International Journal of Science and Research (IJSR)
ISSN: 2319-7064
Impact Factor (2018): 7.426

Growth and Characterization of L-Valine Doped Zinc Sulphate Single Crystals

S. Ajitha[1], A. Ancy[2]

[1, 2]Department of Physics, Nanjil Catholic College of Arts and Science, Kaliyakavilai

Abstract: ***Pure and L-Valine doped zinc sulphate crystals have been successfully grown from aqueous solution by slow evaporation technique. Doped crystals were grown in a period of three weeks. The lattice parameters were determined by PXRD analysis. FTIR analysis was used to confirm the presence of various functional groups in the grown crystals. Optical properties studies by UV analysis. Thermal studies analyzed by TG/DTA.***

Keywords: ZnS, PXRD, FTIR, lattice parameter, UV, TG/DTA.

1. Introduction

A crystal is an atomic tessellation, a tri dimensional jigsaw puzzle in which every piece is the same shape. A crystal assembles itself out of its own constituent disarray the puzzle puts itself together, each piece falling as through by chance into its correct location[1].

Crystallography or the science of crystals is today of crucial importance in many, often unrecognised ways. Understanding the nature of crystals, especially their atomic structure, is vital for many practising scientists and for industry. A particular drug is patent – protected by supplying a powder diffraction pattern, or occasionally a full crystal structure determination. Crystallography is one of the most interdisciplinary subjects in science [2].

2. Materials and Methods

Analytical reagent grade(AR) samples of L-Valine doped zinc sulphate along with distilled water were grown by slow evapouration method for single crystals. Pure zinc sulphate and L-Valine doped Zinc sulphate with in 0.03 mol% solution was prepared. The beaker is closely tightly with polythene paper, and the small holes are made for the perfect evaporation. The period of crystal growth was 15 to 20 days. After completion of growth, the crystals were harvested. A large size crystals were selected for the experiments. The formed crystals were carefully harvested from the beakers. The crystals are dried using filter paper. Growth of pure and L-Valine doped ZnS crystals by slow evaporation technique is reported. The grown crystals are characterized by powder XRD, FTIR, UV and TG/DTA analysis.

3. Results and Discussion

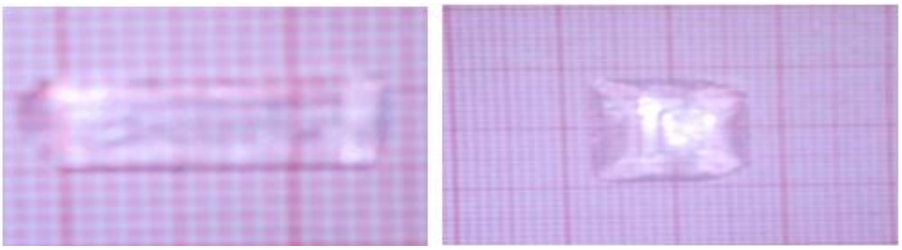

Figure 1: Photograph of all the grown crystals

Table 1: PXRD Intensity for different 2θ peaks for pure ZnS crystal

Angle in degree 2θ	Intensity	d- spacing	Miller indices [hkl]	Lattice Parameter
15.2625	26.74	5.80538	004	
16.0669	28.14	4.91008	110	
20.3559	100.00	4.36283	112	
22.2282	65.49	3.99939	114	
25.0621	21.18	3.55322	113	
29.8735	19.74	2.99099	114	
30.8941	25.95	2.89447	204	a=11.810
33.4526	13.29	2.67873	020	b=12.247
36.0953	07.00	2.48843	115	c=6.192
40.0230	06.72	2.25283	116	
45.5815	14.30	1.99020	312	
48.8908	20.83	1.86295	313	V=895.592

Table 2: PXRD Intensity for different 2θ peaks for L-V doped ZnS single crystal

Angle in degree 2θ	Intensity	d- spacing	Miller indices [hkl]	Lattice Parameter
14.8052	27.69	5.98365	004	
17.6104	29.52	5.03631	110	
19.8925	100.00	4.46339	112	
21.7731	69.65	4.08196	114	
24.6213	22.68	3.61582	113	
29.4190	19.01	3.03616	114	a=11.830
30.4601	33.20	2.93472	204	b=12.256
34.4108	09.68	2.60630	020	c=6.192
39.1820	13.90	2.29922	115	
45.1615	13.18	2.00773	116	
48.4779	15.22	1.87785	312	
52.7444	07.16	1.73556	313	V=887.329

