}. The peaks at 1517.28 cm^{-1} and 1340.9cm^{-1} show the N-O anti symmetric and symmetric stretching behavior of atoms. They also show the strong absorption due to S=O. NH bending with medium absorption observed at 1582.54 cm^{-1}. Peaks at 1288.7cm^{-1} and 1239.5 cm^{-1} confirm C-N and C-O stretching with strong absorption. C-H wag is observed at 1191.89 cm^{-1}. Peaks at 1157.06 cm^{-1}, 1072.2 cm^{-1} and 1119.2 cm^{-1} show C-N stretching with medium absorption. Peaks at 1458.8 cm^{-1} and 956.7 cm^{-1} are due to C-H bending behavior of atoms. O-H bending with medium absorption is confirmed at 923.2 cm^{-1}. Peaks at 797.4 cm^{-1} and 769.8 cm^{-1} are due to CH out of plane deformation. Peak at 723.58 cm^{-1} is due to CH rocking with medium absorption. SO2 deformation scissoring or CH2 twisting reflects 556.67 cm^{-1} peak. Peak at 444.06 cm^{-1} shows the branched alkanes presence with medium absorption [4-6].

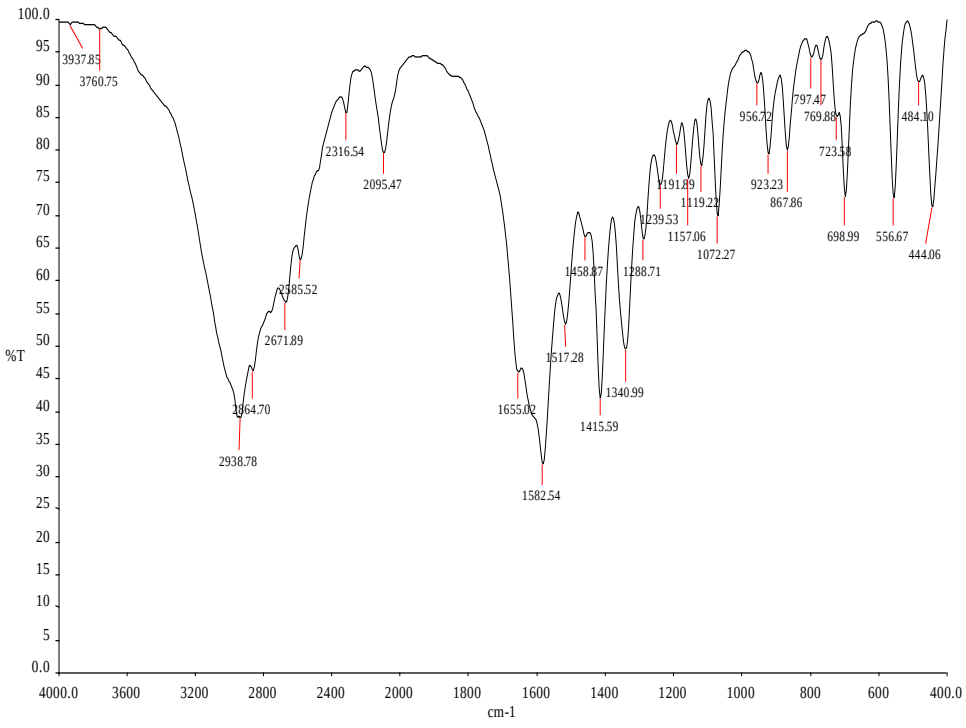

Fig.3 FTIR spectrum of MSD crystal

3.4 Optical studies:

The optical absorption studies of MSD crystal was recorded in the region between 200-1100nm, using Perkin Elmer lambda 35 spectrometer at a scanning speed of 480nm/min. The recorded spectra shown in fig.4(a) shows the absorbance found to be good in the entire visible and IR region. MSD crystal has good absorbance at 208.6nm. There is no significant absorption after this value. This shows the presence of amino acid as the advantages where the strongly conjugated bond leads to wider transparency range in the UV-Vis spectral regions fig.4(b).[7].

