DandaoXuebao/Journal of Ballistics
ISSN: 1004-499X
Vol. 38 No. 1 (2026)

Insight Into Crystal Growth and Optical, Morphological, Compositional Properties of Glycine Oxalate Dihydrate (2:1 Molar Ratio) Single Crystals for Third Order Nonlinear Optical Applications

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Abstract

Glycine oxalate dihydrate crystals in a 2:1 molar ratio were obtained through using the slow solvent evaporation technique at ambient conditions. The method yielded well-formed, high-quality single crystals over a controlled period, demonstrating reproducible growth under ambient conditions. These crystals provide a reliable material for further studies on their third-order nonlinear optical applications. The nonlinear optical properties of the grown single crystal were investigated using the Z-scan technique. The open and closed aperture Z-scan measurements demonstrates a clear nonlinear absorption and refractive behaviour under laser excitation, confirming the presence of third-order nonlinear optical material. These results indicate the potential of the material for nonlinear optical device applications. SEM was utilized to analyze the surface morphology of the grown single crystals. The SEM images reveal well-defined crystalline grains with faceted, layered, and plate-like morphologies, indicating good crystallinity and anisotropic growth behaviour. The compact and uniform surface features confirm the efficient formation of good-quality single crystals. Energy-dispersive X-ray (EDX) analysis was performed to determine the elemental composition of the sample. The results indicated the presence of (C), (N), and (O), with weight percentages of 42.83 %, 20.19 %, and 36.97 %, respectively, reflecting the accurate and reliable quantification of these elements in accordance with standard compounds. The UV-DRS studies was performed to investigate the optical behaviour of the synthesized material through absorbance, and transmittance studies. The refractive index and reflectance spectra further elucidate the dielectric nature and optical stability of the material.

Keywords: Optical, Z-Scan, SEM, EDAX, Photonics and Nonlinear optical applications

1). Introduction

Glycine oxalate dihydrate (2:1 ratio) is an organic molecular crystalline compound formed by the stoichiometric combination of the amino acid glycine and oxalic acid dihydrate **[1, 2]**. Amino acid-based crystals have gained significant attention owing to their inherent biocompatibility, structural flexibility, as well as their capacity to establish stable hydrogen-bonding interactions within the crystal lattice **[3]**. Glycine exists in a zwitterionic form and readily participates in intermolecular interactions, while oxalic acid acts as an efficient proton donor, facilitating strong ionic and hydrogen-bonding interactions within the crystal lattice **[4, 5]**. This association of two glycine molecules with one oxalate unit and water molecules promotes enhanced molecular packing and structural stability, which is instrumental in the nucleation and growth of well-defined single crystals **[6]**.

In the present investigations, glycine oxalate dihydrate (2:1 ratio) single crystals were successfully prepared using the slow evaporation solution growth method under room temperature conditions **[7, 8]**. The

surface morphology of the prepared crystals was analyzed using SEM, and the elemental composition was establish by means of EDX characterization **[9]**. The third-order nonlinear optical (NLO) response, combined with good crystalline quality, suggests that glycine oxalate dihydrate is a promising material for nonlinear optical applications including optical limiting, optical switching, and photonic device fabrications **[10]**.

2). Synthesis method

Single crystals were synthesized using glycine and oxalic acid dihydrate as precursor materials in a (2:1 molar ratio). The accurately weighed reactants were dissolved in deionized water to prepare a clear solution. The resulting mixture was subjected to constant agitation for 5 h with the aid of a magnetic stirring apparatus to achieve complete dissolution and compositional uniformity. Upon completion of the agitation process, the solution underwent filteration through whatman filter paper to eliminate residual insoluable particulates. The clear filterate was subsequently allowed to remain quiescent in a contamination-free ambient environment at room conditions to promote gradual solvent evaporation. Controlled evaporation led to the formation of transparent and well-defined single crystals, confirming the successful synthesis of the material.

