



## CRYSTALLITATION OF ZINC SULPHATE SINGLE CRYSTAL AND ITS CHARACTERIZATION

R.Tharanya\* P.Kayalvizhi\*

\*Assistant Professors, Department of physics, Adhiyaman arts and Science College for women, Uthangarai, Krishnanagiri.

### Abstract

Zinc sulfate ( $ZnSO_4 \cdot 7H_2O$ ) an inorganic material has been crystallized slow evaporation method.  $ZnSO_4 \cdot 7H_2O$  is highly soluble in water and the solubility is found to be increased almost linearly with the increase of temperature. At room temperature around  $32^\circ C$  the solubility was found to be 93gm/50 ml. the FT-IR spectroscopy study was performed on pure zinc sulphate crystals to identify the present of functional gropes .the grown crystal has been subjected to powder X-ray diffraction to determine the unit cell dimensions and the crystal structure. The UV-Vis spectrum showed that the material has wind optical transparency in the ultra-violet region [1].

### 1. Introduction

Zinc sulphate heptahydrate possesses wide range of application in the field of telecommunication, solar system for solar energy storage devices. The search for new frequency conversion material over the past decade has led to the discovery of many organic materials with high nonlinear susceptibilities. However their often inadequate transparency, poor optical quality and lack of robustness, low laser damage threshold and inability to grow into large size have impeded the use of single crystal in various devices recent interest is centered inorganic crystals because pure inorganic materials typically have excellent mechanical and thermal proprieties crystallization of heptahydrate sulphate material such as zinc vitriol( $ZnSO_4 \cdot 7H_2O$ ) of high purity has become an important field of research for both academic and industrial application in various areas like medical agricultural and chemical industry. The pure zinc sulphate crystals were grown at slow evaporation from aqueous solutions [1,2].

### 2. Materials and Method

Analar grade zinc sulphate and doubled distilled water were used in the crystallization process. In the first stage a mother solution of 50 ml was prepared using recrystallized salt of ( $ZnSO_4 \cdot 7H_2O$ ) and stirred well with a magnetic stirrer for 3 hours to attain saturation.the solution was carefully filtered allowed to evaporate at room temperature.the pure zinc sulphate seed crystal were prepared by the natural slow evaporation method.During natural evaporation process,the crystallizers were covered with perforated polyethylene sheet and kept in a dust free champers colorless and transparent  $ZnSO_4 \cdot 7H_2O$  crystals were harvested after a period of 4-5 weeks[1,3].

### 3. Result and Discussion

**Solubility of  $ZnSO_4 \cdot 7H_2O$ :** the solubility data at constant temperature are essential to determine the level of super saturation a small fluctuation in the temperature will effect this super saturation to grow good quality crystal.

#### Crystallization of Zinc Sulphate Single Crystal

Hence the solubility of the solute in the water solvent was determined before starting the growth process the solubility of  $ZnSO_4 \cdot 7H_2O$  has been studied in the temperature range of  $33^\circ c$  to  $34^\circ c$ .it is clearly seen that  $ZnSO_4 \cdot 7H_2O$  is highly soluble in water the solubility is found to be 93gm/50ml of water[4].

Mass growth rates of  $ZnSO_4 \cdot 7H_2O$  crystal are presented in fig 1. It was observed that the mass growth rate initially increases gently with time and then it increases rapidly ub to certain time and then it become constant Highly transparent and well faceted large size crystal with the dimension of 1.5cmx0.4cmx0.5cm were grown from aqueous salutation by slow evaporation method shown in fig 2.

#### 3.1 Infrared Spectroscopy

The FT-IR of zinc sulphate single crystal was recorded using bruker alpha spectrophotometer KBr pellet technique in the range of 4000 to 400/cm to identify the functional groups and determining the molecular structure of the grown crystal it was taken from sacred arts college, Tirupattur.