1 Powder X-Ray Diffraction

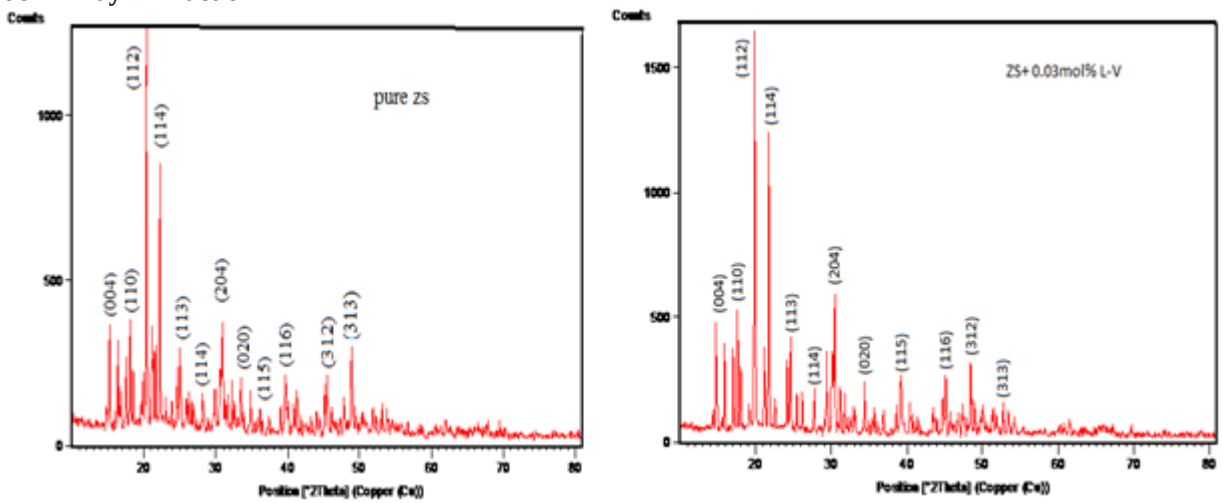

Figure 2: PXRD Spectrum for pure ZnS and L-V doped ZnS

The powder method is used to determine the value of the lattice parameters accurately. Lattice parameters are magnitudes of the unit vectors a, b, and c which define the unit cell for the crystal. The powder XRD (PXRD) patterns obtained for the L- Valine doped Zinc sulphate crystal in the present study is well matched with the JCPDS (22-1019) data. The numerous sharp peaks found in the PXRD patterns give a clear cut proof of the crystalline nature of all the grown crystals. The phases have been clearly indexed doping of Zinc sulphate in 0.03 mol% concentration produce a slight shift in the Bragg angle. The peak intensity of pure crystals is decreased / increased with increasing concentration of Zinc sulphate in the host lattice the observed sharp peaks and low full-width at half maximum confirm that the crystalline nature of the grown crystals. This may be due to the absorption or substitution of doped atom in lattice sight. There is slight variation in the lattice parameters depending upon the impurity addition and at the same time, the total crystal structure is not affected.

