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Investigation on the solubility of Nonlinear Optical Material (NLO) of TMACC Single crystal

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***Abstract:** In this work, we present NLO material of Tetramethyl Ammonium Cadmium Chloride (TMACC) single crystal has grown from aqueous solution by slow evaporation method at room temperature using mixed solvents of deionized water. The grown crystal was subjected to powder XRD and SEM method to identify the lattice parameters and confirm the formation of desired crystalline phase in the grown crystal. Differential scanning calorimetric (DSC) studies carried out to analysis the thermal behavior of TMACC. Photoluminescence (PL) have been carried out the spectrum of shows the blue emission of grown crystal.*

Key words: ***Powder XRD, SEM, DSC, PL.***

I. INTRODUCTION

Nonlinear optical (NLO) materials are finding a broad spectrum of real time applications such as optical computing process, optical storage technology, and laser technology and frequency conversion [1,2]. This type of materials a play an important role in the field of telecommunication, optical switching and optical processing. In view of this, it is desired to find new NLO crystals which have a shorter cut-off wavelength. High quality organic NLO crystals must possess sufficiently large NLO coefficient, transparent in UV region and high laser damage threshold power and easily grown with large dimensions [3,4]. Due to these challenging applications the growth of single crystals of optical materials is very important for the fabrication of technologically important devices. From an application point of view the great effort has been made for the growth and characterization of TMACC crystals, additives play a vital role in enhancing their physical properties [5]. In the present work we take initiation for growing Tetramethylammonium cadmium chloride crystal by slow evaporation solution growth technique at room temperature and their structural, thermal and hardness properties were reported.

II. EXPERIMENTAL

A. Purification of TMACC

The starting materials were Merck GR-grade chemical re-agents. The most essential feature of growth of high quality crystal is the purification of material. It is essential to increase the purity to be put able level before proceeding further [6]. The purity of the synthesized salt was further improved by successive recrystallization process. There crystallized crystal was used to prepare the saturated solution. In this way the impurity content of TMACC crystal was minimized.

B. Conformation Studies

Proper Understanding of the grown crystal and for using them for technological application they have to be characterized accurately. The grown crystals were subjected to single crystal X-ray diffraction using Enraf Nonius (AD-4) to determine the lattice parameter. The elements present in the crystal were confirmed by SEM analysis **.** Thermal analyses were determined by DSC studies with heating rate from room temperature to 700°C using Perkin Elmer and Mettler Toledo apparatus.

III. RESULTS AND DISCUSSION

A. Powder X-ray diffraction

Tetramethyl ammonium cadmium chloride single crystal belongs to hexagonal system at room temperature with space group $P6_3/m$. The lattice parameter values were found to be a=9.09 Å, b=9.09 Å, c=6.68 Å. The prepared

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sample was scanned over the range of 10^0 C to 80 ^{0} at a scan rate of 2^0 min^{-1}. The following derivation of the relationship between diffracted intensity and absorption,

$$I_x = K\ V/\ \mu$$

I_x is the measured intensity of diffracted line of crystalline compound of sample, V is the volume fraction of the component, μ is absorption coefficient of the specimen and k is a constant for any particular line of the particular material [6,7]. The powder X-ray diffraction of TMACC crystal shown in Fig(1).

