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*Vasyl StefanykPrecarpathian
National University*

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G. Susithra, D. Vidhya, S. Ramalingam

(Cd)SO_4 doping on L-Valine crystal for the enhancement of opto-electronic and laser properties

Department of Physics, A.V.C. College, Mayiladuthurai, Tamilnadu, India

L-Valine is a good molecular platform to induce NLO properties for Laser application and considerable laser properties can be improved by injecting suitable electron administrated substitutional groups. This work is an attempt to make laser properties enhancement by increasing Cd solvent concentration on L-Valine compound. The recorded XRD peaks addressed the significant increment of laser properties of the composite crystal. The results obtained for pure L-Valine was; a =5.221 (Å), b= 6.910 (Å) and c = 4.994 (Å) and associated indices of refraction was n_1 = 1.511, n_2 = 1.583 and n_3 = 1.599. The results of 2 % $CdSO_4$; a = 5.580 (Å), b= 7.011 (Å) and c= 5.091 (Å) and associated indices of refraction was n_1 = 1.561, n_2 = 1.612 and n_3 = 1.633. For 5 % $CdSO_4$; a = 5.599 (Å), b = 7.831 (Å) and c= 5.659 (Å) and associated indices of refraction was n_1 = 1.588, n_2 = 1.661 and n_3 = 1.699. For 10 % $CdSO_4$; a=5.592 (Å), b= 7.612 (Å) and c= 5.822 (Å) and associated indices of refraction was n_1 = 1.661, n_2 =1.693 and n_3=1.593. For 15 % $CdSO_4$; a = 5.551 (Å), b = 6.332 (Å) and c= 4.734 (Å) and associated indices of refraction was n_1 = 1.501, n_2 = 1.599 and n_3 = 1.593. The non linear refractive index was changed from 3.891×10^{-8} cm^2/W to 4.231×10^{-8} cm^2/W for 2 %, 5 %, 10 % respectively and Third order non linear optical susceptibility($\chi^{(3)}$) was ranged from 4.621×10^{-6}esu to 5.091×10^{-6}esu.The restored chemical potential for non linear optical (NLO) and birefringence properties was found to be improved. The scattering capability of homo nuclear and hetero nuclear bonds along with the $CdSO_4$ voids was identified for attaining non linear scattering process.

Keywords: $CdSO_4$, birefringence, susceptibility, non linear refractive index, EDAX.

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Introduction

The optical intrinsic properties of the material usually depend on the involvement of electric and magnetic fields associated with molecular dipole moments existed in the molecular material. It is always predominant in heterogeneity of the material since the availability of metallo-ionic bonds in the metal oxide materials and heteronuclear bonds in organic semiconducting materials. The optical vector components are active when the dipole lenity domains in the material medium. In this work, as the metal sulphate was doped with organic species like Valine compound, the heterogeneity of the medium is considerably increased and when the concentration of the heterogeneity improved, the non linear coefficients significantly developed and thus the light interaction with molecules for transmission and processing of light signal is controlled. Here, it was attained by increase the concentration of metal sulphate on L-Valine compound. It induces the macroscopic third-order nonlinearity in the molecular sites of material strengthens intrinsic optical properties.

These days, there is enormous demand on novel metal sulphate supported organic crystal materials used for the fabrication of analog and digital devices to process the signal with ultrahigh frequency, digital signal processing (DSP), opto-electronic modulator process, summing and imposing of digital signals and amplifying. In current decades, many works have been carrying out to prepare metalloid crystal preparation process to enhance NLO properties for achieving organized NLO applications for Laser devices to construct ultra-fast opto-electronic communicable, ultra-frequency processing and subtraction electronic equipments [1-2]. Metalloids Crystal with high degree of heterogeneity is able to produce additive interaction between material and light energy by inducing dipole-dipole scattering potential. They are commonly used for operating of Laser-Photonic devices for laser propagation and processing [4-5]. In this course of action, metalloids complex crystals have received substantial intension to explore its NLO applications for the transmission, optical frequency summing and electro-optical processing [6-9].

I. Experimental Methods

In this work of crystal synthesis, organic L-Valine base was used as starting material and CdSO$_4$ (AR grade) was acted as doping material and 98:2, 95:5, 90:10 and 85:15 stoichiometric ratios have been used to analyse the associated parameters. The CdSLV crystal was calculated as per the following reaction:

$$C_5H_{11}NO_2 + CdSO_4.6H_2O \rightarrow Cd\ [C_5H_{11}NO_2]\ SO_4$$

The Cadmium sulfate hexahydrate was dissolved in distilled water at required temperature and it was stirred moderately using magnetic stirrer. The organic base L-Valine was then mixed to the solution of CdSO$_4$ at different concentration of 2 %, 5 %, 10 % and 15 % respectively. Then the solution was stirred up for about an hour time endlessly and heated for 20 minutes until the absolute dissolution at the state of starting materials was reached. The heated solution for growing crystal was filtered and allowed to dehydrate at room temperature. The crystals were grown and raw was obtained by slow evaporation technique in the duration of 10 days. The cleanliness of crystal was additionally enhanced by recrystallization process and single crystal was obtained.

