COMPARATIVE STUDY OF DCTZ, BCTZ AND BCBT SINGLE CRYSTALS

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Abstract: The application of single crystals in the newest technology is evident from the recent developments in semiconductors, polarizers, transudes and infrared detectors etc Single crystals of DCTZ, BCTZ and BCBT were grown via slow evaporation technique .The physicochemical properties of the grown crystals were analyzed. The EDS studies carried out on the grown samples , surface features were studied through SEM analysis and Etch pattern analysis.

Keywords: EDS, SEM, ETCH.

I. Introduction: The organo-metallic thiocyanate complexes are appropriate for recognizing blue-violet light by frequency doubling of laser radiation. The experiments conducted by the eminent scientists all over the world strongly favour the possible use of this class of materials for various non linear optical applications and photonics device fabrications. It is fascinating to note that the metal thiocynate complex family crystalline compounds suggest a mixture of molecular structures in turn these complexes are capable of efficient frequency conversion of IR radiation to ultraviolet wavelength. In meta thiocynate complexes, the thiocynate has the capability to interconnect metal ions with its own donors S and N .

Previously, Zhang and Zelmon have reported the growth of a new NLO crystal $[(18C_6)K][Cd(SCN)_3]$; KCCTC, which is transparent from 220 to 3300 nm and shows second harmonic generation (SHG). Recently, Zhang and Shou] and Zhang and Huang have reported the growth and characterization aspects and powder SHG of $[(18C6)Li][Cd(SCN)_3]$; CLTC,

respectively. The thermal and optical properties of ACCTC $[(NH_4)]$ $[Cd(NCS)_3].C_{12}H_{24}O_6$ was reported by V. Ramesh and K. Rajarajan .

In the present work, The structural details of DCTZ, BCTZ and BCBT were already reported. However, the characterization aspects of surface studies were not reported elsewhere. Hence in the present case, efforts were made to synthesize and grow the good quality single crystals and thereby the EDS, SEM and ETCHING studies were carried out on the sample and reported for the first time

II. Experimental Procedure: The title compound DCTZ, $[(NH_4)_2]$ $[Zn(NCS)_4].2C_{12}H_{24}O6.H_2O$, the result of the reaction of ammonium thiocyanate, 18-crown-6 and zinc(II) chloride in aqueous solution, exhibits and unusual supramolecular structure. Bis[(18-Crown-6)Potassium] [Tetrakis(isothiocyanato)Zinc(II)]; $[18C_6(K)]_2[Zn(SCN)_4]$; BCTZ single crysal was grown via slow evapouration technique. BCBT compound was synthesized by a mixture of 18-crown-6, Pottassium thiocynate and Barium Chloride by slow evaporation method.

III. Results and Discussion:

Energy Dispersion Spectrum (EDS) Analysis of DCTZ ,BCTZ and BCBT

The stoichiometry compositon of a grown crystal of DCTZ,BCTZ and BCBT was found by Energy Dipersice Spectroscopy analusis using XRD NCCA 200 System connected to Leo –stero scan 440 scanning electron microscope.The EDS[1-4] patterns of DCTZ ,BCTZ and BCBT are shown if fig (2,3) which gives information about the presence of various elements in weight percentage.From the graph it is clear that the grown crystals DCTZ ,BCTZ consists of carbon,oxygen,sulpher,zinc and clorine atoms present in weight percentage. BCBT EDS spectrum shows that the title compound consists of Barium,Oxygen Sulpher Pottassium and Chlorine atoms. This information clearly indicates the molecular formulae of the grown crystal DCTZ and BCTZ .The quantitative results of the weight% of the grown samples are also indicate in the below graphs.