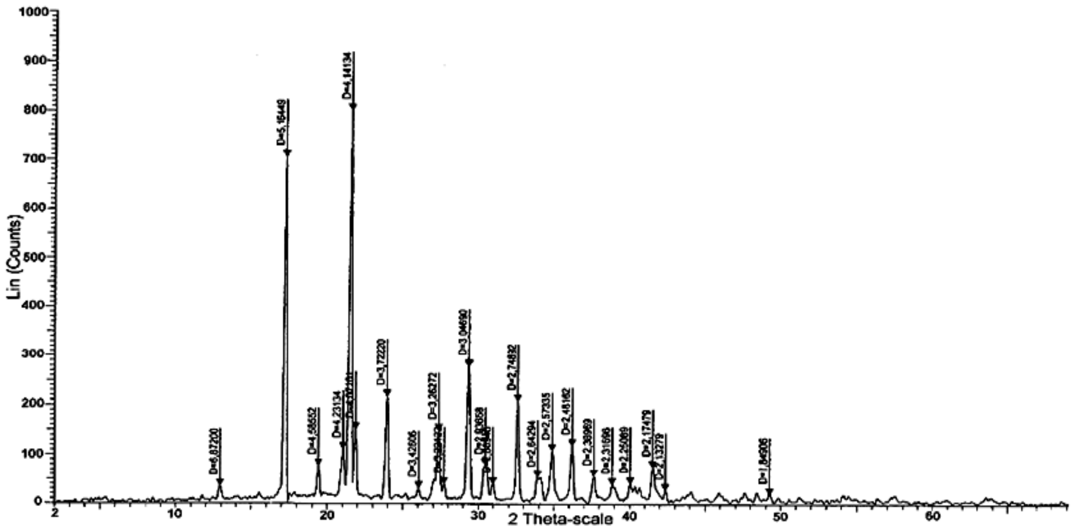

Fig. 1 Powder XRD pattern of TMACC 2:1 ratio crystal

B. Scanning Electron Microscope(SEM) Analysis

In SEM, the surface of solid sample is scanned in a raster pattern with a beam of energetic electrons. The back scattered and secondary electrons produced from the surface by the interaction of the primary electron beam with loosely bound electrons of the surface atoms serve as the basis of SEM. When the electron beam scans the specimen surface, there will be a change in the secondary electron emission according to the surface texture. The SEM images of the crystals of pure TMACC shown in Fig(2).

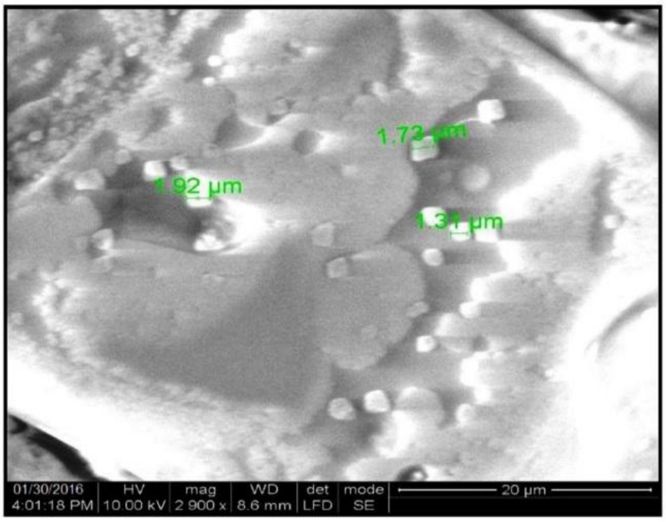

Fig. 2 SEM analysis of TMACC crystal

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C. Thermal Studies

Differential scanning calorimetric (DSC) studies of TMACC has been carried out in the temperature range 30 to 400K at the rate of 10 K/min. The DSC thermo gram of the TMA-$CdCl_3$ crystal shown in Fig (3). No peak corresponding to structural phase transition was observed between 30 and 94 K. A very broad exothermic peak was observed at 128K. The sharpness of the peaks denotes a good crystallinity of the grown crystal. The following decomposition pattern is proposed for the observed weight loss [8,9-10].

Step I

440 to 620 K

$[N(CH_3)_4]$ **$CdCl_3$** ---------------→ **CdCl** + $N(CH_3)_4$ Cl

Formula weight: 293.5   184.97 + 109.17(36%)

Step II

620 to 750 K

$CdCl_2$ ---------------→ **Cd î + Cl î + Cl î** (Complete decomposition)

Formula weight: 184.97 + 35.17 + 35.17 (64% weight loss)