1.1. Recording details.

The XRD signal sequence was recorded for all as prepared metalloid crystals and the clear XRD spectrum was recognized by mapping out the analysis.

The FT-IR vibrational spectral pattern was recorded to perform analysis using a Bruker IFS 100V vib-rot module spectrometer. The filter process is performed by making multiple scanning and the sequential modulation pattern of finger print as well as substitutional regions was recorded.

The FT-Raman vibrational wavenumber pattern of same compositewas recorded using same Bruker spectrometer adopted with Raman module supported with a Nd:YAG laser source setup being operated at 1.063 µm line width with 700 mW power.

1.2. SHG Measurement methods.

The third harmonic generation (THG) efficiency was estimated by the latest version of the powder pellet technique which was developed by Kurtz and Perry using an Nd: YAG, laser beam pulsed at 10 ns with a working frequency of 10Hz at 532 nm. The sample was grind as a fine powder and filtered by high pressure method. The sample iskept at micro-capillary tube as a sample holder and it was mounted in the path of the laser beam of 1.5 mJ pulse obtained by splitting the original laser beam. The laser pulse was repeated and the measurement was taken for five times.

II. Results and discussion

2.1. Powder XRD analysis.

The XRD signals were obtained for pure and CdSO$_4$ doping of 2 %, 5 %, 10 % and 15 % L-Valine compound and it is presented in Figure 1. Different modulated signals were observed for different CdSO$_4$ doping concentration. The PXRD signals for pure and 2 % of CdSO$_4$ are found to be same and other patterns were different. The first set of pattern was obtained in the series of glancing angle of 22°, 24°, 27°, 29°, 33°, 35°, 37°, 45°, and 53° which can be assigned to (030), (001), (100), (131), (111), (121), (200), (220), and (330) planes for pure and 2%. The second pattern was determined to be 23°, 24°, 28°, 29°, 34°, 34°, 38°, 47°, and 51° which can be assigned to (030), (001), (100), (131), (111), (121), (200), (220), and (330) planes for pure and 5%. But for the 10% of CdSO$_4$, the peaks have been observed at 23°, 25°, 28°, 30°, 32°, 36°, 38°, 45°, and 54° which can be assigned to (030), (001), (100), (131), (111), (121), (200), (220), and (330) planes. For 15 % of CdSO$_4$, the peaks were obtained which is similar to the 2 % of doping with minimum peak intensity. From this observation, it was clear that the sample was optimized at 10% of doping in which all the peaks were obtained very clearly with maximum intensity. In addition to that, the peak shift was obliviously right position and it confirms orthorhombic crystal lattice of present metal-sulfate crystal [10-11]. The synthesized crystal doped with CdSO$_4$ and due to which zwitter ionic zones were created and present crystal possess schottky defect which is very favor for generating NLO susceptibility effect. Moreover, defect was also useful to generate anharmonic kinetic energy among electrons and thereby the boosting the frequency of entering ray.

By suiting CdSO$_4$ in interstitial position on the regular lattice, sub planes were observed to be distorted little bit and deviated from good order and thereby the dislocation density in the lattice site was improved. The respective primitive cell parameters were calculated for CdSO$_4$ at 2 %, 5 %, 10, and 15 % with respect to a, b, and c-coordinate directions and are presented in Table 1. By the existence of lattice on molecular cloud, high degree of crystallinity configuration observed in crystal, the space group was calculated to be $P2_12_12_1$ and screw axes intersect in space were found and thus two dimensional defects was ensured. As per the estimated data, the crystal parameters for 2 %; a = 5.580 Å, b = 7.011 Å, and c = 5.091 Å and crystal dimension was $\alpha = \beta = \gamma = 90°$, for 5 %, a = 5.599 Å, b=7.831 Å, and c =5.659 Å, for 10 %, a=5.592 Å, b=7.831 Å, and c = 5.822 Å. Along with all, the observed values of pure and 15 % of doping were found to be same.It was also ensured that, as prepared metallic sulfate crystal was with limited dimensions. The fluctuated parametric values in x, y, and z coordinate showed all sub planes, have rather extended by metal atoms can free to dislocate as frenkeldefect. By verifying all parameters, the volume of unit cell for 2 %, 5 %, 10 % and 15 % was 199.16 Å^3, 249.87 Å^3 and 250.37 Å^3 respectively, Illustrates the complete dimensions of unit cell and low dense packing arrangement in the crystal is depends on the doping concentration. It is defective crystal due to metal sulphate and doping makes positive effect on

