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Growth And Characterization Of DL-Nor Leucine Magnesium Nitrate Crystals

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ABSTRACT

BACKGROUND: In the recent research, the non linear optic material has emerged its application in modulation, switching, data storage and frequency conversion for developing the technologies. Such a new semi organic non linear optic crystal DL-Nor Leucine magnesium nitrate crystal has been grown. **OBJECTIVE:** An innovative semi organic crystal of DL-Nor Leucine magnesium nitrate crystal has been grown by slow evaporation technique in a short period of time. **RESULTS:** The X ray diffraction studies inveterate the presence of the intermingled compound in the crystal and possess orthorhombic structure. 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 technique and revealed smooth microstructure with dense nature. 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 study. **CONCLUSION:** A new semi organic crystal material has wider absorption range and exhibits good thermal stability. This type of material with good non linearity nature is applicable for optoelectronic and communication systems.

INTRODUCTION

Non linear optical crystals have efficiency in UV-Vis region which are important for laser and material processing. Normally, inorganic NLO crystals are large in size, but its non linear efficiency is lower when compared to organic crystals. Hence to eradicate this problem, combined effect of organic and inorganic crystals forming semi organic crystal growth is the solution. Amino acids and their complexes belong to organic materials that have non linearity nature.(Chandrasekaran *et al*., 2012) In this work, efforts have been made to synthesize amino acid mixed inorganic complex crystals to improve chemical stability, thermal stability, nonlinear property etc., The present work showed that the amino acid DL-Nor Leucine has been selected and mixed with magnesium nitrate to form a new semi organic crystal variety for material processing applications. (Newman *et al*., 1990; Venkataramanan *et al*., 1995)

Experimental Procedure:

Synthesis and Growth:

High purity salt (99.9%) purchased from E-Merck was used for the crystal growth. The DL-Nor Leucine mixed Magnesium nitrate was synthesized at room temperature by dissolving magnesium nitrate and DL-Nor Leucine in de-ionized water in equal ratio. The solution was stirred well using a magnetic stirrer for 20 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.

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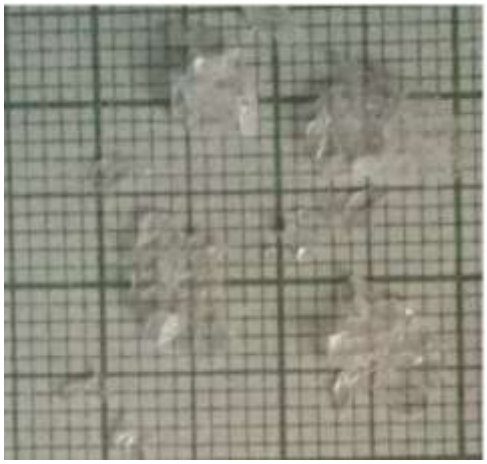

Fig. 1: Photograph of MND crystals

Characterization:

The single crystal X ray diffraction studies of the crystals were carried out 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 FTIR Spectrum was recorded in the range of 4000-400cm^{-1}using Perkin Elmer spectrum RX I. The optical studies spectrum was recorded in the range 200nm-1100nm using Perkin Elmer lambda 35 model. The surface morphology of the compound crystal was analyzed 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 tested by Kurtz and Perry technique (Kurtz *et al*, 1968) using a Q switched high energy Nd:YAG laser (1064 nm) emitting laser pulses with radiation.

RESULT AND DISCUSSION

Single XRD studies:

The structural analysis of the grown MND crystal was subjected to single crystal X-ray. Single crystal XRD study was carried out using ENRAF NONIUS CAD4 X ray diffractometer and reveals that crystal possesses orthorhombic system. The unit cell dimensions were a=5.59 A˚, b= 9.94 A˚, c= 11.87 A˚, α=β=γ= 90˚ and cell volume was 659 A˚^{3}.

Powder XRD studies:

The Powder XRD pattern for MND crystal is compared with standard Joint Committee of Powder Diffraction Standard card. As can be seen well defined peaks are identified at the orientations 16.9˚, 22.7˚, 28.3˚, 34˚, 38.2˚, 43.2˚, 45.8˚. These peaks of the compound crystal are clearly observed from the pattern(fig.2) indicating the presence of magnesium nitrate and dl nor leucine in the compound crystal MND. The observed peaks are in fair agreement with JCPDS data[JCPDS data no. 19-0765, 05-0072]. It can be perceived that highly crystalline material with strong orientation is reflected by the peak at 38.2˚, 43.2˚, 45.8˚, confined the presence of magnesium nitrate. Peaks at 16.9˚ and 22.7˚ show the presence of amino acid leucine group (Michael R.C. Williams *et al*,, 2013). The sharp peaks show that the compound crystal MND has very good purity and crystallinity nature.