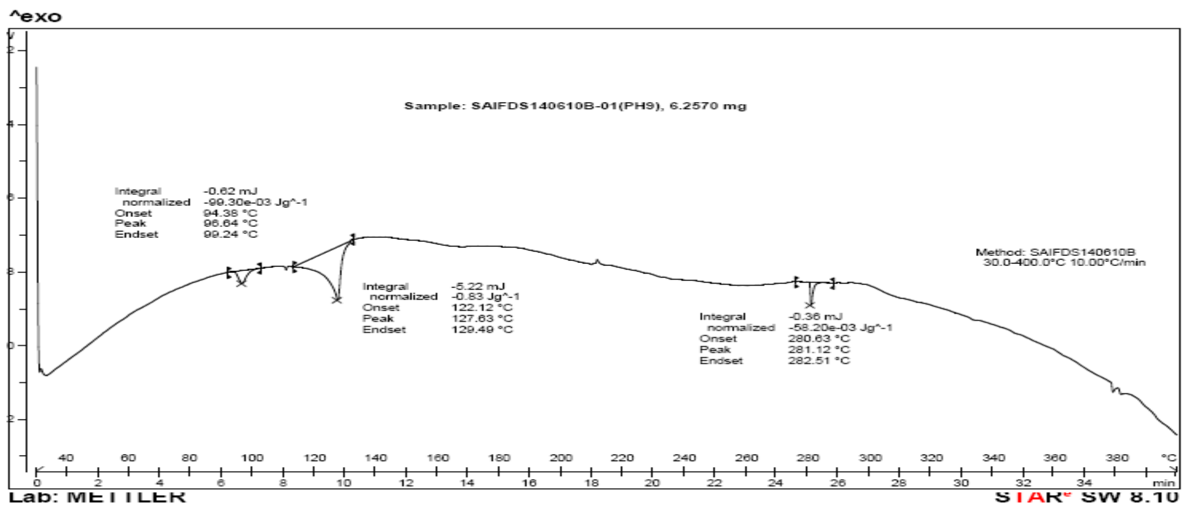

Fig.3. DSC Thermo gram of TMACC

D. PL Studies

PL study of the TMACC crystals was carried out at room temperature and is shown in Fig (4). To detect the lower concentration of defects, the photo luminous studies are performed rather than optical absorption. This is a mechanism where the impurity an absorption of light gives rise to bound excited state from which it returns to its ground state abiding in the analysis of color center creation mechanism [11,12]. The PL emission spectrum of TMACC samples was recorded in the range 400-700 nm. The spectrum emitted by the radioactive recombination of photon generated minority carrier, is a direct way to measure the band gap energy. However, large amount of impurities induces a large free carrier density in the bands. Consequently, a different carrier interaction causes remarkable modification of the line shape and spectral energy of the feature [12,13]. The peak at 440 nm shows the blue emission from the crystal due to the radioactive recombination of photo generated minority carriers [14,15]. It is used to measure the band gap energy and is found to be 2.83 eV.

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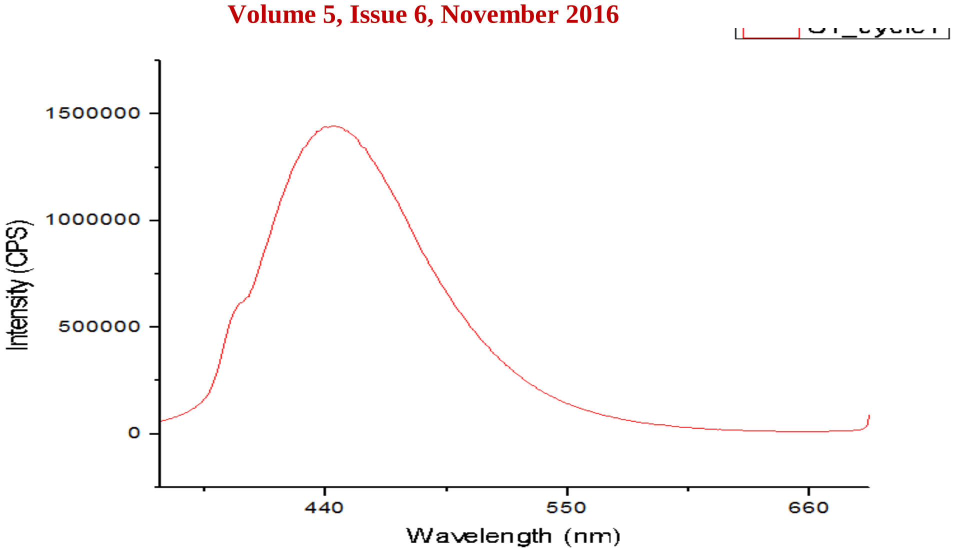

Fig 4 PL study of TMACC

IV. CONCLUSION

The single crystal XRD studies prove that the grown TMACC crystals belong to monoclinic crystal system. The particle size of the grown crystal is characterized by Powder XRD analysis. Transparent and semi-transparent crystals of well-defined morphologies were obtained. From SEM analysis, it is concluded that there is a formation of voids of different shapes on the surface of the grown crystals. Differential scanning calorimetric (DSC) studies of TMACC has been carried out in the temperature range 30 to 400K at the rate of 10 K/min to find out the thermal stability of grown crystal. The Photoluminescence spectrum showed the blue fluorescence emission. The NLO properties of TMACC crystal used in optical storage technology, and laser technology.

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