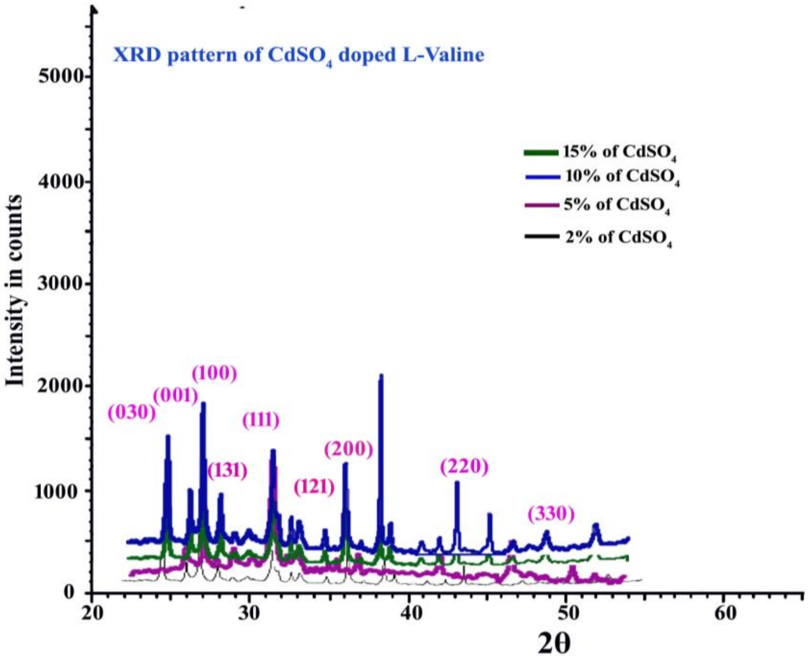


Fig. 1. XRD diagram.

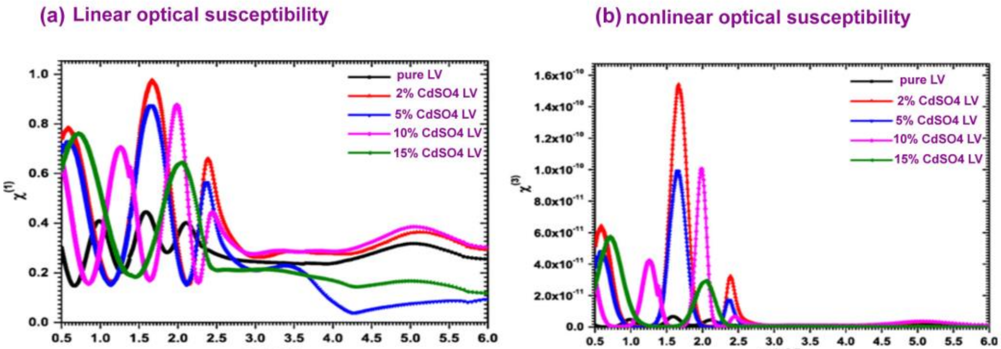
Table 1.

Crystalparametersof $CdSO_4$doped L-Valine

S. No.	Parameters	Values			Structure/Crystal
1	Spacegroup	$P2_1P2_1P2_1$			
2	Meltingpoint Stoichiometry Frameworkgroup Deg. offreedom	281°C C5H10CdNO6S(2) C1[X(C5H10CdNO6S)] 66			
3	Unitcell	2% $CdSO_4$	5% $CdSO_4$	10% $CdSO_4$	
	a	5.580	5.599	5.592	
	b	7.011	7.831	7.612	
	c	5.091	5.659	5.822	
4	Refractiveindex				
	n_1	1.561	1.588	1.661	
	n_2	1.612	1.661	1.693	
	n_3	1.633	1.699	1.593	
5	$\alpha = \beta = \gamma$	90°/Orthorhombic			
6	Volume	199.16Å^3	249.87 Å^3	250.37 Å^3	
7	Birefringence (Δn) Kλ/t	0.078-0.356			
8	NLO efficiency	3.298-4.889 $I^{2\omega}= X\ I^{2\omega}$			
9	Transmittance	0.345-1.729 μm			
10	complexity	611-697			

the crystal property. The non linear optical susceptibility was measured for all doping concentration and it is displayed in Figure 2. As per the linear optical response (2 (a)) of the medium of different doping concentration, the response curves confirmed the optical Kerr effect and it was effective with respect to the doping level of $CdSO_4$. The very effective non linear responsibility was obtained for the sample of doping level 10 % whereas rest of others are low. The non linear refractive index (2(c)) was also confirmed the cross-phase modulation, and Raman amplification existed in the material medium. The third order non linear optical susceptibility was measured for 2 % and 10 % of doping level where the effective non linearity was observed for 10% of doping concentration. As in the Figure 3, the effective production of higher order magnetic dipole moments called susceptibility emphasized with very good responsive at 492 nm.

As the present crystal is NLO active, positive birefringence effect was fully observed and it was calculated for all doping concentration. It was ranged from 0.078 - 0.356 which is narrow range that facilitates the tuneable ability with different frequency and it was also proved by obtaining different refractive indices for different planes; 1.561, 1.612, and 1.633 for 2 %, 1.588, Non linear optical susceptibility



(a) Linear optical susceptibility

(b) nonlinear optical susceptibility

(c) nonlinear refractive index

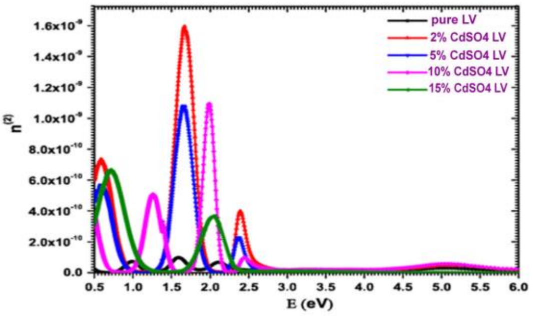


Fig. 2. NLO Susceptibility.

