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Growth and character revolutionize of Non-Linear Optical crystal by the impact of fertilizer on primary synthesis process of γ- $C_2H_5NO_2$ from $(NH_4)_2SO_4$, dihydrated $CaCl_2$ as solvents

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ABSTRACT

A semi-organic Non-linear optical urea doped γ-glycine single crystal from ammonium sulphate ($(NH_4)_2SO_4$) and calcium chloride ($CaCl_2$) as solvents was synthesized by the molecular ratio of 1:1:1 and 4:1:1 allow growing to get seed form the super saturated solution by slow evaporation techniques. The burnt of urea disturb the growth process of gamma glycine within the limited period. It was confirmed by SEM studies and EDX investigation.

Keywords: semi-organic NLO crystal, slow evaporation techniques, urea doped, SEM, EDX analysis

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INTRODUCTION

Glycine is well known amino acid crystallizes from its aqueous solution in different polymorphic forms. So far, there were three different polymorphic forms reported for glycine ($C_2H_5NO_2$), metastable α, unstable β and the stable γ at ambient conditions [1]. γ-glycine crystallizes in *trigonal space group* with cell parameters a=b=7.19(5) Å, c=5.60(4) Å α=β=90°and γ=120° thus, making it suitable for piezoelectric and nonlinear optical (NLO) applications [2]. The nonlinear optical (NLO) properties of large organic molecules and polymers have been the subject of extensive theoretical and experimental investigations during the past two decades [3]. Usually the gamma glycine preparation is done by changing its solvent materials [4, 5, 6, 7].here, this research method is also dependent that techniques [8]. In this research work the growth of gamma glycine grown simultaneously from two different solvent and also this Semi-organic NLO crystal doped with fertilizer. The effect of urea on gamma glycine is the main objectives and origin of this research work because of urea is an organic compound and it is used in many multi-component solid fertilizer formulations and also Urea is highly soluble in water and therefore it's also very suitable for use in fertilizer solutions. For fertilizer use, granule are preferred over prills because of their narrower particle size distribution, which is an advantage for mechanical application and the urea molecule is planar in the crystal structure, but the geometry around the nitrogens is pyramidal in the gas-phase minimum-energy structure [9].This research work involves some impacts of urea on titled crystal by the way of SEM and EDX investigation.

MATERIALS AND METHODS

Single crystal of γ-glycine is grown by slow evaporation technique at room temperature using water –ammonium sulfate as solvent and Calcium chloride as another. The starting materials glycine and ammonium sulfate and urea were taken in the equimolar ratio (1:1:1).The calculated amount of salts was dissolved in double dissolved water at

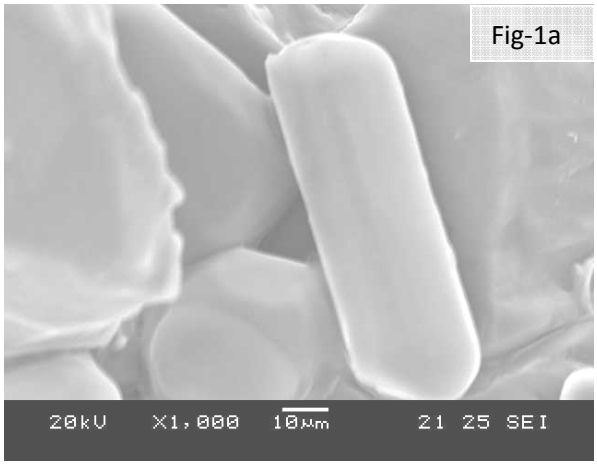
ambient conditions and similarly as the same for calcium chloride in the ratio of 4:1:1. The solution stirred well for about 6h using a magnetic stirrer to obtain a homogenous mixture. The saturated solution was filtered using whatman (41) filter paper. The filtered solution was poured into a beaker and it is kept at room temperature in a dust free compartment for slow evaporation process .The induction period for γ-nucleation is 48 hours. After 2 days a colorless powder are obtained at the bottom of the beaker and perti-dish. The powder harvested from the beaker and one again filtered by whatman filter (41) for crystallization process, after some period once again some colorless powder deposited at the bottom of the beaker which is smaller amount than the previous state. The sample was collected from the beaker and it was send to primary investigation process by SEM and EDX studies.

