International Journal of Technical Research and Applications e-ISSN: 2320-8163,
www.ijtra.com Special Issue 37 (February, 2016), PP. 86-89

STUDIES ON STRUCTURAL AND PHYSICAL PROPERTIES OF POTASSIUM ZINC SULPHATE HYDRATE SINGLE CRYSTAL

S. Karthick, A. Albert Irudayaraj, A. Dhayal Raj, R. Vinayagamoorthy
PG and Research Department of Physics,
Sacred Heart College, Tirupattur, Vellore, India.
s.karthick5583@gmail.com

ABSTRACT: **Potassium Zinc Sulphate Hydrate (PZSH) crystals have been grown by slow evaporation technique at room temperature. The single crystal X-ray diffraction results reveal that PZSH belong to monoclinic system with space group $P2_1/C$. The presences of various functional groups of the PZSH have been confirmed by FTIR analysis. EDAX result reveals the presence of PZSH elements in the sample. The optical transparency and optical band gap of the PZSH are determined by UV-Visible analysis. PZSH is found to be optically transparent in the entire UV-Visible region. The NLO property of the PZSH has been measured by Kurtz Perry powder method and PZSH has been found to exhibit second harmonic generation. The second harmonic generation efficiency of PZSH is equal to 0.49 times than that of KDP. The thermal stability and decomposition process of PZSH are studied by TGA and DTA. PZSH is found to have poor thermal stability. Saturation magnetization (M_s), coercive field (H_c) and remanent magnetization (M_r) of PZSH are measured by VSM analysis.**

KEYWORDS: **Monoclinic, Second Harmonic Generation, Saturation magnetization, Coercive field, Remanent magnetization,**

I. INTRODUCTION

Potassium Zinc Sulphate hydrate is an inorganic material belonging to the family of Tutton's salts. This family of salts have the formula $M'_2M'' (XO_4)_2(H_2O)_6$ (M´ = K, NH4, Rb, Cs; M´´ = Mg, Mn, Co, Ni, Cu, Zn; X = S, Se)[1]. These materials contain two cations, M^+ and M^{2+} crystallized in the same ionic lattice. Tutton's salts are of historical importance, because they are obtainable in high purity and served as reliable reagents and spectroscopic standards[2] and some of these Tutton's salts are used as UV light filters[3]. In the present work Potassium Zinc Sulphate hydrate crystals are grown by slow evaporation technique and their structural, optical, magnetic and thermal properties are studied.

II. EXPERIMENTAL

SYNTHESIS AND CRYSTAL GROWTH

PZSH is synthesized based on the following reaction:

$$K_2SO_4 + ZnSO_4.7H_2O \longrightarrow K_2Zn(SO_4)_2.H_2O$$

All the reagents used were of analytical grade. Potassium Sulphate and Zinc Sulphate were taken in the ratio 1:1 and dissolved in deionized water and the solution was left undisturbed. After few days, Potassium Zinc Sulphate Hydrate raw material was collected from the bottom of the beaker. Super saturated solution of Potassium Zinc Sulphate Hydrate (PZSH) was prepared at

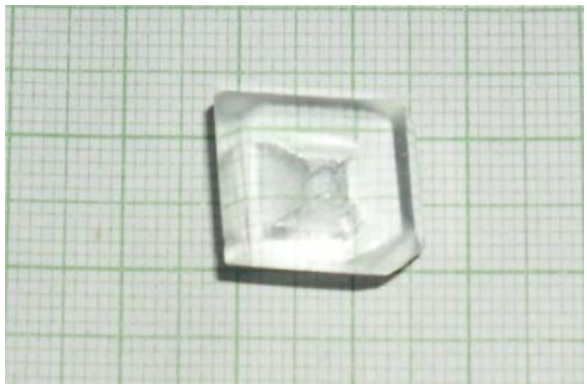

Fig.1 Grown Potassium Zinc Sulphate Hydrate Crystal

room temperature and the solution was filtered twice using Wattmann filter paper.

The pure super saturated solution was allowed to crystallize by slow evaporation at room temperature.

Grown crystals were harvested and subjected to recrystallization thrice in order to improve the purity of the crystals. Good quality, optically transparent, colorless crystals were harvested after a growth period of 7 weeks. Picture of the grown PZSH crystal is shown in figure 1.