1.661, and 1.699 for 5 % and 1.661, 1.693, and 1.593 for 10 %. From this observation, it was observed that the plane Polarization induced by the effect of schottky defect along with molecular Scattering. As per the obtained results, the extraordinary ray gets additional frequency when compared with ordinary ray and the plane vibrations of molecules in interplanes are not parallel. So, the polarization of planes can be operated by electric and magnetic vector components and faithful birefringence effect was produced. Therefore, it is possible to change refractive indices and the birefringence can be controlled by operating voltage on crystal. As per the previous work, NLO efficiency of SiO_2 was measured and it was as 3.298, whereas it was calculated to be 3.298-4.889 for the present case and it was higher than popular semiconductor. This is only possible only by the effect of phonon momentum of lattice. The photon operated mechanism was estimated as optical-transmittance and it was calculated to be 0.345 - 1.729 µm and such range was also belongs to UV to IR regions. So the laser activity was started at UV and ended with IR via visible. It was also observed that, the frequency of activity can be amplified between two ranges. The complexity of as prepared crystal was ranged from 611 - 697 which are very uniqueto establish active inter-molecular energy to be opt for generating laser light.

2.2. Laser measurement

The present crystal is laser active since the good response of NLO susceptibility has taken in effect. Usually the NLO efficiency is to be calculated to evaluate NLO calibre which is very significant to interpret the non linearsusceptible nature. For such measurement, second and third order NLO dataacquired from Z-Scan measurement using He-Ne laser source setup. Here, the DF length is maintained at 1.49 mm by which the third order non linear optical property is estimated and it is portrayed in Figure 4-6.

Third order non linear optical susceptiblity

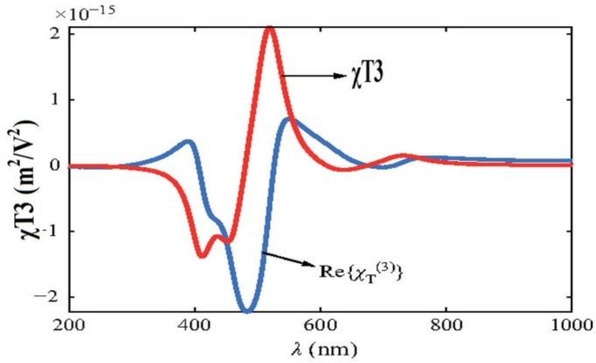


Fig. 3. Thirdorder NLO Susceptibility.

Light dispersion ratio in different coordinates of the crystal medium as third order nonlinear refractive index, optical absorption coefficient and hyper susceptibility [12-14]. In thiscase, the Z-direction and the laser field intensity was determined and it is predominant method to capture third order non linear refractive index (n_2), optical-absorption coefficient (β) and hyper-susceptibility (χ^3) of material medium. It is alsovery important to determine thelateral sign and magnitude of phase change, ΔΦ and also capable of handling optical limiting factor.

Three aperture configurations (closed, open and ratio) are preserved to obtain precise results and the measurement graph is shown in Figures 4-6. The refractive index for third order was traced to be $4.895 \text{ x } 10^{-8}$ cm^2/W. It is very remarkable and the crystal material at doping of 10 % $CdSO_4$ explicit inbuilt non linear susceptibility. As in the Table 2, the nonlinear absorption coefficient was $0.0981 \text{ X } 10^{-4}$ cm^2/W, measured at open aperture measurement ($\beta = (2\sqrt{2}\ \Delta T) / (L_{eff} x I_0)$). The observed data is greater than all reserved for this application and effective for electric field polarization density nonlinearly. The NLO hyper susceptibility ($\chi^{(3)} = [(\text{Re}\ \chi^{(3)})^2 + (\text{Im}\ \chi^{(3)})^2]^{1/2}$) was measured as $7.89 \cdot 10^{-6}$esu and it is reliable to explore volatile birefringence effects.Third order NLO coefficient n_2 was as $-4.895 x 10^{-9}$ cm^2/W for the sample doping at 10%. The Work-hardening coefficient was calculated to be 3.211 that offer remarkable stability of the present crystal medium. The measured hyperpolarizability (β) was determined as $4.011 \cdot 10^{-4}$ cm/W, shows stabilized NLO effect in the crystal medium under the electric vector. All the laser measurement values demonstrates theself defocusing performance of the material medium which is well organized by positive coefficient of refractive index of material in different coordinates. The NLO coefficient on saturation temperament is a good optical coherence ofthe material medium that provideslinear characteristics of output laser power to input power.

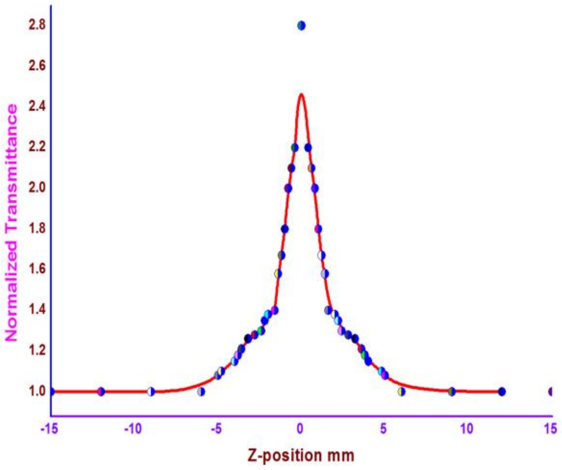


Fig. 4. Z-Position.