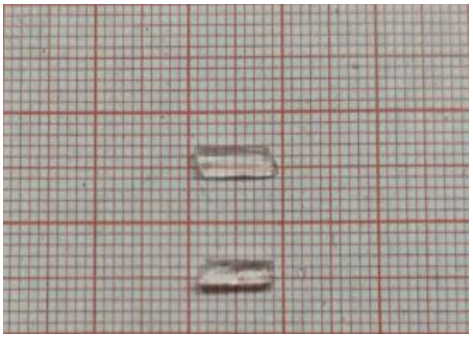

Fig. 1 (a) Photographic picture for GOD (2:1 Ratio) single crystals

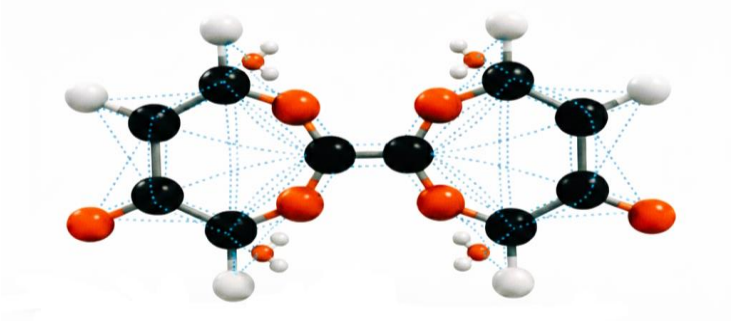

Fig. 1 (b) Molecular structure of glycine oxalate dihydrate (2:1 molar ratio)

3). Results and discussion

3.1). Z-scan

The Z-scan studies is a well-established and sensitive employed analytical technique evaluating the third-order nonlinear optical characteristics of crystals [11, 12]. It enables quantitative determination of simultaneous nonlinear absorption and nonlinear refraction by transversely translating the specimen across the focal plane of an incident focused Gaussian laser beam and recorded the transmitted intensity [13]. In this technique, an open aperture configuration is utilized to determine the nonlinear absorption coefficient (β), while a closed-aperture measurement provides information about the nonlinear refractive index (n2).The method offers high precision in quantifying the magnitude and sign of the nonlinearities, including reverse saturable absorption, saturable absorption, self focusing, and self defocusing effects.owing its experimental simplicity and effectiveness, the Z-scan technique has become a standard characterization technique for technological applications such as optical limiting, laser protection, all-optical switching, and other nonlinear photonic devices [14].

A Polarized Gaussian laser beam operating in the fundamental TEM00 fundamental mode was directed through a

plano-convex lens with a focal length of 200 mm, thereby producing a tighlty focused beam waist (w_o) of 8.95 μm at the focal plane **[15, 16]**. The sample under investigation had a physical thickness (L) of 1mm, while the corresponding Rayleigh length (Z_R) of the focused beam was theortically estimated 0.398 mm. During the experiment, the laser beam was propagated along the negative to positive z-axis (from –z to +z), and it was observed that the sample thickness exceeded the Rayleigh length (L>Z_R), satisfying the required condition for the intended nonlinear optical measurements **[17]**. This alignment ensured that the light matter-interaction between the incident laser radiation and the material occurred within the effective focusing region, allowing for precise characterization of the sample's optical properties.

The third-order nonlinear optical parameters of the grown single crystal were systematically examined employing the Z-scan method at a wavelength of 632.8 nm. Based on the closed aperture Z-scan results, the nonlinear refractive index (n_2) was determined as 1.76 x 10^{-16} m^2/W, revealing a measurable third order nonlinear refractive response **[18]**. The nonlinear absorption coefficient (β) was evaluated as 1.29 x 10^{-8} m/W, using these values, the real of the third-order nonlinear optical susceptibility were computed as χ Real(3) = 6.69 x 10^{-15}esu, and imaginary of the third order susceptibility was determined as χ img (3) is 1.65 x 10^{-12}esu, respectively. Consequently, the total magnitude of the third-order susceptibility was determined to be χ (3) = 1.65 x 10^{-12}esu, indicating that the sample demonstrate strong nonlinear absorption dominated behaviour, with a measurable nonlinear refractive contribution, highlighting its potential suitability for applications in nonlinear optical devices such as optical limiting, switching, and photonic modulation **[19]**.

The nonlinear optical behavior of the crystal was further characterized through open and closed aperture Z-scan measurements. The open-aperture Z-scan exhibited a pronounced valley with no peak, demonstrating the existence of reverse saturable absorption (RSA) and revealing strong intensity-dependent nonlinear absorption behaviour within the material. In contrast, the closed-aperture Z-scan displayed a distinct peak-valley configuration, corresponding to a positive nonlinear refractive index and demonstrating self-focusing behaviour. Together, these observations confirm that the material indicates both significant nonlinear absorption and measurable nonlinear refraction, suggesting its suitability for device applications in optical limiting, laser protection, and all-optical switching **[20]**.