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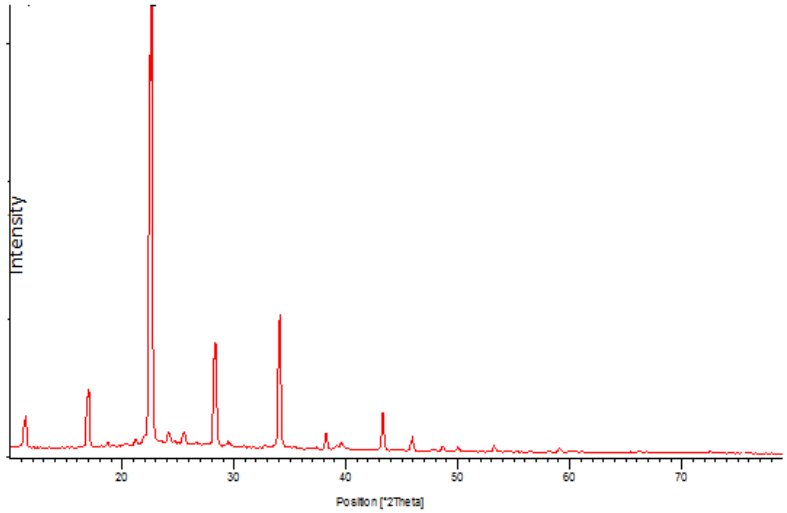

Fig. 2: Powder XRD of MND crystal

FTIR studies:

Infrared spectrum studies were carried out to expound the presence of functional groups. In the FT-IR spectrum of grown crystal of MND, the peaks are observed according to the wave number ranging from 4000 - 400 cm^{-1}. The sample is made as a pellet by using KBr. The FTIR spectrum of DL-Nor Leucine magnesium nitrate crystal (MND) is shown in fig.3. The spectrum shows the presence of all the functional groups in MND crystal. The OH and NH stretching vibrational bands are observed at 3771.76 cm^{-1} and 3427.97 cm^{-1}. The CH stretching bands are observed at 2955.11 cm^{-1} and 2935.28 cm^{-1} showing medium absorption. The overtone of CH bending due to Fermi resonance is observed at 2671.45cm^{-1}, whereas peak at 2317.76 cm^{-1} shows the stretching between halide group. Peak at 2096 cm^{-1} shows the anti symmetric stretching among nitrogen atoms. The C=C stretching shows the medium absorption at 1655.47 cm^{-1}. The peaks at 1517.34 cm^{-1} and 1339.5cm^{-1} show the N-O anti symmetric and symmetric stretching behavior of atoms. They also show the strong absorption due to S=O. N-H bending with medium absorption observed at 1583.14 cm^{-1}. Peaks at 1287.08cm^{-1} and 1238.69 cm^{-1} confirm C-N and C-O stretching with strong absorption. C-H wag is observed at 1191.8 cm^{-1}. Peaks at 1157.18 cm^{-1}, 1072.92 cm^{-1} and 1119.33 cm^{-1} show C-N stretching with medium absorption. Peak at 958.58 cm^{-1} is due to C-H bending behavior of atoms. O-H bending with medium absorption confirms at 924.18 cm^{-1}. Peaks at 795.84 cm^{-1} and 770.12 cm^{-1} are due to CH out of plane deformation. Peak at 725.48 cm^{-1} is due to CH rocking with medium absorption. Peak at 557.01 cm^{-1} may be due to SO_2 deformation scissoring or CH_2 twisting reflection. Peak at 444.06 cm^{-1} shows the branched alkanes present with medium absorption (George Socrates, 2001; Foil A. Miller *et al.*, 1952; John Coates, 2000).