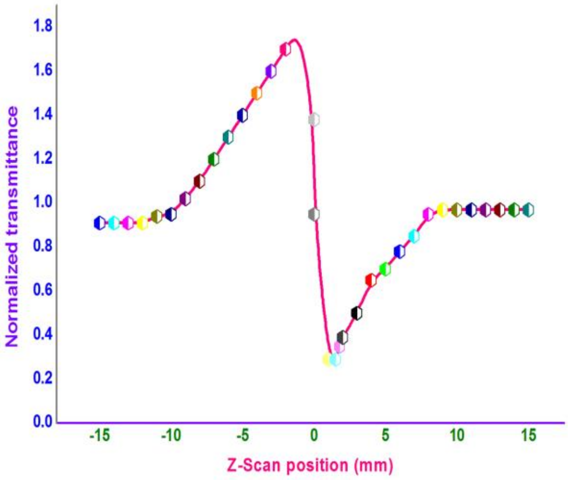


Fig. 5. Z-ScanDiagram.

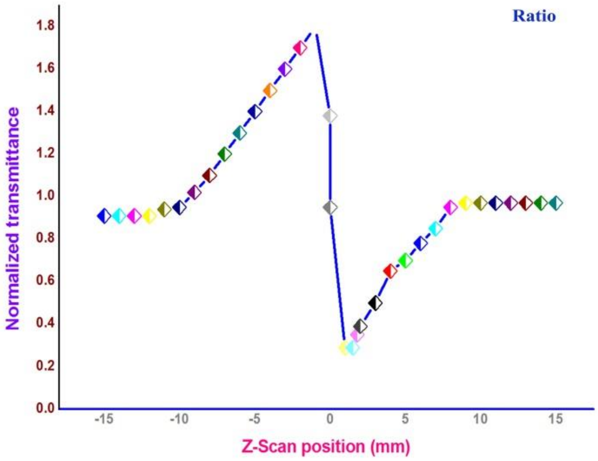


Fig.6. Ratio Z-ScanDiagram.

Table 2.
NLO parameters from Z-Scan measurement for $CdSO_4$ doped L-Valine

S. No.	Parameters	Values
1	Optical path distance (Z)	90 cm
2	Aperture radius (r_a)	2.5 mm
3	Optical path length	50 cm
4	Incident intensity at focus (Z=0)	3.65 GW/cm^2
5	Effective thickness L_{eff}	3.779 mm
6	Linear refractive index (n_0)	1.726
7	Nonlinear refractive index (n_2)	$3.891 \cdot 10^{-8}$ cm^2/W
8	Nonlinear absorption coefficient (β)	$0.0981 \cdot 10^{-4}$ cm^2/W
9	Third order nonlinear optical susceptibility ($\chi^{(3)}$)	$4.621 \cdot 10^{-6}$esu
10	Lower cutoff wavelength	391 nm
11	Work-hardening coefficient	3.211
12	Third order NLO coefficient n_2	$-4.895 \cdot 10^{-9}$ cm^2/W
13	β	$4.011 \cdot 10^{-4}$ cm/W
14	$\chi^{(3)}$	$7.89 \cdot 10^{-6}$ esu

2.3. Raman shift of nonlinear optical effect.

The coherent anti-stokes Raman scattering is generated due to third-order nonlinear susceptibility in the material medium, as the present case is non linear optical crystal, it was measured in Nonlinear Raman spectroscopy techniques. The Raman-active molecular transitions taking place when the molecular material medium involved with inelastic scattering induced by non linear optical effect. So, the third-order nonlinear susceptibility of the material medium can be estimated as much as possible. Here, the Raman shift spectrum was recorded for the sample at pure and doped at 2 %, 5 %, 10 % and 15 % of $CdSO_4$ and it is presented in Figure 7. The Raman peak of L-Valine crystal was observed at 310 cm^{-1} with weak intensity which was assigned to C-C-C bonds. Whereas, for the L-Valine doped with $CdSO_4$, the respective Raman peak was observed at same wave number but it was assigned to Cd-S bond with moderate intensity. Moreover, the same peak was observed with very strong intensity for doping level at 10 %. Here, the intensity was improved by the increment of force constant of Cd-S bond and thereby the non Centro-symmetry was improved by such availability of bonds in the material medium. The ultimate impact of such non Centro-symmetry in the material significantly develops the non linear susceptibility on the material medium. But, at 15 % of $CdSO_4$, the peak intensity was observed to be decreased much which indicates the order of the crystallinity was decreased at that percentage of doping. From this observation, it was clear that the non linear property was effectively optimized at 10 % of $CdSO_4$ doping on L-Valine.