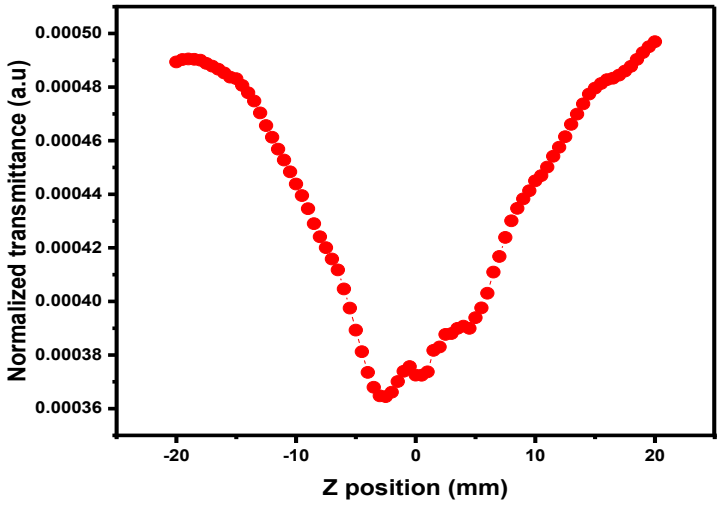

Fig. 2 (a) Z scan – Open aperture

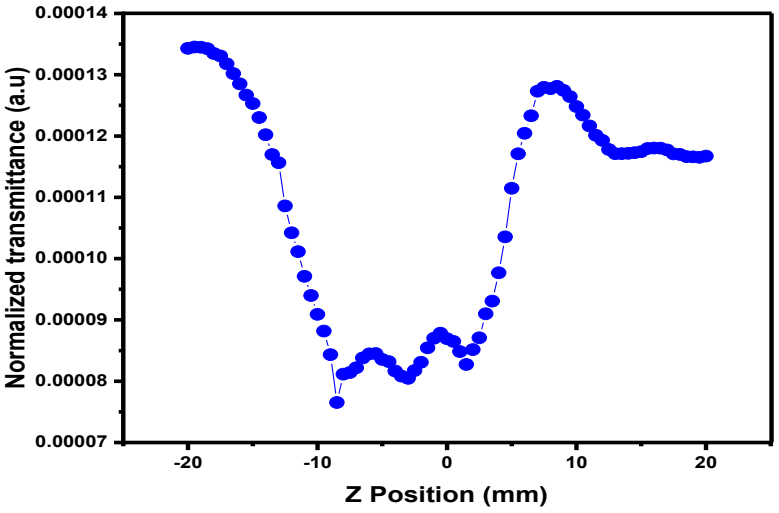

Fig. 2 (b) Z scan – Closed aperture

Table. 1 Nonlinear optical parameters of the prepared sample obtained from Z-Scan measurements

Parameter	Value
Sweeping Distance	40.0 mm
Step Distance	0.5 mm
Bandwidth	High
Wavelength (λ)	632.8 nm
Power of the Laser (E_P)	12.0 mW
Diameter of the Laser beam (d)	5 mm
Focal Length of the Lens (f)	200 mm
Radius of the Aperture (r_a)	2 mm
Radius of the beam at Aperture (w_a)	4.5 mm
Z-Scan Sample Thickness (L)	1 mm
Linear refractive index (n_o)	1.5
Transmittance (T)	70 %

3.2). UV-DRS analysis

The UV-DRS spectroscopy of the prepared material was acquired over the spectral range of 200-800 nm to evaluate its optical behaviour. The material exhibits a lower cut-off wavelength at 312 nm, indicating good optical transparency across the visible spectral region. The absorption observed near the cut-off region is attributed to electronic transitions, primarily (n-π*) and (π-π*) transitions associated with the molecular functional groups. These transitions arise from non-bonding electrons of hetroatoms (-NH_2, -COOH) and the electrons of carbonyl (C=O) groups. The absence of significant absorption within the visible spectral region demonstrate the applicability of the crystal for optical device applications **[21]**.