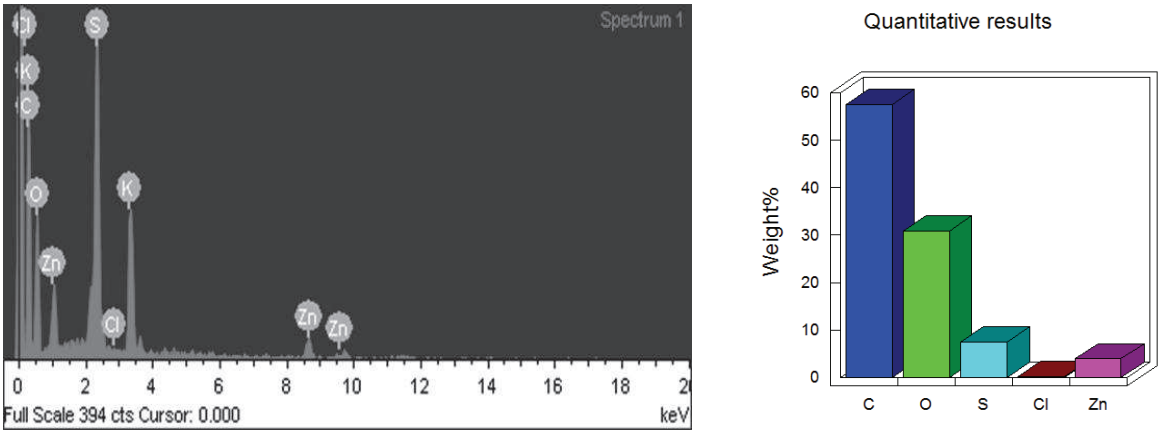

Fig: 1: EDS Spectrum and Quantitative analysis of DCTZ

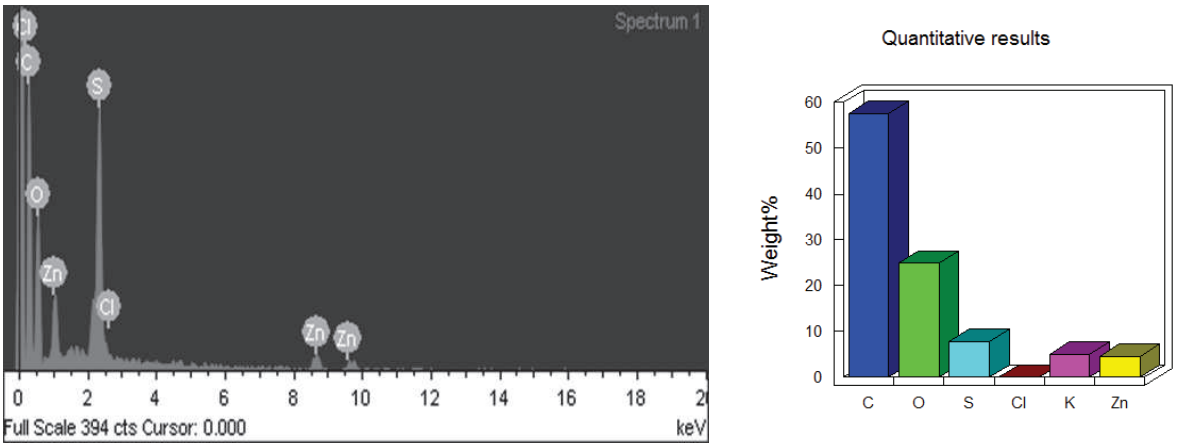

Fig: 2: EDS Spectrum and Quantitative analysis of BCTZ

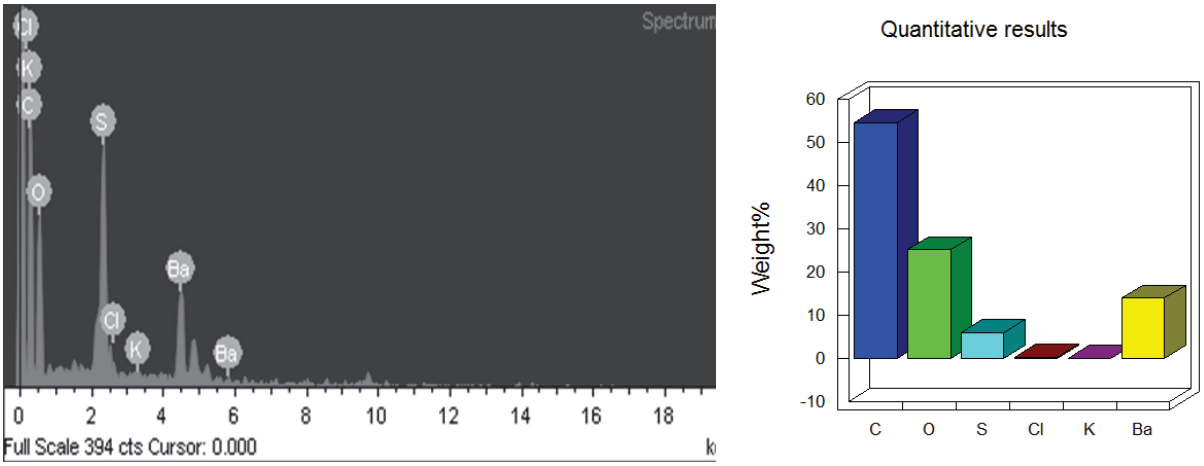

Fig: 3: EDS Spectrum and Quantitative analysis of BCBT

SEM Analysis: It is clear from the SEM micrographas of DCTZ that the crystal surface contains voids of irregular size and dendrites like growth pattern of micro crystals.The presence of valleys and cracks are predominantly seen on the surface of the crystal. The SEM micrograph taken on the plane of BCTZ and BCBT clearly shows that the crystal possess almost smooth surface and free from cracks and large size voids. Few micro crystals are viewed on magnification. The shape of the crystals are pyramydical which is actual shape of the crystal BCTZ. It is believed that the grown crystals can be subjected to such defects unless growth parameters like temperature and ph is carefully optimized[1-9].The SEM images of DCTZ,BCTZ and BCBT are shown in the figures(4,5and 6) respectively.