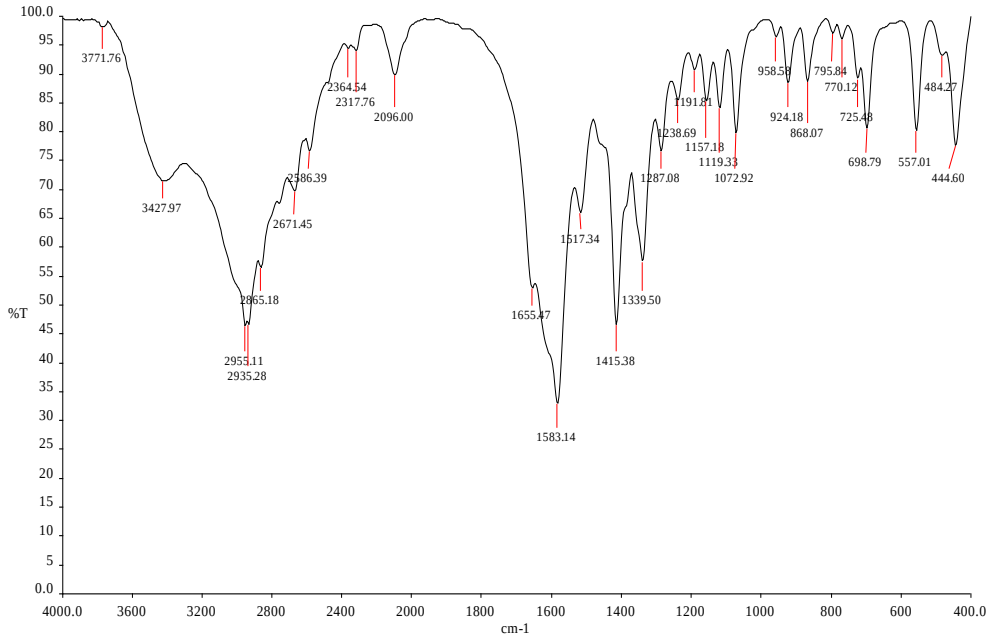

Fig. 3: FTIR spectrum of MND crystal

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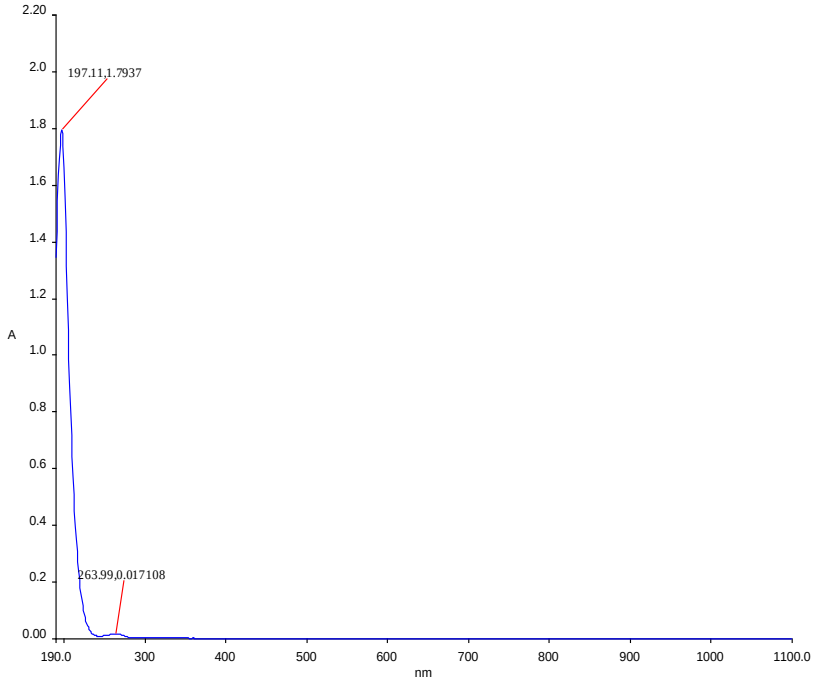
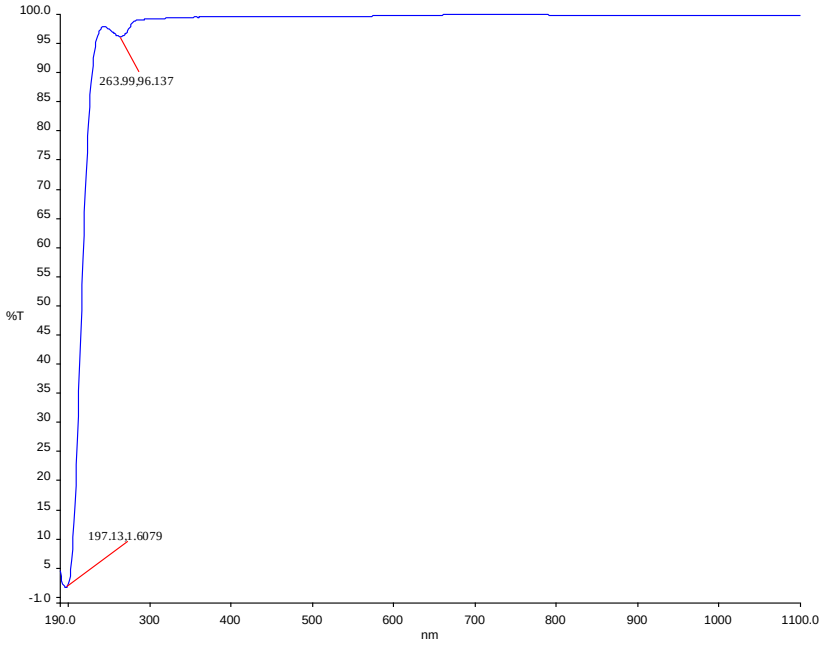
Optical studies:

The optical absorption spectra of MND crystal was recorded in the region between 200-1100nm, using Perkin Elmer Lambda 35 model spectrometer at a scanning speed of 480nm/min. The recorded spectra shown in fig.4(a) show the absorbance found to be good in the entire visible and IR region. MND crystal has good absorbance at 197nm. This lower cut off wavelength leads to wider transparency range also in the UV-Vis spectral regions, which has advantage for semi organic NLO crystal applications fig.4(b). (Ramachandraraja *et al*., 2013).



(a)



(b)

Fig. 4: Optical studies of MND crystal

Surface morphology:

A SEM micrograph of MND crystal surface is pointed out in fig.5. The crystal surface has a smooth and dense microstructure without porosity.

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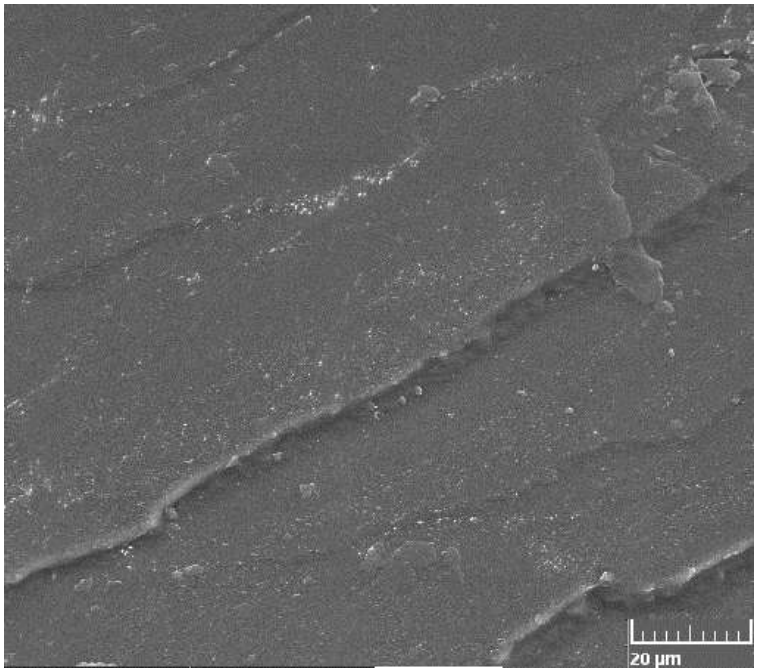

Fig. 5: SEM analysis of MND crystal

Elemental analysis:

Energy dispersed X ray analysis was carried out for categorizing the presence of elements in the MND crystal. The energy peaks obtained for different elements are given in fig.6 for the title compound crystal. The presence of magnesium, nitrogen, oxygen atoms are long-established in DL-Nor Leucine mixed magnesium nitrate crystals.