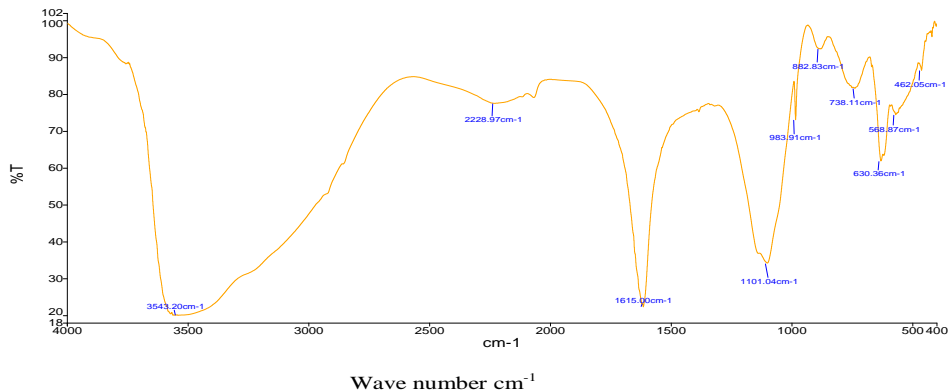


Fig 1 FT-IR zinc- sulphate crystal

The absorption due to various functional groups is shown in fig 3. The stretching vibrations of the water molecule are expected in the region 3500-3600/cm. The broad vibrational band observed at  $3543.2\text{cm}^{-1}$  is attributed to the symmetric stretching mode of water molecule. The medium broad noticed at  $1615.0\text{cm}^{-1}$  is assigned to the bending vibrational mode of water molecule the bend observed at  $738.1\text{cm}^{-1}$  is assigned to the liberation mode of water molecule in general a free  $\text{SO}_4^{2-}$  ion has four fundamental vibration, namely a non degenerate mode ( $\nu_1$ ) at  $983.9\text{cm}^{-1}$ , doubly degenerate mode ( $\nu_2$ ) at  $630\text{cm}^{-1}$  and triply degenerate vibration ( $\nu_3$  and  $\nu_4$ ) at  $1101.04\text{cm}^{-1}$ , respectively. The band observed at  $462.05\text{cm}^{-1}$  is reasonably assigned to the ( $\nu_1$ )  $\text{SO}_4^{2-}$  mode the peak appeared at  $983.9\text{cm}^{-1}$  is reasonably assigned to the ( $\nu_1$ )  $\text{SO}_4^{2-}$  non degenerate mode [5].

### 3.2 X-Ray Diffraction

X-ray diffraction pattern for the powdered sample of grown crystal was presented in fig .X-ray diffraction was carried out using a strip alpha powder X-ray diffractometer. It was taken from Bargur arts and Science College. It was solved by using JCPDS software package.

The crystal structure was orthorhombic, primitive lattice parameters [6].