III. CHARACTERIZATION

Crystals of Potassium Zinc Sulphate Hydrate (PZSH) were subjected to various characterization studies. The phase analysis, structure identification and lattice parameters determination of the grown crystals were carried out by single crystal X-ray diffraction studies using Bruker Kappa APEXII diffractometer. The functional groups of the sample were confirmed by FT-IR spectroscopy using Perkin Elmer Spectrum TWO Fourier Transform Infrared Spectrophotometer by KBr pellet technique. The quantitative elemental analysis of PZSH was carried out by energy dispersive X-ray spectroscopy by using the instrument FEI QUANTA 200F energy dispersive X-ray micro analyzer. The optical transmission range of the Potassium Zinc Sulphate Hydrate (PZSH) crystals was determined by Varian Cary 50 Bio UV–Vis spectrophotometer. A Q-switched Nd:YAG laser beam of wavelength 1064 nm was used for Second harmonic generation efficiency studies. The magnetic properties of PZSH were determined by LAKE SHORE 7400 VSM at room temperature. The thermal analysis including TGA and DTA was carried out using a simultaneous thermal analyzer SDT Q600 V8.3 Build 101, heating rate 20ºC/min in nitrogen atmosphere.

SINGLE CRYSTAL X-RAY DIFFRACTION ANALYSIS

Good quality PZSH crystal was subjected to single crystal X-ray diffraction analysis using Bruker Kappa APEXII diffractometer with monochromatic, graphite filtered, CuKα radiation. For structure solution and refinement SHELXTL software was used. The results obtained reveal that PZSH belong to monoclinic system with space group $P2_1/C$. The lattice Parameters are, a= 6.189 Å, b= 12.228Å, c= 9.09189 Å, α = 90˚, β = 90˚ and γ =104.92˚. The unit cell dimensions and crystal structure of the crystal of PZSH is similar to the Tutton's crystals family[4-6].

FOURIER TRANSFORM INFRARED (FTIR) SPECTRAL ANALYSIS

Figure 3 shows the FTIR spectrum of PZSH recorded in the wavenumber range between 400 cm^{-1} - 4000 cm^{-1}. The vibration peaks at 3411 cm^{-1} and 2090 cm^{-1} are due to OH symmetric stretching. The sharp peak observed at 1620 cm^{-1} is due to OH asymmetric stretching. The band observed at 1107 cm^{-1} is due to SO_2 out of plane wagging. The vibration peaks observed at 757 cm^{-1} and 618 cm^{-1} are due to SO_2 deformation. The band observed at 561 cm^{-1} is due to SO_2 rocking.

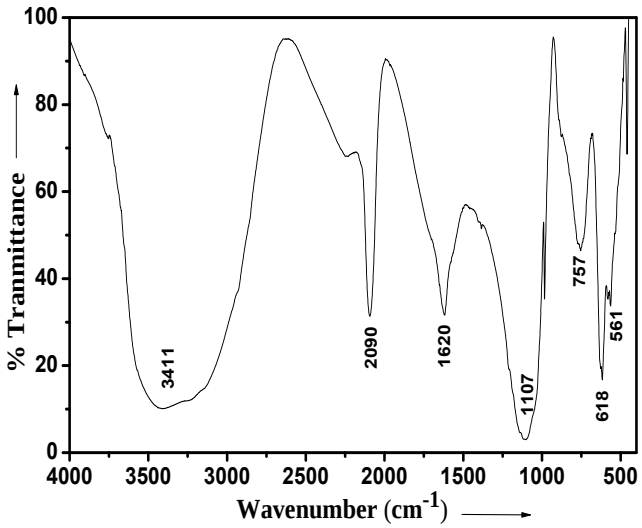

Fig. 3 FTIR Spectrum of PZSH

EDAX Analysis

Energy dispersive X-ray analysis (EDAX) is an analytical technique which is used to obtain useful information regarding the chemical composition of materials. The grown crystal was subjected to EDAX. The EDAX spectrum and elemental composition of PZSH is depicted in figure 4. The results confirm that the grown crystals are PZSH crystals.