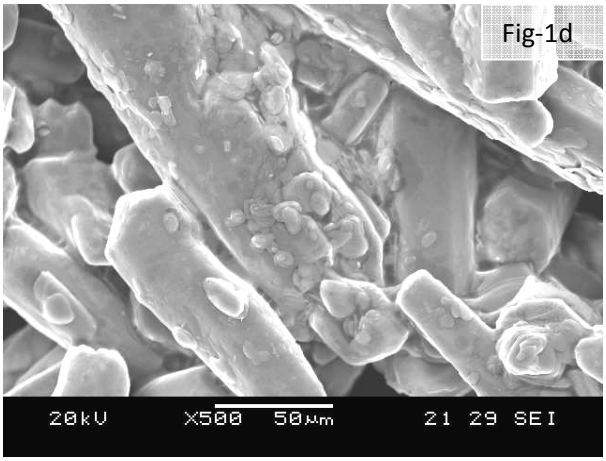
RESULTS AND DISCUSSION

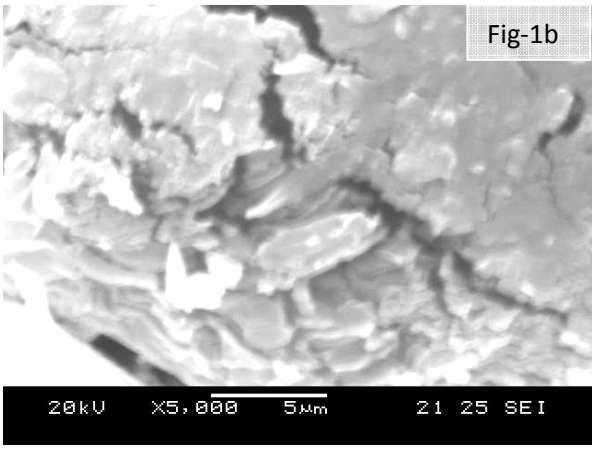
3.0.1. SEM Analysis:

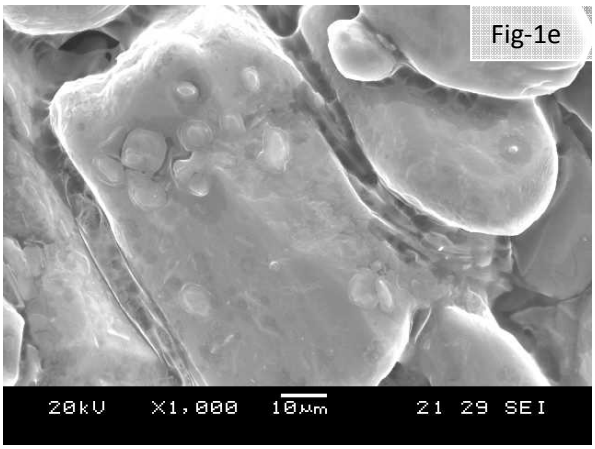
After the synthesis progression of urea doped γ glycine, the solution was filtered by wattmann filter41 and which was pouring in to Petridish and 50 mL beaker. The beaker and Petridish was covered by transparent sheet and makes some holes on it for the slow evaporation process and it was allowed for the seed preparation itinerary. After 48 hours, some powders were deposited at the bottom of the Petri dish and beaker. We collected the samples and dried at 33^0C for finding the primary character investigation. First the harvested sample was allowed to study surface analysis by Scanning electron microscope. The SEM analysis result shows the primary surface investigation (Fig - 1^{abc}, 1^{def}) from the SEM image figure, it is confirmed that some of Ammonium sulphate and calcium chloride powder were deposited at the bottom of the beaker within 48 hours. The grain size of the particle is from 1µm to 10 µm and 5 µm to 50 µm.It was verified by Scanning electron microscope photograph results [10].

Figures: 1 SEM images of Ammonium sulphate (1a-1c) and Calcium chloride (1d-1e).