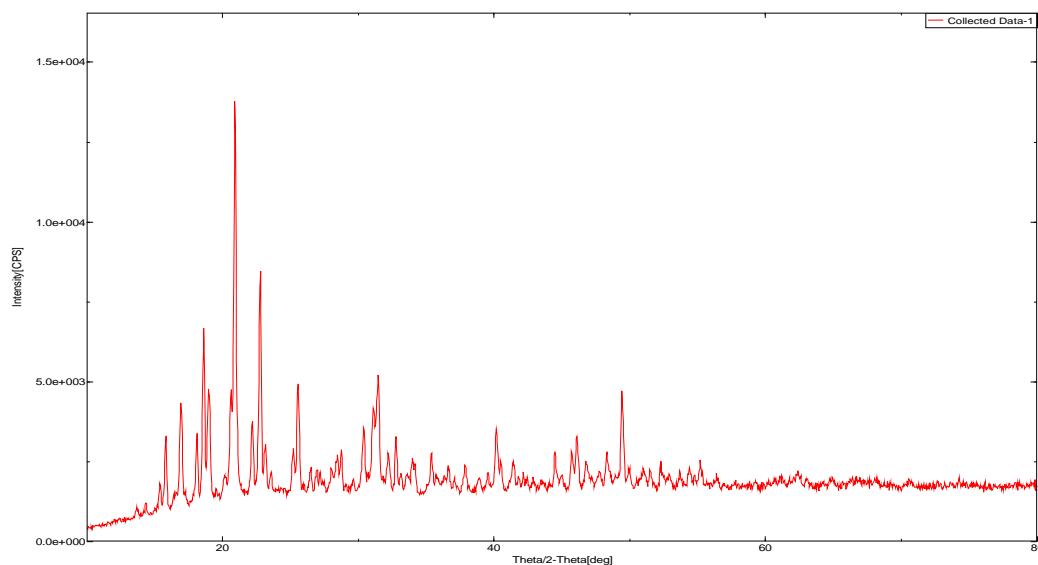


Fig .2 XRD of zinc sulphate



### 3.3 UV – Visible Studies

The UV – visible spectrum of zinc sulphate was scanned in the range of 200 – 800 nm shown in figure. The spectrum has low absorption in between 200 – 400 nm. So the zinc sulphate crystal expected to be transparent in the UV range 400 – 800 nm infrared that the spectrum is losing transparency which may be due to intrinsic loss mechanism by the interaction of electromagnetic radiation of visible infrared region[7,8].

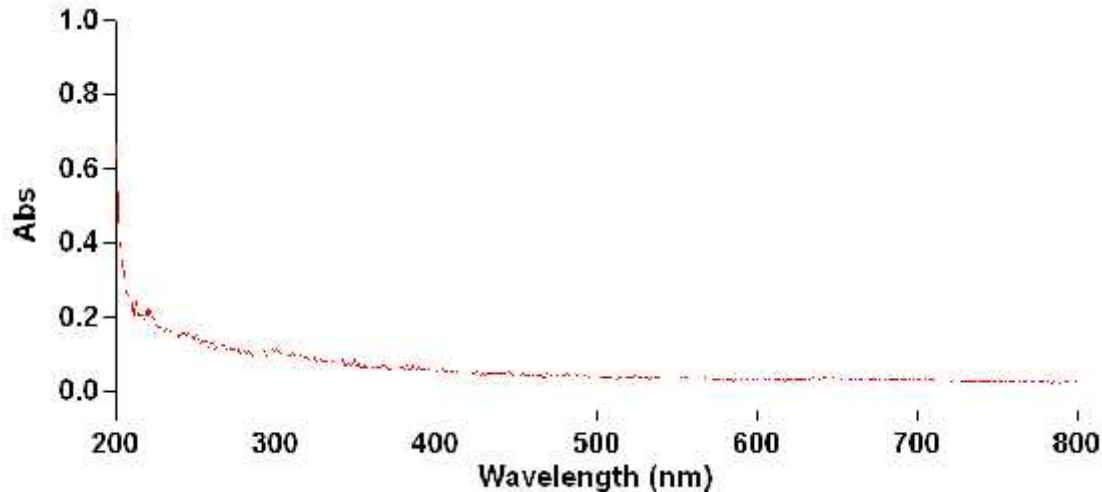


Fig 3 Uv-Visible Spectrum of Zinc Sulphate

### 4. Conclusion

The pure  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  single crystals were grown from aqueous salutation by slow evaporation method. The solubility of  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  has been studied in the room temperature around  $32^\circ\text{C}$  the solubility was found to be 93/50ml. good transparent single crystal of  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  was obtained over a growth period of 24 days. The crystals with the dimension of  $1.5\text{cm} \times 0.4\text{cm} \times 0.5\text{cm}$  were grown from aqueous salutation by slow evaporation method. The presence of functional groups in zinc sulphate has been identified by FT-IR spectral analysis. The powder X-ray diffraction study confirms the lattice parameter values. The UV-Vis spectrum the zinc sulphate crystal is found to be 99% transparency in the region.