2.4. Vibrational study.

The change of dipole moment with respect to the electric and magnetic vectors of the light in the material medium explains the vibrational transitions of the heteronuclear bonds of the molecular medium. Here, the availability of the hetero nuclear bonds is more than homonuclear bonds. So, change dipole moment during the vibrational transitions can be measured by the number of the IR and Raman bands available with maximum intensity at far IR and mid IR regions. The recorded IR spectrum for pure L-Valine, doped with $CdSO_4$ at 2 %, 5 %, 10 % and 15 % were depicted in Figure 8. The first spectrum showed the spectral lines of pure and doped with $CdSO_4$ at 2 %, 5 % and 15 % where no spectral line observed with maximum intensity that showed no vibrational transitions taking place at far and mid IR region. It clearly indicates no active heteronuclear bonds such as Cd-S, S-O (dipole moments) present. Whereas in the case of 10 % of doping sample, several peaks with very strong intensity available in far and mid range of IR spectrum. This explicit the crystal with well ordered

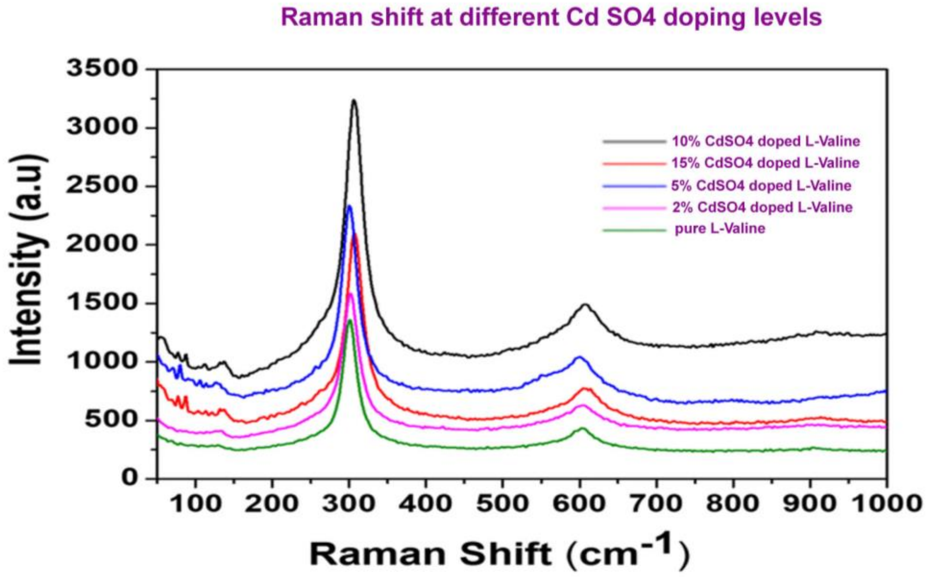
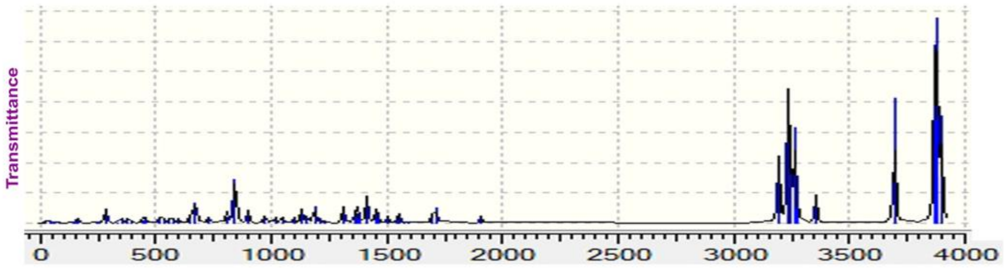


Fig. 7. Ramanspectrum.

(a) IR spectrum 2,5,15 % of CdSO4 doped L-Valine



(b) IR spectrum 10 % of CdSO4 doped L-Valine

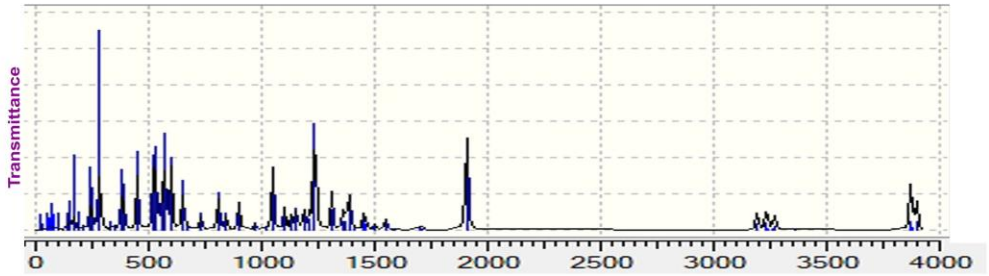


Fig. 8. IR spectrum.

crystallinity of the perfectly doping is found.

2.5. SEM examination.

The surface of the as prepared crystal at 10 % of $CdSO_4$ was magnified at 20, 10, 5 and 2 μm and is displayed in Figure 9 in which width was measured and it was found to be 15.24mm. The SEM image at 20 μm explicit cascade plates like crystal and Pico range porosity like structure was found at 10μm where the void fractions was appeared. The well formedimage at 5 and 2μm was showed smooth surface and the images clearly detailed the amalgamation of organo-metallic medium.