The optical transmittance of the material is found to be high throughout the visible spectrum, which indicates minimal scattering and defect concentration in the grown sample. The good transparency suggests that the material possesses low optical loss and is appropriate for photonic and opto-electronic applications.

The dispersion of refractive index as a function of wavelength exhibits normal dispersion characteristics, in which the refractive index gradually decreases with increasing wavelength. This behaviour indicates the typical dielectric response of the material in the optical region. Similarly, the reflectance spectrum recorded over the studied wavelength range shows a systematic variation with photon energy. These optical characteristics further confirm the dielectric nature, optical homogeneity, and stability of the synthesized crystal **[22]**.

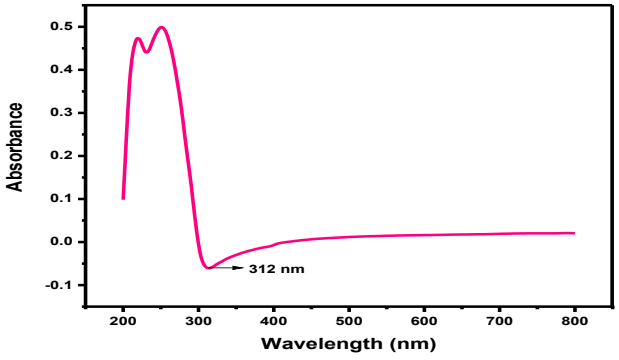

Fig. 3 (a) UV DRS – Absorbance

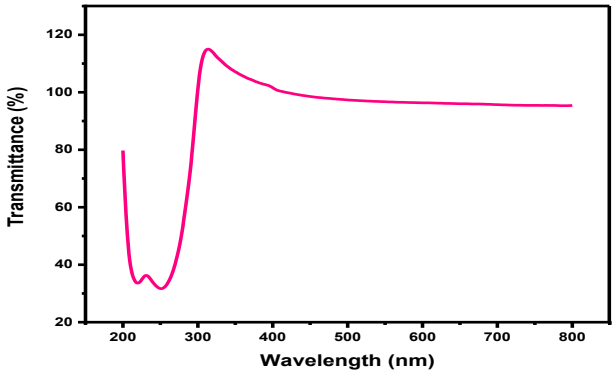

Fig. 3 (b) UV DRS – Transmittance (%)