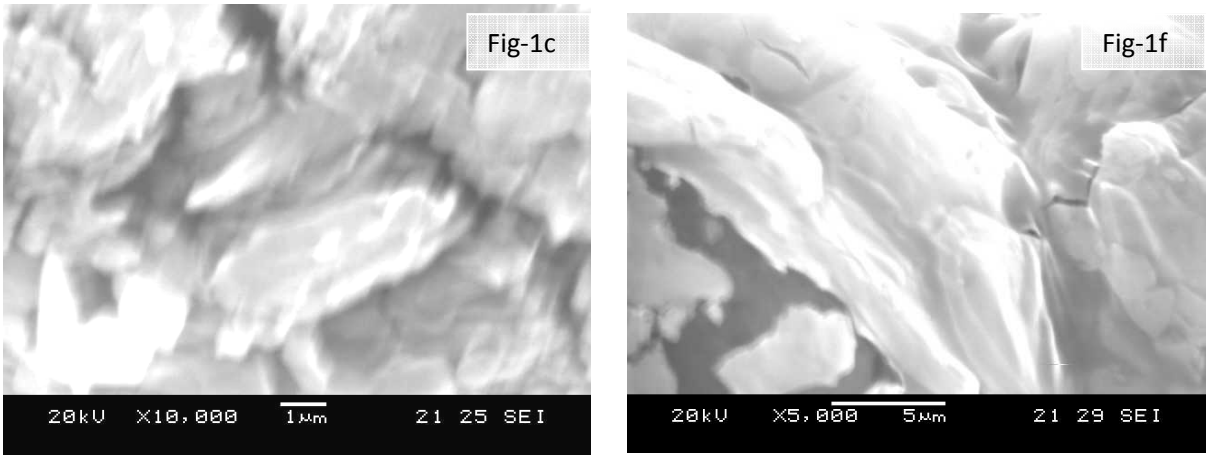


Figure: 2 EDX Graphs (Energy dispersive X-ray Graph of Pre-deposition Sample form the Solution)

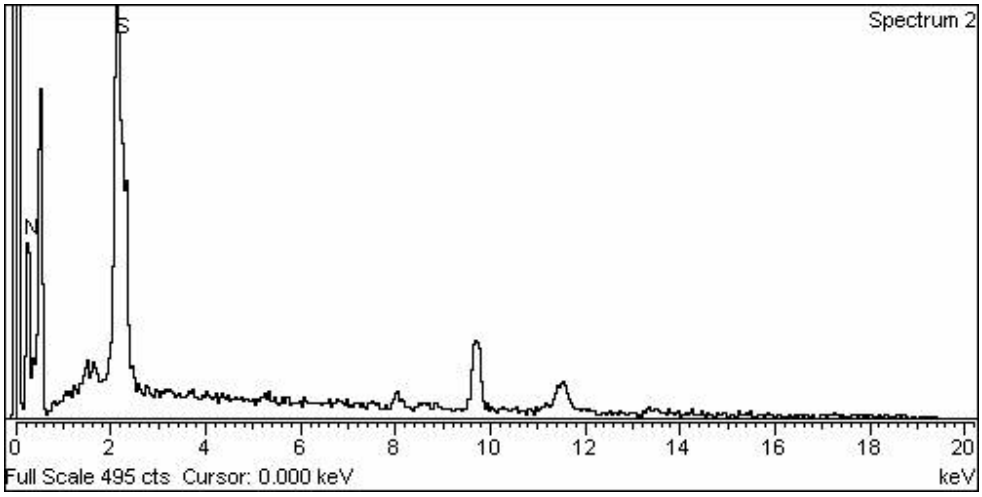


Fig-2a : Energy dispersive X-ray Graph of Pre-deposition Ammonium sulphate Sample