FTIR Analysis

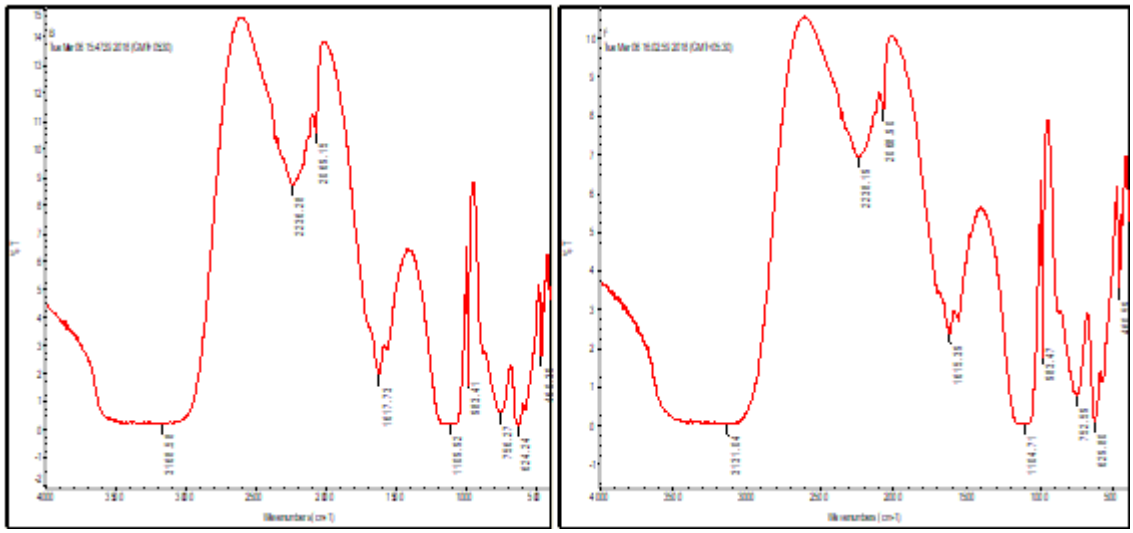

Figure 3: FTIR spectrum for pure and 0.03 mol% L-V doped ZnS

Table 3: FTIR absorption frequencies(cm^{-1}) for L-V doped ZnS in force constant

Wave number ϑ (cm^{-1})		Band assignment	Force constant K	
Pure	0.03 mol% L-V doped ZnS		Pure	0.03 mol% L-V doped ZnS
3168.58	3131.04	N−H symmetric stretch	0.05564	0.05433
2236.28	2238.19	C≡ N stretching	0.19089	0.19057
2069.15	2068.90	C== stretching	0.15153	0.15150
1617.73	1615.39	C=C stretching	0.09263	0.09236
1109.92	1104.71	C−O stretching	0.0498	0.04933

The FTIR spectra of pure and L- Valine doped Zinc sulphate crystals are shown in figure 3. The FTIR spectra of doped crystals show a strong NH absorption peak at about 3168.58 cm-1. Since L- Valine doped Zinc sulphate more shows NH stretch vibrations are introduced due to doping and as a result at 3131.04 $cm^{-1}$the NH absorption peak becomes

stronger. The wave number 2236.28- 2238.19 cm^{-1} is C≡ N symmetric stretching vibration. The wave number region 2069.15-2068.90 cm^{-1} is assigned to C=C stretching vibration. The wave number region 1617.73-1615.39 cm^{-1} is assigned to C=C stretching vibration mode. The wave number region 1109.92-1104.71 cm^{-1} is assigned to C-O symmetric stretching vibration mode. The wave number region 983.41-983.47 cm^{-1} is assigned to C-H bending vibration. The wave number region 756.27-752.59 cm^{-1} is assigned to C-H out of plane bending vibration. This assignments confirm the presence of various functional group present in the material.