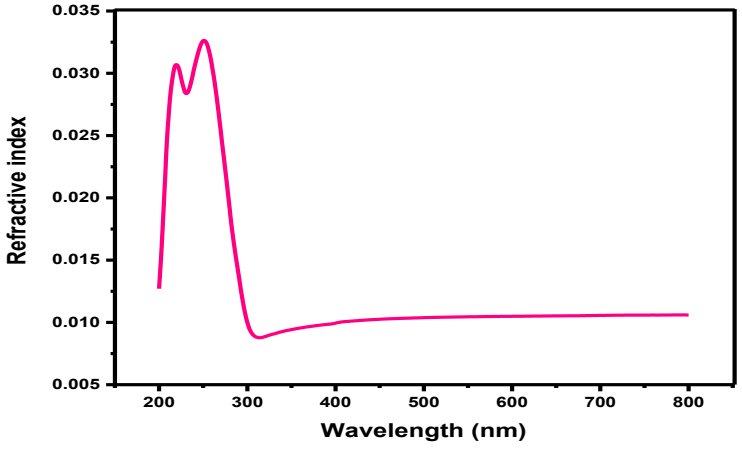

Fig. 3 (C) UV DRS – Refractive index

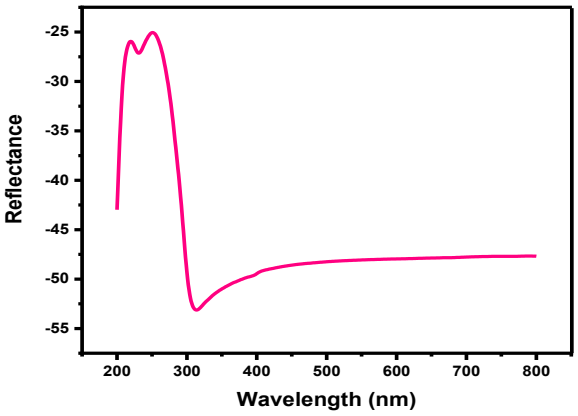

Fig. 3 (d) UV DRS - Reflectance

3.3). Energy dispersive X-ray analysis

EDS studies of the sample revealed the detection of carbon (C), nitrogen (N), and oxygen (O), with all elements detected using their respective K-series lines **[23]**. The apparent concentrations of C, N, and O were measured as 8.40 %, 6.00%, and 8.01% respectively, corresponding to K ratios of 0.08400, 0.01069, and 0.02694. The calculated weight percentages were 42.83 wt% for C, 20.19 wt% for N, and 36.97 wt% for O, summing to a total of 100.00 wt%. The associated standard deviations (σ) were 0.90, 1.33, and 0.83 for C, N, and O, respectively, indicating reliable measurements **[24, 25]**. Comparision with factory standards identified the elements as C (vitamin C), N (boron nitride, BN), and O (silicon dioxide, SiO_2), confirming the accuracy and validity of the EDS analysis **[26, 27]**.

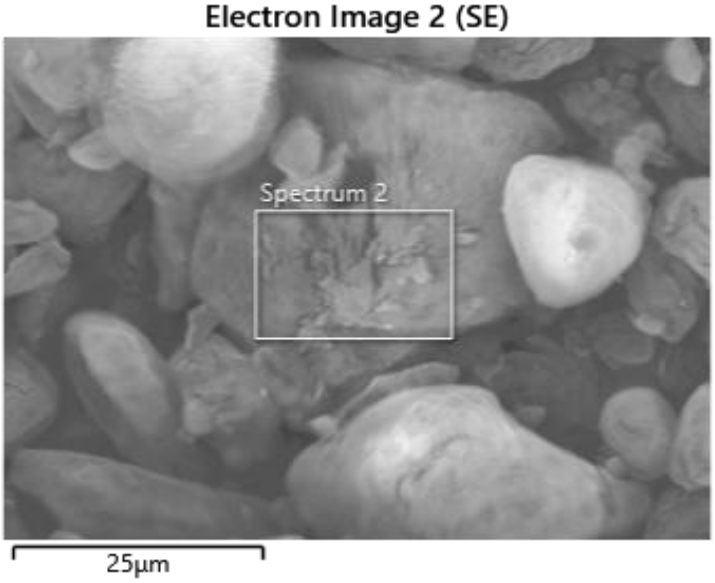

Fig. 4 (a) Electron image of EDX spectrum in the grown crystal

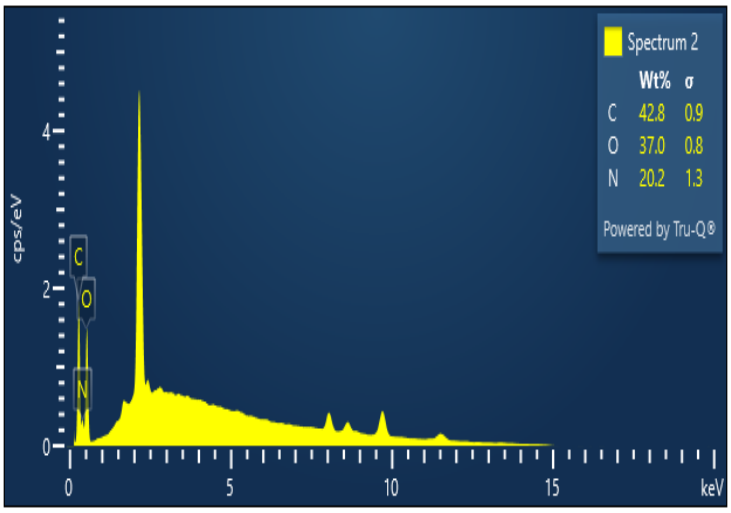

Fig. 4 (b) EDX spectrum of the grown crystals

Table. 2: Energy-dispersive X-ray (EDX) atomic compositional of the grown crystal

Elements	Signal type	Line	Apparentconcentration	K Ratio	Wt %	Wt % sigma	Standard name	Factory standard
C	EDS	K series	8.40	0.08400	42.83	0.90	C Vit	Yes
N	EDS	K series	6.00	0.01069	20.19	1.33	BN	Yes
O	EDS	K series	8.01	0.02694	36.97	0.83	SiO_2	Yes
Total					100.00			