3.0.2. EDX Studies.

The collected sample from the solution was dried by 33^{o}C and its chemical characterization was found by the EDX investigation. The energy dispersive X-ray graph implies that, the pre-deposition sample of urea doped gamma glycine from ammonium sulphate as solvent solution contains only N and S in the range of 0.68% molecular weight. The spectrums processing the peak possibly omitted ranges are 1.500, 8.020, 9.711, 11.517 keV and number of iterations is only three. And for the calcium chloride as solvent for urea doped gamma glycine pre deposition sample contains Ca and Cl in the range of 0.98% molecular weights at the magnification range of 20kV with 500 magnification and its Peaks possibly omitted ranges are 0.520, 1.500, 2.133, 8.020, 9.711, 11.517 keV. Here the number of iteration is only one. The EDX graphs and its ingredients content tables show the above discussions.Fig-2[ab], table 1a, 1b.

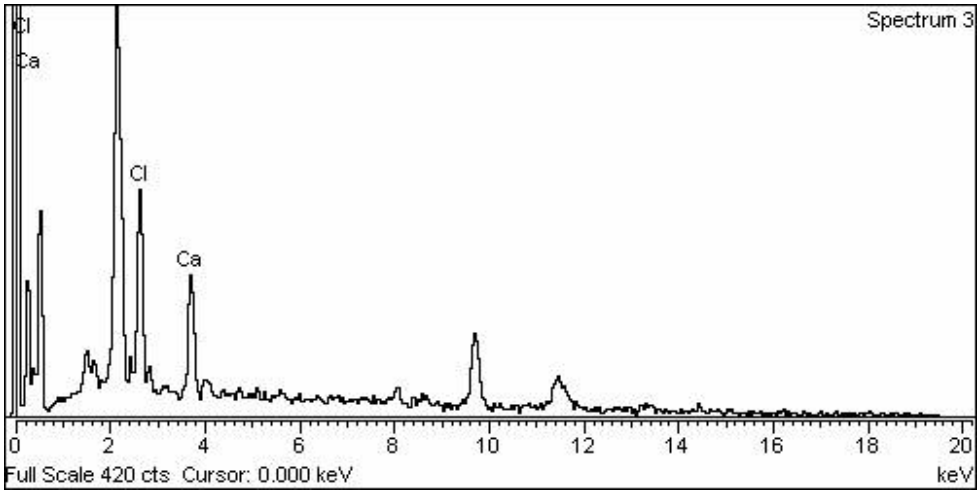


Fig-2b : Energy dispersive X-ray Graph of Pre-deposition Calcium chloride Sample.

Table 1a: EDX Calculated value for pre-deposition ammonium sulphate sample

Element	App(Conc.)	Intensity(corrn)	Weight %	Weight %(sigma)	Atomic %
NK	0.07	0.1557	0.45	0.47	80.96
SK	0.24	1.0242	0.24	0.04	19.04
Total			0.68	0.51	

Table 1b: EDX Calculated value for pre-deposition calcium chloride sample

Element	App(Conc.)	Intensity(corrn)	Weight %	Weight %(sigma)	Atomic %
Cl K	0.49	0.9633	0.51	0.03	55.00
Ca K	0.43	0.8939	0.47	0.04	45.00
Total			0.98	0.07	

CONCLUSION

The urea doped γ glycine was synthesized from ammonium sulfate and calcium chloride as solvents by slow evaporation techniques in the ratio of 1:1:1 and 4:1:1. The primary investigation was done for the pre-deposition sample and it shows that, 0.68 percentage of Ammonium sulphate form the calculate value and 0.98 percentages of calcium chloride are deposited at the bottom of the beaker of urea doped gamma glycine solution within two days. Cause for that urea binding together with γ glycine. When we added urea with synthesis process of gamma glycine directly from the basic preparation it may causes some of the ratios of ammonium sulphate and calcium chloride separated and deposited at the bottom of the beaker with in 48 hours. This pre-deposition sample may reduce the growth speed of the gamma glycine and also it may change the character of gamma glycine. The grain size 10µm and ingredients N and S are confirmed by use of SEM and EDX investigation for urea doped gamma glycine from ammonium sulphate as solvent and also The grain size 50 µm and ingredients Ca and Cl are confirmed by use of SEM and EDX investigation for urea doped gamma glycine from calcium chloride as solvent.

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