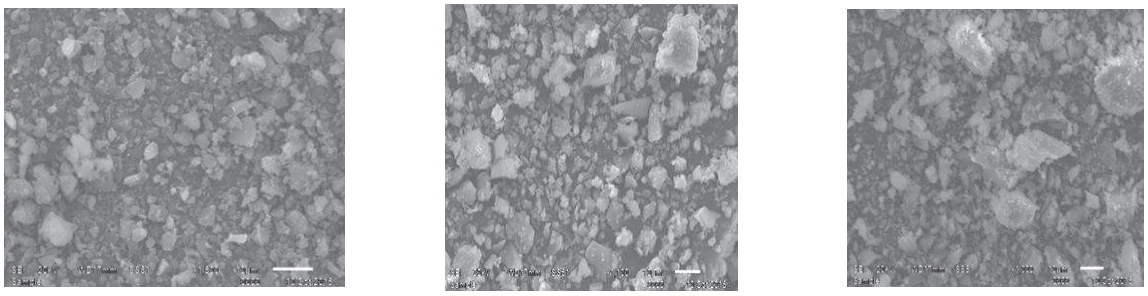

Fig:4. SEM image of DCTZ **Fig:5.** SEM image of BCTZ **Fig:6.** SEM image of BCBT

Etching Studies: The crystals with defects can destroy the mechanical and electrical properties which damage the usefulness of the crystals. The nonlinear optical properties depend upon the crystalline perfection. Etching is one of the desirable study to identify defects in the grown crystals. When the crystal is dissolved in a solvent, the reversal of the growth takes place by giving the well defined etch pits. Etching of surface using desirable solvent gives information about the surface features[10-11].

Optical microscopy was employed to study the surface features of grown crystal DCTZ, BCTZ and BCBT. The compound crystal was immersed in water for etching period of 5s, 10s . The crystals was taken out, gently desired with the tissue paper and instantly placed under the optical microscopy. For the etching periods of 5s, 10s , layer type patterns were observed. The fig (7,8 and 9) depicts the itching patterns. The etching study revealed the formation of layer pattern, which is an indicative of two dimensional mechanisms .

Fig:7. Etching pattern of DCTZ

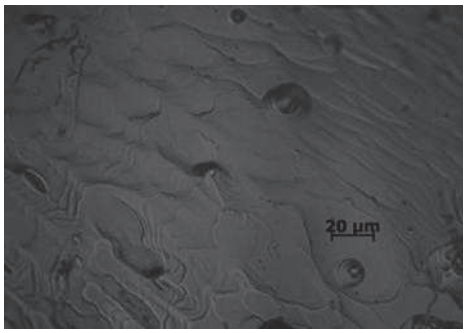

Fig:8. Etching pattern of BCBT

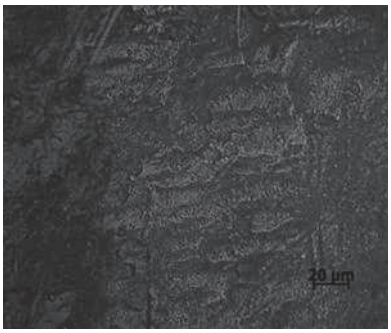

Fig:9. Etching pattern of BCTZ

IV. Conclusion: The surface studies of Organic-Inorganic single Crystals DCTZ, BCTZ and BCBT were carried out by using different analysis techniques. The SEM image of the crystal confirms the different type of pattern of the crystals. The presence of carbon, oxygen, sulphur, chlorine, zinc, potassium and barium in the grown crystal was confirmed using EDS spectral analysis. Imperfections present in a material can influence seriously the device characteristics. Hence the assessment of physical as well as the chemical imperfections in crystalline materials is essential in the field of material research. Etching techniques can be applied to understand the behaviour of dislocations and also to reveal the growth history of the crystals. DCTZ, BCTZ and BCBT Crystal surface studies, composition of the crystals were compared in this paper.

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