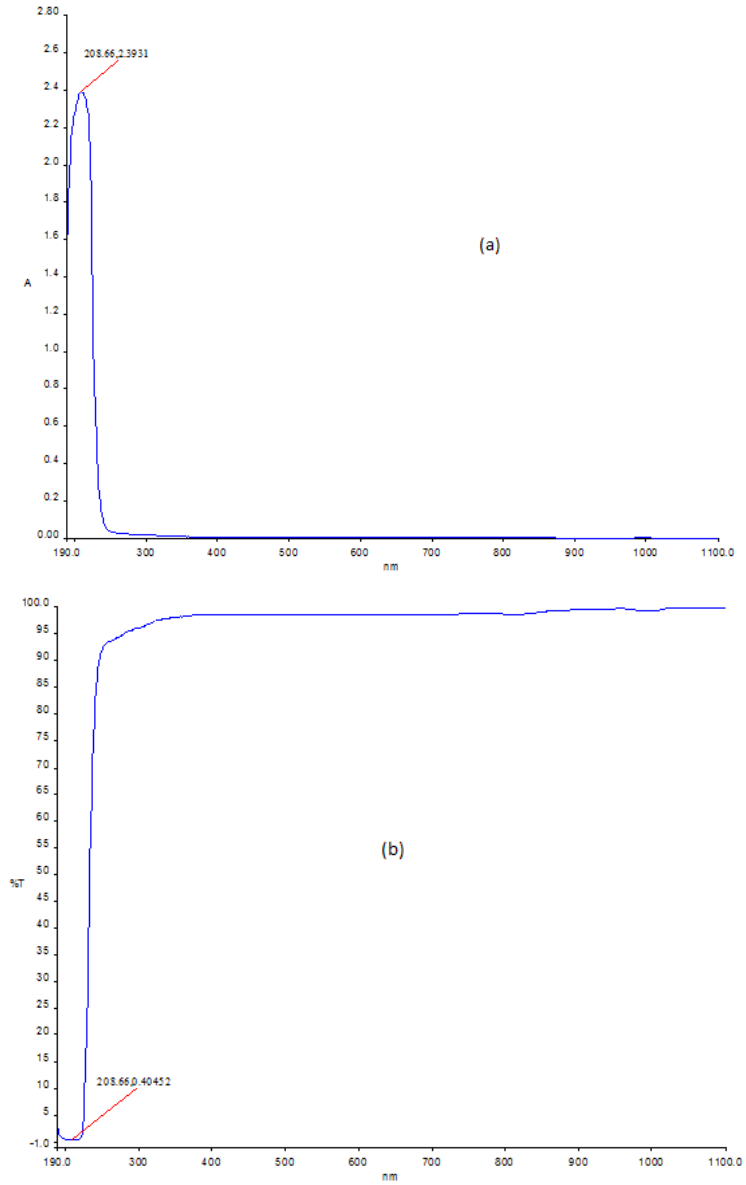

Fig.4 Optical studies of MSD crystal

3.5 Surface morphology:

A SEM micrograph of MSD crystal surface is reported in fig.5, the crystal surface is rough and denser. It has irregular columnar structure and overgrown clusters with porosity.

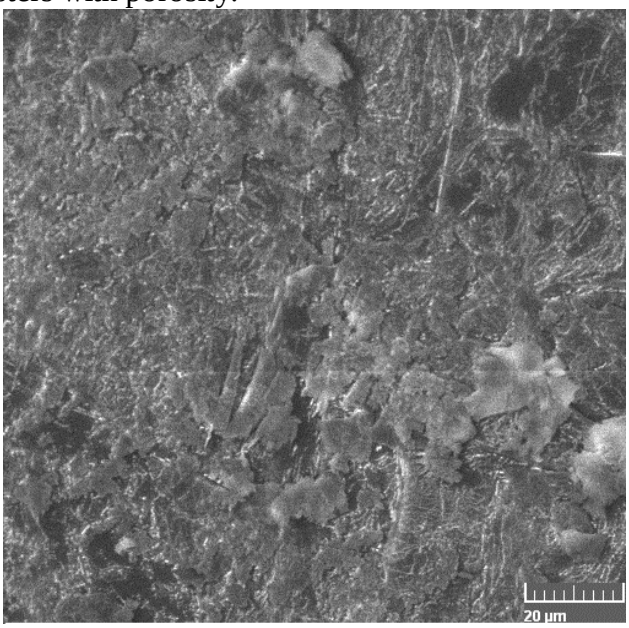

Fig.5 SEM image of MSD crystal

3.6 Elemental analysis:

Energy dispersed X ray analysis was carried out for categorizing the presence of elements in the crystal. The energy peaks obtained for different elements are reported in fig.6 for MSD crystal. The presence of Mg, O, N, S atoms are confirmed in DL-Nor Leucine magnesium sulphate crystals.