3.4). Scanning electron microscopy

The surface topography and microstructural features of the grown single crystals were investigated through SEM, the acquired micrographs are illustrated in Fig. 5 (a-e) at different magnifications. The SEM image shown in Fig. 5 (a) reveals irregularly shaped crystalline grains with sharp edges and fractured surfaces, indicating the formation of well-developed crystals with a distinct crystalline nature **[28]**. The observed non-uniform grain boundaries suggest the occurrence of multiple nucleation sites during the initial stages of crystal growth**.**

Fig. 5 (b) illustrates the SEM micrograph recorded at a higher magnification, were plate-like crystals with clearly defined facets and edges are prominently observed. The presence of such faceted structures confirms the anisotropic growth behaviour of the crystals and indicates preferential growth along specific crystallographic planes. The absence of excessive agglomeration further demonstrates the controlled growth process and improved crystallinity of the crystals **[29].**

The SEM micrograph depicted in Figure. 5 (c) shows layered and stacked crystal structures with uneven and stepped surfaces. These morphological features suggest a stepwise layer growth mechanism, which is widely observed in solution-growth single crystals. The presence of surface steps and terraces indicates gradual deposition of growth units during crystallization, leading to the development of ordered crystal structure **[30]**.

Fig. 5 (d) presents a magnified view of the crystal surface, revealing smooth, wavy patterns along with embedded crystalline formations. These surface features can be attributed to growth striations formed due to fluctuations in supersaturation and temperature during the growth process. Such striations are characteristic of solution-grown crystals and provide evidence for continuous and stable crystal growth

The SEM micrograph shown in fig. 5 (e) exhibits a compact and densely packed surface morphology with fine micro-cracks distributed across the surface. The observed micro-cracks may arise from internal stress developed during the drying or post-growth handling of the crystals. Nevertheless, the overall compactness and uniform grain distribution indicate good structural stability and homogeneity of the prepared material **[31]**.

Overall, the SEM analysis confirms the formation of well-developed crystalline structures with distinct morphological features, good surface uniformity, and anisotropic growth behaviour, clearly indicating the efficient formation of good-quality single crystals **[32]**.