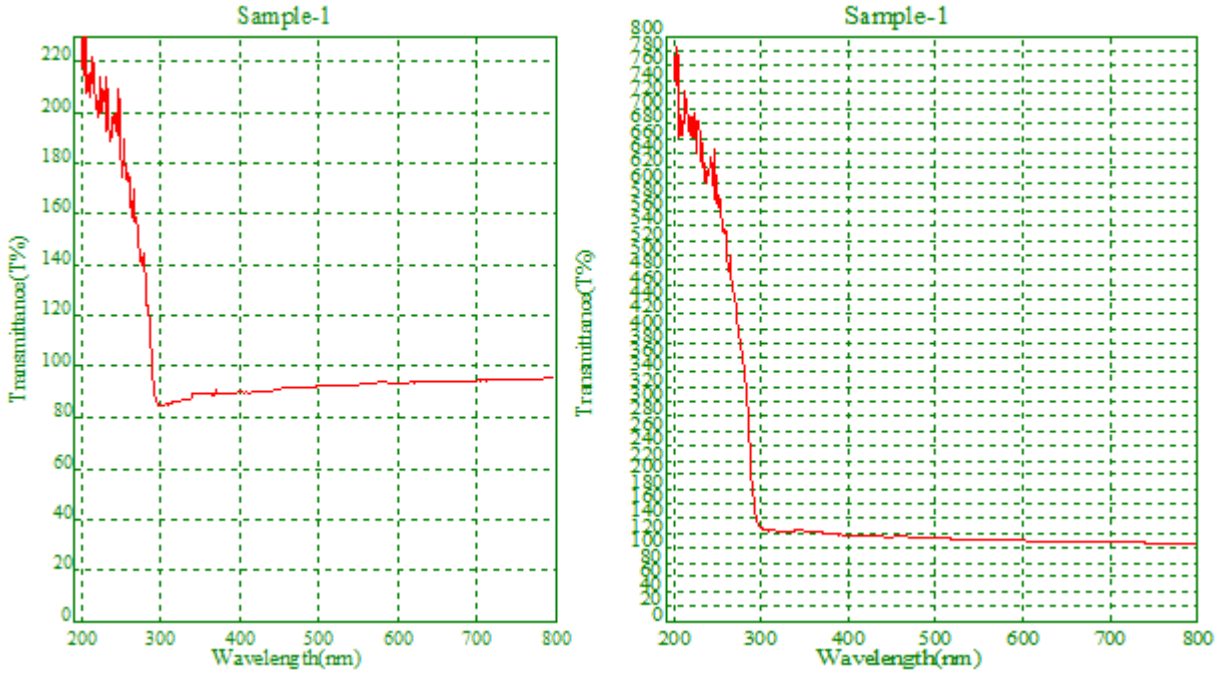

Figure 4: UV spectrum for pure ZnS and L-V doped ZnS

Table 4: UV Energy band gap

System	λ	E_g
Pure	298	4.1687
0.03 mol % L-V doped ZnS	299	4.1551

UV- Visible spectrum analysis has been carried for pure and L- Valine doped zinc sulphate. It reveals the transparent wave length region in the visible region which gives the hints for the insulating nature of crystals. The absorption and transmission spectra for pure and L- Valine doped zinc sulphate are shown in figure 4.5&4.6. The absorption and the transmission spectra are very important for NLO material. Because of a non linear optical material can be of practical use only if it has wide transparency window. The large transmission in the entire visible region and cut off wave length enables it to be a potential material for second and third harmonic generation. The band gap energy E_g was found to be 4.1551 and 4.1687eV for pure and L- Valine doped zinc sulphate single crystals. From the result we conclude the crystal is semiconductor.