2.6. DSC-TGA studies.

The DSC and TGA curves for present crystal are portrayed in Figure 10 where the thermal stability was clearly smooth and the crystal undergoes an irreversible exothermic transition at 226 °C, this is the λ-point at which decomposition starts. At such temperature,

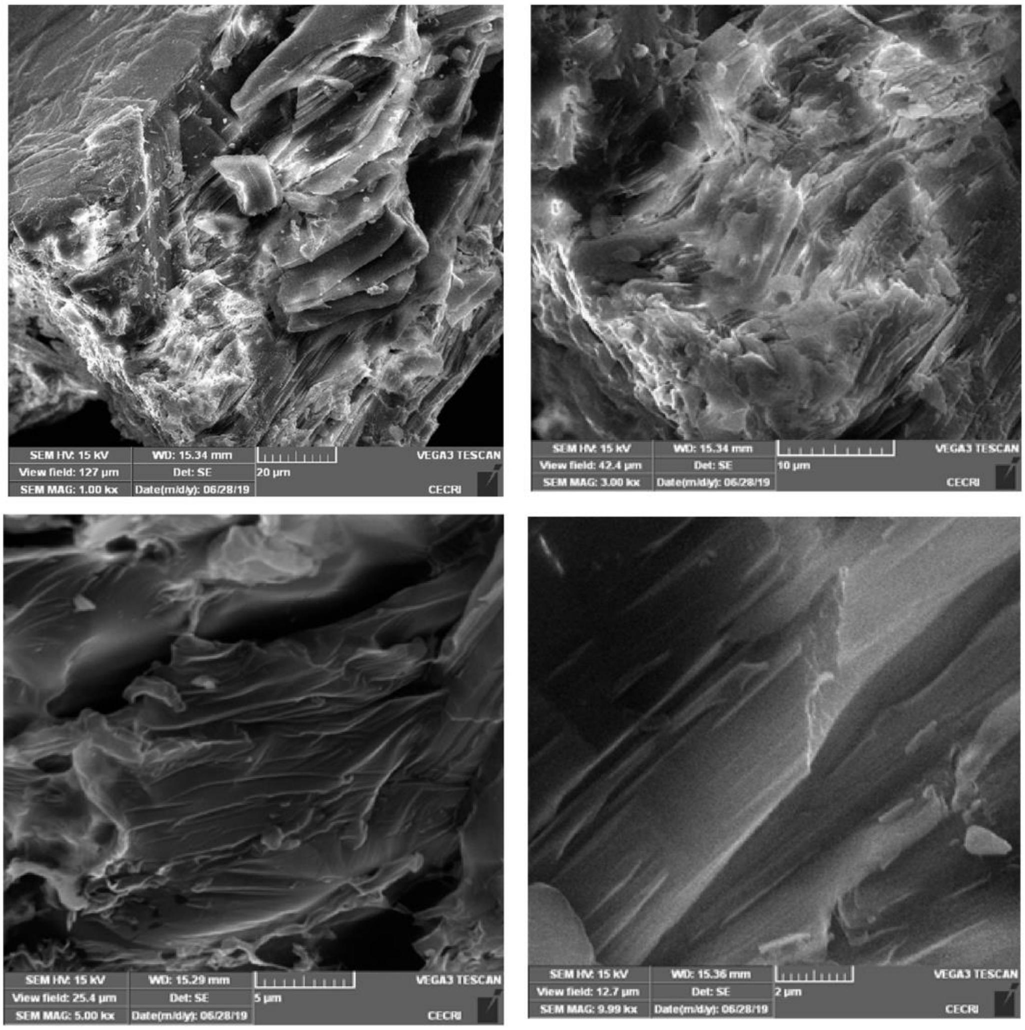


Fig. 9. SEM images.

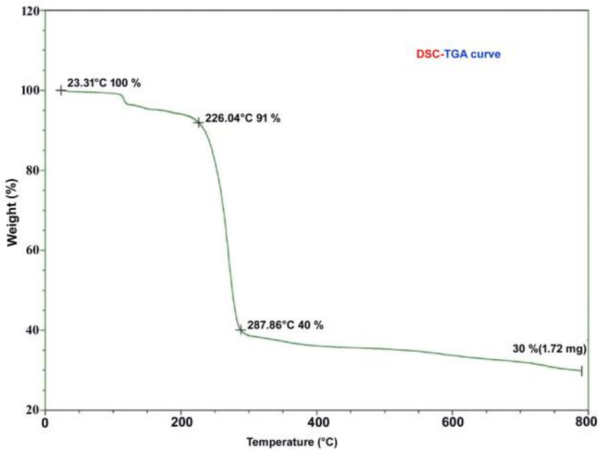


Fig. 10. DSC-TG curve.