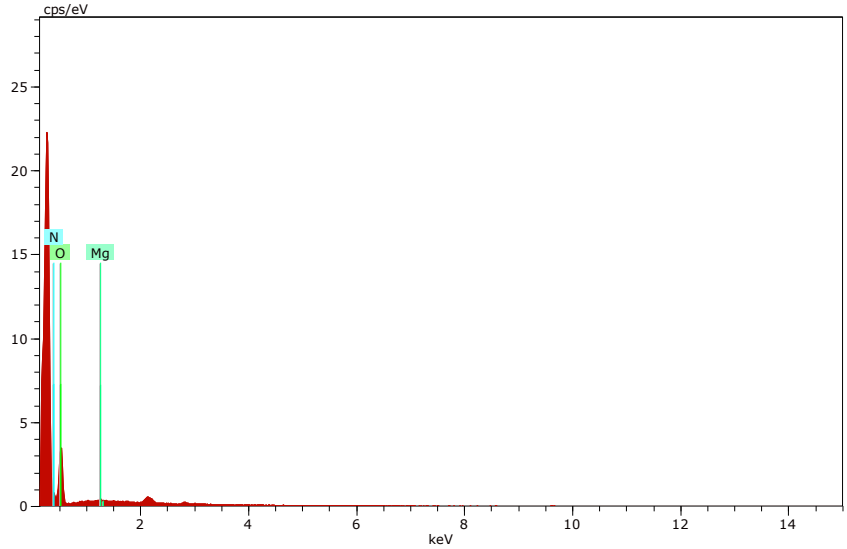

Fig. 6: EDAX spectrum

Thermal analysis:

The thermal stability of the MND crystal was investigated using Thermo gravimetric 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 material was shown in fig.7. The TGA curve shows the weight loss appeared in the range between 250-340˚C. This is due to the decomposition of the nitrate present in the compound crystal. The DTA curve shows the endothermic peak at 320˚C. Hence, the thermal stability of MND crystal was upto 250˚C.

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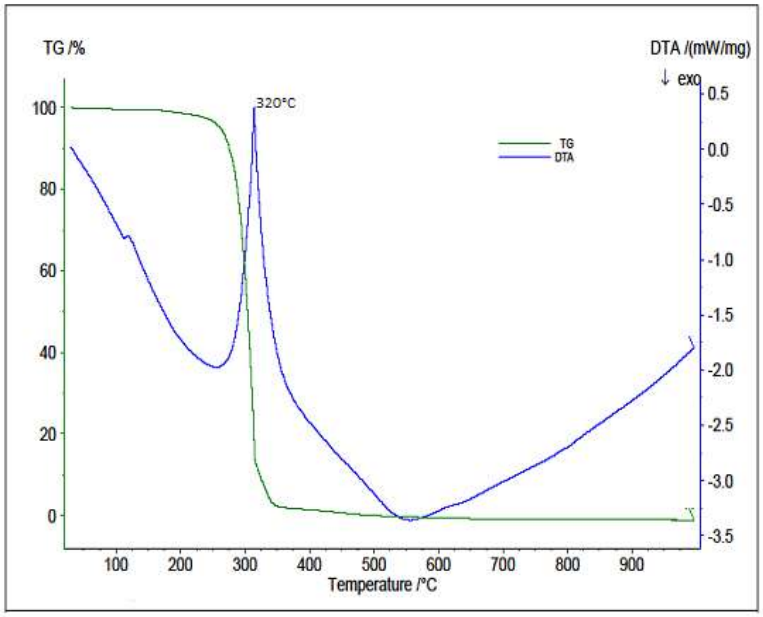

Fig. 7: TGA-DTA curve of MND crystal

NLO studies:

An energetic study has been employed to hit upon the NLO property of the compound crystal. The NLO conversion efficiency was tested using Kurtz and Perry setup. A Q-switched Nd:YAG laser beam (Quanta ray series) supplied by Spectra Physics, USA and Coherent Molectron power meter, USA, of wavelength 1064nm was used with an input power of 0.68 J. This study showed the SHG conversion efficiency of MND crystal is about 2.26 times than that of the standard KDP crystal. This result recommends that the MND crystal can be efficient for non linearity optical device applications. (Kurtz *et al.*, 1968)

Conclusion:

A new semi organic material of DL-Nor Leucine mixed magnesium nitrate crystal (MND) has been grown by slow evaporation technique. The X ray diffraction studies confirm the presence of the intermingled compound. The crystals possess orthorhombic structure. FTIR spectrum reveals that the functional groups of the grown crystals are capable of the anticipation. The crystal has very good optical absorption and transmission in the entire visible region. The surface morphology was analyzed by SEM analysis and the report showed the presence of smooth and dense micro-structural surface. The elemental analysis shows the presence of atoms in the compound crystal such as magnesium, nitrogen and oxygen atoms. The thermal analysis shows the thermal stability and purity of the crystal. The weight loss appeared in the range between 250-340˚C. This is due to the decomposition of the nitrate present in the compound crystal. The DTA curve shows the endothermic peak at 320˚C. The non linearity nature of the crystal is proved by SHG efficiency and it is 2.26 times that of standard KDP crystal. Further, the crystal material may be considered for NLO and other optoelectronic applications for future work.

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