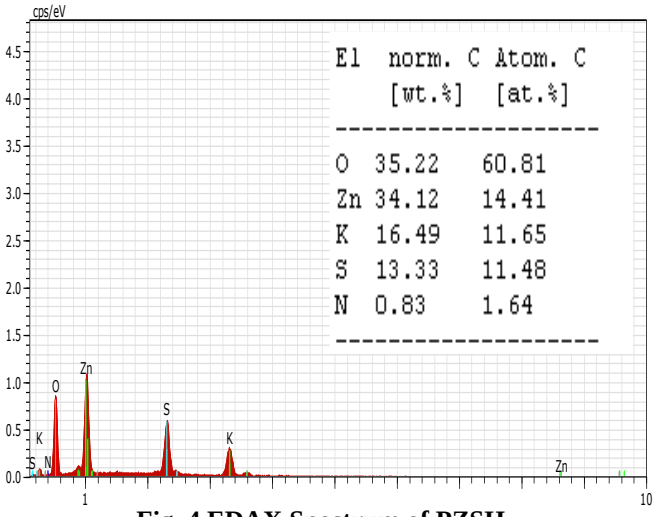

Fig. 4 EDAX Spectrum of PZSH

UV-VISIBLE SPECTRAL ANALYSIS

The optical transmission spectrum of PZSH is shown in figure 5. The transmission spectrum was recorded in the range 200 – 800 nm. The crystal is highly transparent in the entire visible region, whereas it has a UV cutoff at 228 nm. The transmission is uniformly high (98%) for light in the visible region of the electromagnetic spectrum. The wide range transmission is an important requirement for a crystal exhibiting NLO behaviors[7]. From the spectrum, the optical energy gap of PZSH is found to be 5.4 eV.

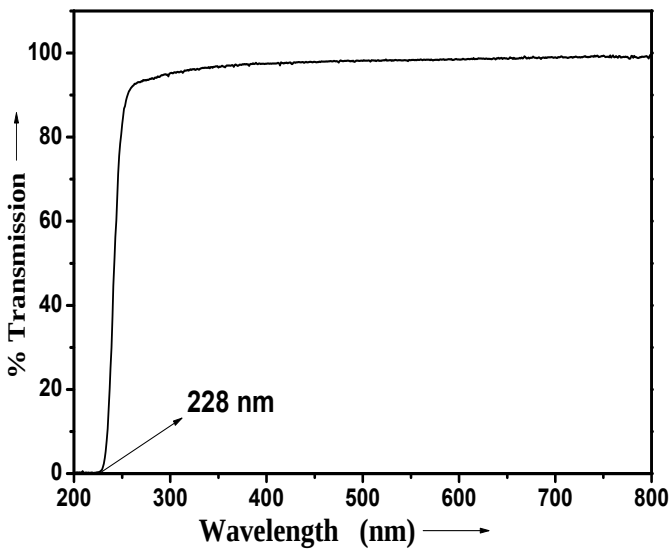

Fig. 5 UV-Visible Spectrum of PZSH

SECOND HARMONIC GENERATION ANALYSIS

The second harmonic generation of PZSH was studied by Kurtz and Perry technique using Q-switched mode locked Nd:YAG laser. The crystal was ground into a homogeneous powder of particles and compactly packed in a one end closed capillary tube and this tube is kept in a sample holder. For a laser input pulse of 4.6 mJ/pulse, the second harmonic signal of 57 mV was produced in PZSH. The SHG efficiency of PZSH is nearly 0.49 times that of KDP (for KDP the output signal was 117 mV).

VSM ANALYSIS

Vibrating Sample Magnetometer (VSM) analysis was carried out to identify the magnetic properties of the material. Magnetic measurements of the PZSH were carried out at room temperature and the hysteresis curve is shown in figure 6.

The magnetization curve showed a mixture of ferromagnetic and diamagnetic signals[8]. The characteristic hysteresis loop of PZSH show that the saturation magnetization increases with increasing field. The saturation magnetization reaches its maximum value of 238.43 X 10^{-6} emu. The amount of magnetism remaining in the sample after removal of saturated field (remanent magnetization) is 11.336 X 10^{-6} emu. The coercivity in the order of 1835.6 G is clearly observed in the B - M curve.