TG/DTA Analysis

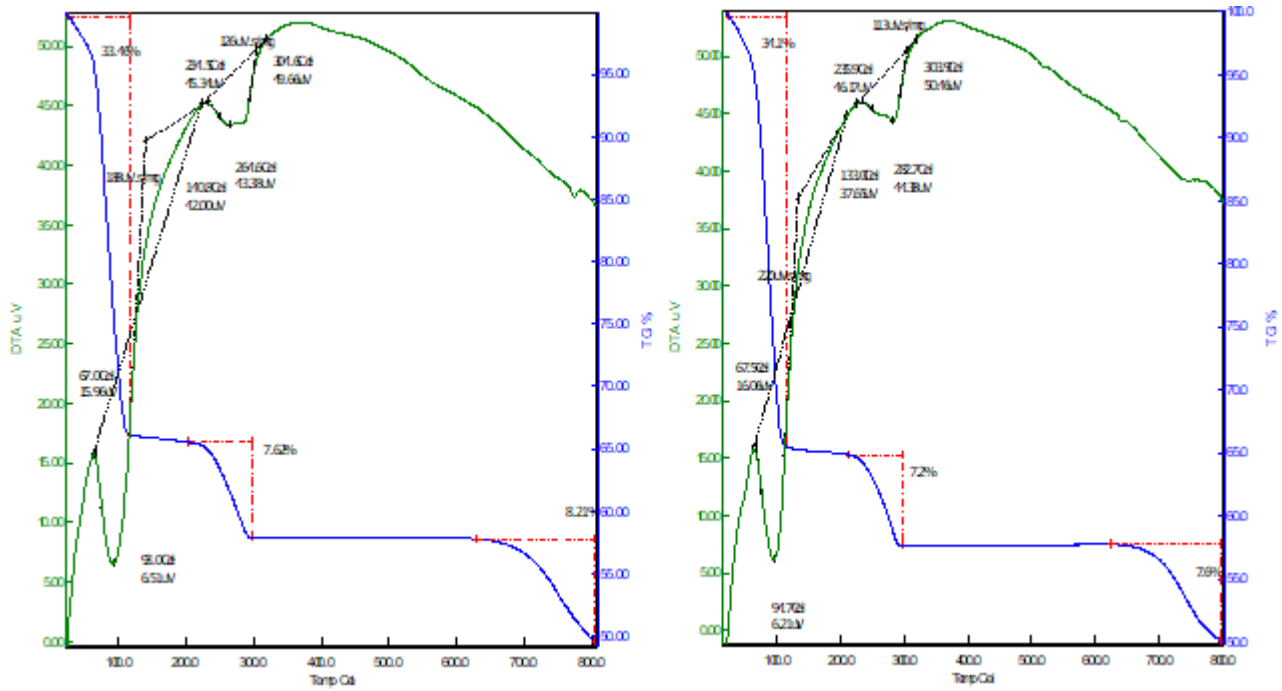

Figure 5: TG/DTA spectrum for pure ZnS and L-V doped ZnS

Table 5: TG/DTA spectrum for 0.03 mol% L-V doped ZnS

System	Endothermic °C	Start the decomposition
Pure	94.7	67.5
0.03 mol % L-V doped ZnS	93.0	67.0

The grown zinc sulphate crystal was crushed into fine powder and thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) were recorded using Q50 W/FMC DTA analyzer in the temperature range from room temperature to 700°C at a heating rate of 30°C/min. The analysis was performed in air atmosphere. In the TG/DTA, the strong endothermic peaks located at 94.7°C, 93°C depict the crystallisation of some of the phases of the decomposed material. The curved portions indicate the regions of thermal stability of pure and 0.03 mol % L-Valine doped Zinc sulphate crystals and the compound formed from it. The grown crystals are losing their weight gradually and slowly upto 67.5°C and 67°C for pure and 0.03 mol % L-Valine doped Zinc sulphate samples. From the figure 680°C (1.256°F; 953k) decomposes (anhydrous) 100°C (heptahydrate) 70°C decomposes (hexahydrate).Glass transition temperature is one of the most important properties of any epoxy and is the temperature region where the polymer transitions from a hard, glassy material to a soft, rubbery material.

4. Conclusion

Good optical quality of single crystals of pure and L- Valine doped zinc sulphate crystals were grown by slow evaporation solution growth method at room temperature. The powder X-Ray diffraction study confirms the lattice parameter values. The lattice parameters have been found by single crystal X-ray diffraction technique. X-Ray diffraction studies confirmed that pure L- Valine doped zinc sulphate crystals were crystallized in orthorhombic system.

The FTIR spectrum reveals that the various functional groups present in the grown crystal. The FTIR studies assign vibrational frequencies. From the UV spectrum, the zinc sulphate crystal is found to be transparent in the UV region and it could be a useful candidate for optoelectronic applications in visible and infrared region. L- Valine doped zinc sulphate single crystal is transparent and found an application in optical studies. Thermal analysis revealed the thermal stability of the grown crystals and shown the suitability of the crystals for device fabrication. Thermal stability of the grown crystal were studied by Thermo gravimetric (TG) and Differential Thermal Analysis (DTA) and found that the grown crystals are suitable for decrease fabrication for frequency conversion applications.

References

[1] Christian bok, “Crystallography”. 2003
[2] A.M Glazer, “Crystallography a very short introduction”, 2006.
[3] Ariponnammal, S. Chandrasekaran, C. Sanjeevi Raja, Digest Journal of Nano materials and Biostructures, 2012, pg: 947-957.
[4] Jin- seon song, Francis K. Mante, William J Ramanow, Synguck kim, Dec-2006, vol (102), pg: 809-815