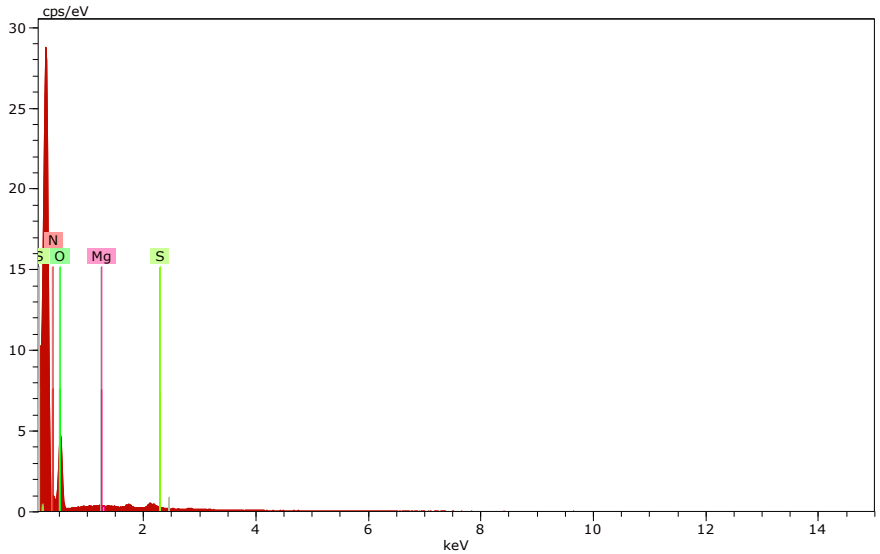

Fig.6 EDAX spectrum

3.7 Thermal analysis:

The thermal stability of the MSD crystal was investigated using Thermogravimetric analysis(TGA) and Differential thermal analysis(DTA) carried out by the instrument NETZSCHSTA449F3 at a heating rate of 20 k/min. in the range between 30˚C - 1000˚C. TGA/DTA curve of the MSD crystal is shown in fig.7. The TGA curve shows the weight loss appeared in the range between 230-320˚C. This is due to the decomposition of the sulphate present in the compound crystal. The DTA curve shows the endothermic peak appears at 315˚C. Hence, the thermal stability of MSD crystal is upto 230˚C.

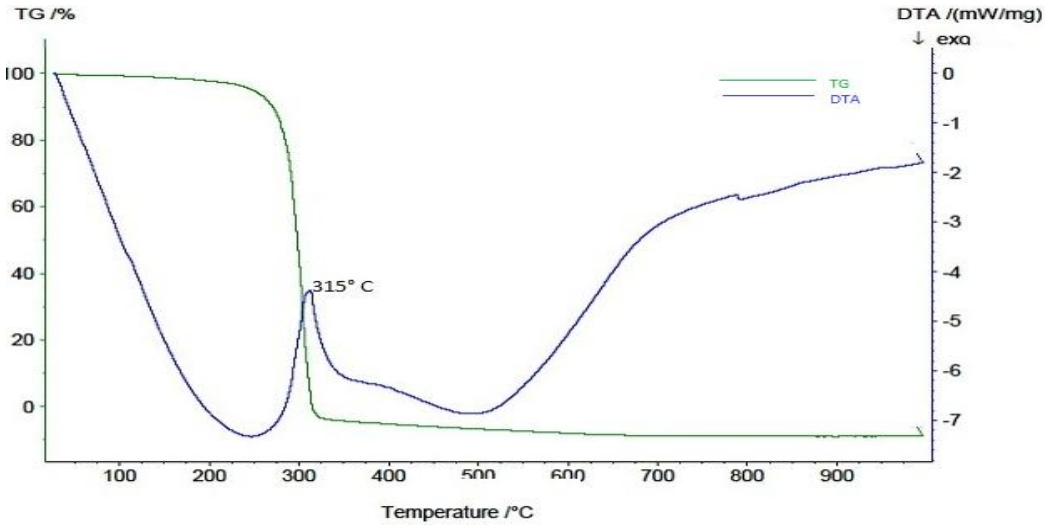

Fig.7 TG-DTA curve of MSD crystal

3.8 NLO studies:

A Non linear optical analysis was carried out by Kurtz Perry method using Q switched high energy Nd:YAG laser of Quanta ray model. A second harmonic signal was obtained at the same time as the standard KDP crystal gave a SHG signal for the same input energy. This observation shows that the SHG conversion efficiency of MSD crystal is about 1.68 times than that of the standard KDP crystal. This result suggests that the MSD crystal can be efficient for optical and photonic device applications by exhibiting its non linearity character.

4. Conclusion:

A new semi organic material of DL-Nor Leucine Magnesium Sulphate crystal (MSD) has been grown by slow evaporation technique. The X ray diffraction studies confirm the presence of elements and its structure as monoclinic in the compound crystal. FTIR spectrum reveals that the functional groups of the grown crystals are competent of the hope. The crystal has very good optical behavior in the entire visible region. The surface morphology was analyzed by SEM analysis. The elemental analysis shows the presence of atoms in the compound crystal. The purity of the crystal is confirmed from the sharpness of the endothermic peaks. The crystal has very good thermal

stability. This crystal material may be considered for many NLO applications due to the better SHG output.

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