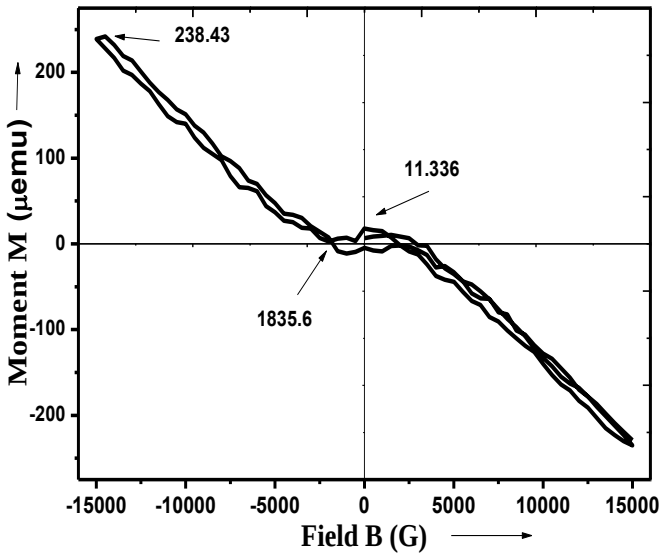

Fig. 6 Hysteresis curve of PZSH

THERMAL ANALYSIS

Thermal properties of PZSH were studied by TGA and DTA. The TGA and DTA spectrum of PZSH was carried out from room temperature to 700° C in the nitrogen atmosphere at a heating rate of 20°C/min. The TGA and DTA curves obtained for PZSH are shown in figure 7. It is seen that the material undergoes three definite phase transitions. The first phase transition indicated by the endothermic peak at 121.1°C corresponds to a weight loss of 24.1%. This phase transition may be due to the melting of potassium ions and evaporation of

water molecules. The other two phase transitions indicated by the exothermic peak at 267.7ºC and the endothermic peak at 475.4ºC correspond to no weight loss. Hence they represent the phase transition of the material from solid to liquid (melting)[9]. Beyond 211.6ºC no significant weight loss is noticed.

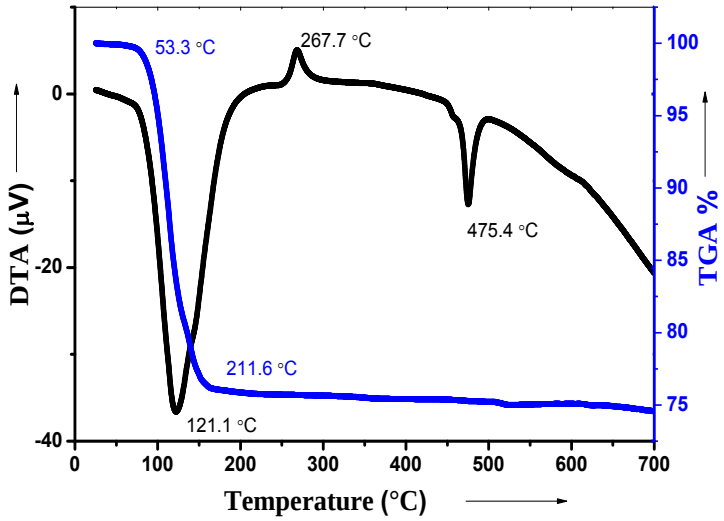

Fig. 7 TGA/DTA curves of PZSH

IV. CONCLUSIONS

Crystal of Potassium Zinc Sulphate hydrate was grown by slow evaporation method. Single crystal X-ray diffraction revealed that PZSH belong to monoclinic system with space group $P2_1/C$. The FTIR analysis confirmed the presence of various functional groups of the PZSH. The chemical composition of PZSH was confirmed by Energy dispersive X-ray analysis (EDAX). The UV–Vis spectroscopic analysis revealed that the PZSH is highly transparent in the entire visible region and has a UV cutoff at 228 nm. From the UV–Vis spectrum, the optical energy gap of PZSH was found to be 5.4 eV. The second harmonic generation efficiency of PZSH was 0.49 times that of KDP. The thermal analysis of PZSH revealed that PZSH undergoes three definite stages of phase transitions with the major weight loss occurring around 121 ºC. The VSM studies showed that PZSH exhibits a mixture of dia as well as ferromagnetic properties.

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