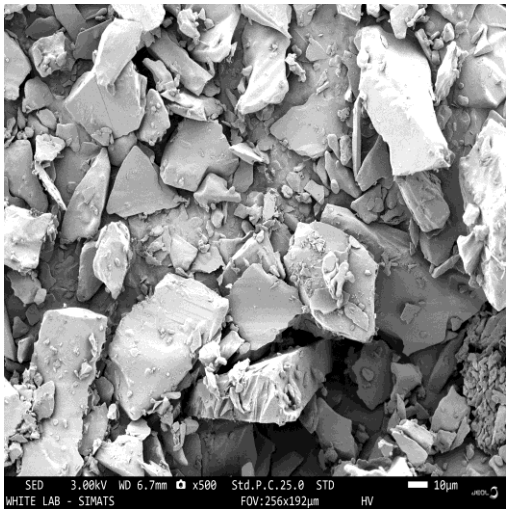

Fig. 5 (a) SEM picture

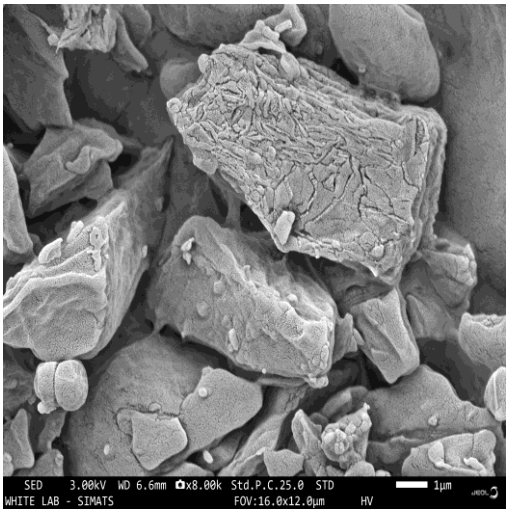

Fig. 5 (b) SEM picture

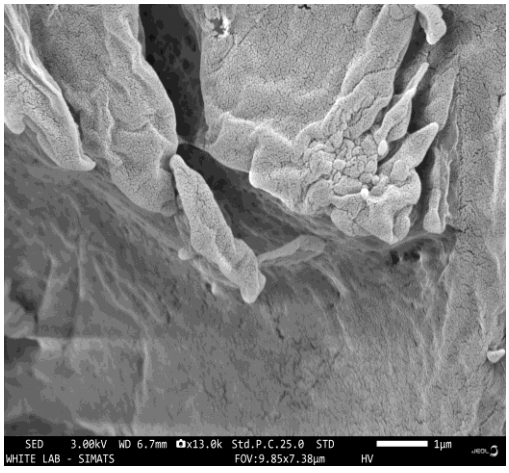

Fig. 5 (c) SEM picture

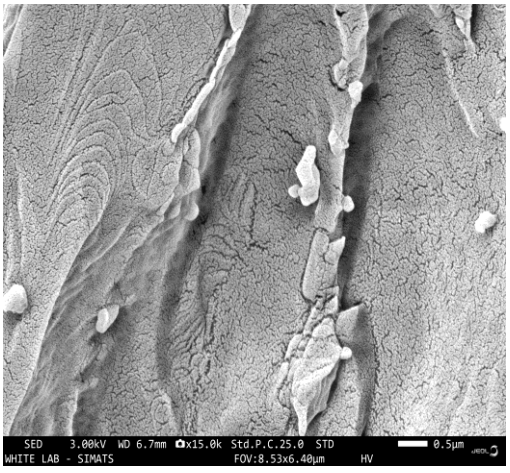

Fig. 5 (d) SEM picture

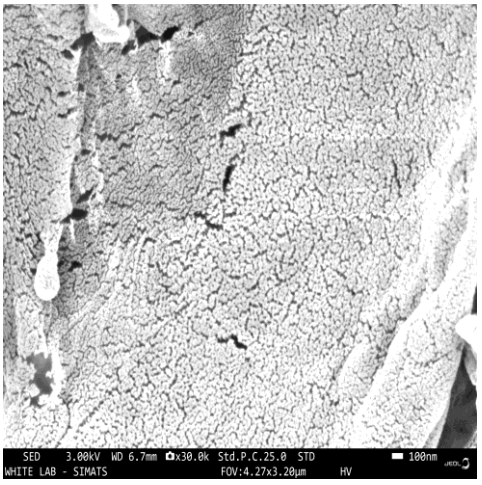

Fig. 5 (e) SEM picture

Conclusion

Single crystals of glycine oxalate dihydrate (2:1 ratio) were successfully synthesized using the slow solvent evaporation solution growth method at ambient conditions. Obtained crystals exhibited good transparency and crystalline quality.EDX spectroscopy established the presence of carbon, nitrogen, and oxygen in the grown glycine oxalate dihydrate (2:1 ratio) crystals, consistent with their expected chemical composition. The absence of impurity-related peaks indicates good material purity. These results support the successful formation of the compound. The SEM analysis demonstrates that the grown single crystals possess well-defined morphologies, including faceted, layered, and plate-like structures, with uniform grain distribution and compact surfaces. The observed features indicate good crystallinity, anisotropic growth behaviour, and structural stability. Z-Scan studies revealed a measurable third-order nonlinear optical response of the synthesized material. The nonlinear refractive index (n_2) was established to be 1.76 x 10^{-16} m^2/W, while the nonlinear absorption coefficient (β) measured 1.29 x 10^{-8} m/W. The calculated third-order nonlinear optical susceptibility (χ (3)) of 1.65 x 10^{-12}esu confirms the suitability of the crystals for nonlinear optical device applications. SEM and EDAX analyses confirmed the excellent crystalline morphology and compositional purity of the compound, and the results indicate that the materials suitable for further nonlinear optical applications. The UV-DRS analysis shows an absorption Lower cut-off around 312 nm and good optical transparency across the spectral region. The absorbance, transmittance, refractive index, and reflectance studies confirm the stable dielectric and optical nature of the prepared material. Thus, the UV-DRS studies of the prepared sample confirm its potential suitability for optoelectronic applications.

Author Contribution

N. Rajasekar : Writing - original draft preparation, funding acquisition, validation, software, formal analysis
K. Balasubramanian : writing - review and editing, visualization, supervision, investigation

Data Availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflict of interest

The authors declare no conflict of interest.

Funding Declaration

This research received no specific grant from any funding agency and the authors received no financial support for the research, authorship, and/or publication of this article

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