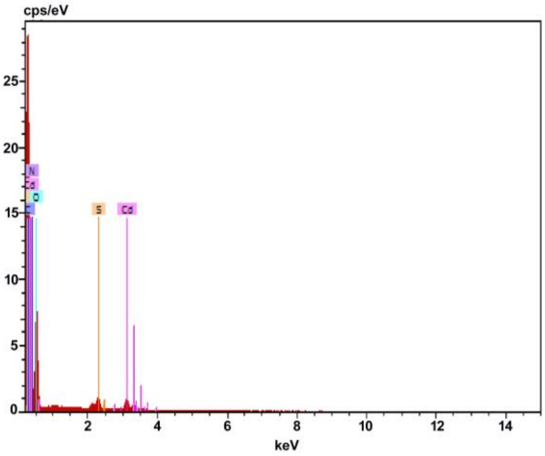


Fig. 11. EDAX spectrum.

thermal stability was decreased and this point onwards the hardness of the crystal was leered. L-Valine was decomposed at 287 °C whereas when it composed with $CdSO_4$, its decomposition was sustained to 700°C. This exothermic peak demonstrated the good structure of crystallinity of the synthesized crystal. The phase change from liquid to vapour state was evidenced by thepeak at 287° C, showed loss of mass.

2.7. EDAX examination

The EDAX graph is nothing but energy dispersion of compositional elements of crystal and is displayed in Figure 11. The elemental composition was detailed in same figure also. 1KeV peak showed organic species whereas the peak at 2.4 KeV showed metal sulphate. The peaks for elemental composition; C, O, N, Cd was observed at 48.88, 30.78, 16.83, 2.72 and 0.88 % clearly illustrated the interactive pressure in energy dispersion.

Conclusion

The $CdSO_4$ doped L-Valine crystal with doping concentration level; 2 %, 5 %, 10 % and 15 % weresynthesized and the crystal samples were characterized. The XRD signals from various planes ensured the orthorhombic lattice structure and the structure was found to be very strong at 10 % of $CdSO_4$ doping. The shift of XRD peaks established the crystallinity is moved to be perfect. The stable formation of crystal was recognized at 10 % of doping sample and it proves the unique stability. The optical Raman bands of sample at 10 % of doping showed the intensive nonlinear susceptibility of the material medium. The predominant molecular packing in orthorhombic lattice structure was displayed and the respective parameters showed the presence of optical Kerr effect. The NLO efficiency was estimated for all samples and itwasfound to be considerably higher at 10 % of $CdSO_4$ doped L-Valine than KDP crystal. The observation different refractive indices on different coordinates confirmed the availability of non-centro-symmetry properties. The DTA-TG graphs layout the hardness of the synthesized composite and EDAX served to find the compositional parts of the crystal.

***Susithra G.* –** M.Sc., MPhil, Ph.D.;
***Vidhya D.* –** M.Sc., MPhil, Ph.D.;
***Ramalingam S.* –** M.Sc., MPhil, Ph.D**.**

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Г. Сусітра, Д. Відх'я, С. Рамалінгам

Легуюча домішка (Cd)SO_4у кристалах L-Валіну для покращення оптико-електронних і лазерних властивостей

Кафедра фізики, A.V.C. Коледж, Майіладутурай, Тамілнаду, Індія

L-валін відноситься до добрих молекулярних платформ щодо індукції властивостей NLO для лазерних застосувань, причому, певні властивості лазера можна покращити шляхом введення відповідних груп заміщення, керуючи при цьому електронну підсистему. Ця стаття є спробою покращити властивості лазера шляхом збільшення концентрації розчинника Cd на сполуці L-валіну. Спостережені піки XRD вказували на значне покращення лазерних властивостей композитного кристала. Результати, отримані для чистого L-валіну наступні: a = 5,221 (Å), b = 6,910 (Å) і c = 4,994 (Å), а відповідні показники заломлення складали n_1 = 1,511, n_2 = 1,583 і n_3 = 1,599. Для 2 % $CdSO_4$ отримано: a = 5,580 (Å), b = 7,011 (Å) і c = 5,091 (Å), а відповідні показники заломлення були n_1 = 1,561, n_2 = 1,612 і n_3 = 1,633. Для 5 % $CdSO_4$: a = 5,599 (Å), b = 7,831 (Å) і c = 5,659 (Å), а відповідні показники заломлення скоали n_1 = 1,588, n_2 = 1,661 і n_3 = 1,699. Для 10 % $CdSO_4$: a = 5,592 (Å), b = 7,612 (Å) і c = 5,822 (Å), а відповідні показники заломлення дорівнювали n_1 = 1,661, n_2 = 1,693 і n_3 = 1,593. Для 15 % $CdSO_4$: a = 5,551 (Å), b = 6,332 (Å) і c = 4,734 (Å), а відповідні показники заломлення n_1 = 1,501, n_2 = 1,599 і n_3 = 1,593. Нелінійний показник заломлення змінювався від $3{,}891\cdot10^{-8}$ см^2/Вт до $4{,}231\cdot10^{-8}$ см^2/Вт для 2 %, 5 %, 10 % відповідно, а нелінійна оптична сприйнятливість третього порядку (χ(3)) змінювалася від $4{,}621\cdot10^6$esu до $5{,}091\cdot10^6$ esu. Виявлено, що відновлений хімічний потенціал для нелінійних оптичних (NLO) властивостей і для подвійного променезаломлення, покращився. Ідентифіковано розсіювальну здатність гомоядерних і гетероядерних зв'язків разом із пустотами $CdSO_4$ для досягнення нелінійного процесу розсіювання.

Ключові слова: $CdSO_4$, подвійне променезаломлення, сприйнятливість, нелінійний показник заломлення, EDAX.