### References

1. J.K. Saha and j. Podder\*Department of Physics, Bangladesh University of Engineering and Technology, Dhaka-1000, Bangladesh.
2. Anderson, J. L., Peterson, R. C. and Swainson, I. P. 2005. Combined neutron powder and X-ray single-crystal diffraction refinement of the atomic structure and hydrogen bonding of goslarite ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ). *Miner Mag.* **69**(3): 259.
3. Baur, W. H. 1964. The refinement of the crystal structure of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  (epsomite). *Acta Crystallogr.* **17**: 1361-1369.
4. Cano, H., N. Gabas and J. P. Canselier. 2001. Experimental study on the ibuprofen crystal growth morphology in solution. *J. Crystal Growth* **224** : 335.
5. Dana, E. S. and W. E. Ford, 1985. *Crystallography and Physical mineralogy*. Wiley Eastern Limited, pp. 760.
6. Dhumane, N. R., S. S. Hussani, V. G. Dongre and M. D. Shirsat. 2008. Study on the effects of Glycine on the nonlinear optical (NLO) properties of Zinc (tris) Thiourea Sulfate (ZTS) single crystal. *Optical Materials* **31**: 328-332.
7. Ema, M. and A. Gebrewold. 1998. BT Altura and BM Altura Report, Department of Physiology, State University of New York, Health Science Centre, Brooklyn. Herzberg, G. 1960. *IR and Raman spectra of poly-atomic molecules*. Van Nostrand, New York, 2nd Ed.
8. Hussani, S. S., N.R. Dhumane, V.V. Nawarkhele, G. Rabbani and M.D. Shirsat. 2008. Growth and High frequency study of non liner optical Zinc ( tris) Thiourea Sulphate Crystal. *Frontiers of Microwaves and Optoelectronics* **141-149** ISBN 978- 81-89927-19-6.
9. Ikeya, M., M. G. Hassan, H. Sasaoka, Y. Kinoshita, S. Takaki and C. Yamanaka. 2000. Strategy for finding new materials for ESR dosimeters. *Appl. Radiat. Isot.* **52**:1209. 210 SAHA AND PODDER.



10. Kanagadurai, R., R. Durairajan, R. Sankar, G. Sivanesan, S.P. Elangovan and R. Jayavel. 2009. Nucleation Kinetics, Growth and Characterization Studies of a Diamagnetic Crystal-Zinc Sulphate Heptahydrate(ZSHH). *E-Journal of Chemistry* **6**(3): 871- 879.
11. Kasatkin, I. A. 2002. Interferometric study of MgSO<sub>4</sub>. 7H<sub>2</sub>O single crystal growth kinetics from solution. *Cryst. Res. Technol* **37**(2-3): 193-205.
12. Kubota, N., J. Fukazawa, H. Yashiro and J. W. Mullin 1995. Impurity effect of chromium (III) on the growth and dissolution rates of potassium sulfate crystals. *J. of crystal Growth* **149** : 113.
13. Ramalingom, S., J. Podder and S. N. Kalkura. 2001. Crystallization and characterization of arthorhombic MgSO<sub>4</sub>.7H<sub>2</sub>O. *Cryst. Res. Technol* **36**(12): 1357-1364.
14. Sgualdino, G., G. Vaccari, D. Aquilano and M. Rubbo. 1987. Growth kinetics of epsomite (MgSO<sub>4</sub>. 7H<sub>2</sub>O). *J. of Crystal Growth* **83**(4): 523-527.
15. Sivanesan, G., P. Kolandaivel and S. Selvasekarapandian. 1993. Laser Raman and FT-IR studies of pure and Zn-doped TGS, *Mat Chem Phys.* **34**: 73.
16. Tomas, G., Petrov Evgenil, B., Trivus, P. Aleksei and K. Asatkin. 1969. Growing crystal from solution. Consultants Bureau, New York **21**: 99-106. (Received revised manuscript on 10 August, 2011).