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# Microhardness and Morphological Analysis of Organic Single Crystal of 1-Glutamic Acid1-Pyroglutamic Acid Hydrate

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**Abstract:** ***1-Glutamic acid 1-Pyroglutamic acid Hydrate organic single crystal grown by slow evaporation solution growth technique at room temperature was subjected to XRD, EDAX, micro hardness and etching analysis. X-ray characterization studies reveal that the grown single crystal forms monoclinic crystal lattice. Chemical composition and mechanical stability of the organic single crystal has been investigated. Etching has been performed to study the growth morphologyin the nonlinear optical material.***

**Keywords:** crystal growth, XRD, EDAX, microhardness and etching analysis.

## 1. Introduction

In recent years, organic NLO materials are attracting a great deal of attention for possible use in optical devices because of their large optical nonlinearity, low cut-off wavelengths, short response time and high laser damage thresholds. Considerable work has been done in order to understand the microscopic origin of nonlinear behaviour of organic materials [1-5]. The NLO properties of large organic molecules and polymers have been the subject of extensive theoretical and experimental investigations during the past two decades. They have been investigated widely due to their high nonlinear optical properties, rapid response in electro-optic effect and large second or third-order hyperpolarizibilities compared to inorganic NLO materials [6]. Organic single crystals of glutamic acid has been the foci of research in the recent times [7-11]. A mixed crystal of glutamic acid and pyroglutamic acid, its structure, similar to that of the title crystal has been solved and reported [12,13]. In the present investigation, we report for the first time, EDAX, microhardness and etching analysis of 1-Glutamic Acid 1-Pyroglutamic Acid Hydrate(GPAH) crystals.

## 2. Experimental

### 2.1 X-ray diffraction analysis

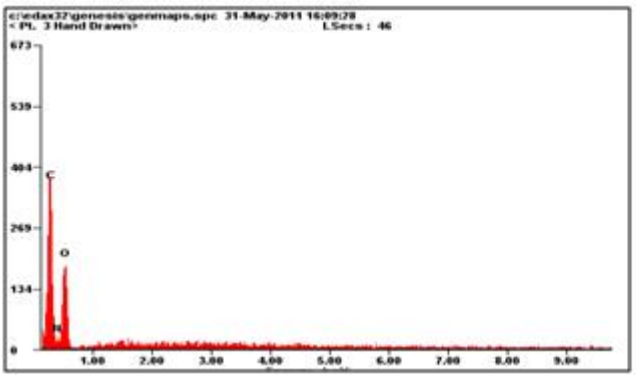
The grown GPAH single crystal was subjected to X-ray diffraction using Bruker Axs Apex 2 single crystal diffractometer and the crystal identity was determined. The unit cell dimensions determined from single crystal X-ray diffraction analysis are presented in table 1.

**Table 1:** Crystallographic data of GPAH crystal

| Parameter | Experimental | Reported |
|---|---|---|
| Molecular Formula | $C_{10}H_{18}N_2O_8.\ 2H_2O$ | $C_{10}H_{16}N_2O_7.\ H_2O$ |
| Lattice Parameters | a = 5.08 (Å) | a = 18.473(1) (Å) |
| | b = 7.13 (Å ) | b = 7.190(1) ( Å ) |
| | c = 18.37 (Å) | c = 5.113 (1) (Å) |
| Angles | $\alpha = 90^0$ | $\alpha = 90^0$ |
| | $\beta = 96.81^0$ | $\beta = 96.77^0$ |
| | $\gamma = 90^0$ | $\gamma = 90^0$ |
| Volume | $661(Å)^3$ | $674.3\ (5)\ (Å)^3$ |
| Crystal System | Monoclinic | Monoclinic |

### 2.2 Energy Dispersive Analysis (EDAX)

EDAX is an analytical technique used for the elemental analysis or chemical characterization of a sample.EDAX spectrums are shown in figures 1 and 2, elemental compositions in table 2 and 3. These two spectrums correspond to the EDAX patterns obtained from the starting composition and the crystal respectively.



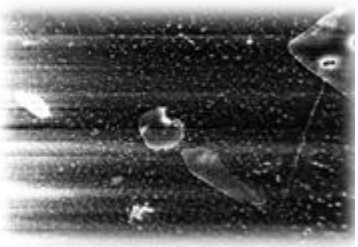



**Figure 1:** EDAX of starting composition

**Table 2:** Data from EDAX analysis of starting composition

| Element | Weight percentage (wt%) | Atomic percentage (At%) |
|---|---|---|
| C | 55.08 | 61.19 |
| N | 11.29 | 10.76 |
| O | 33.63 | 28.05 |

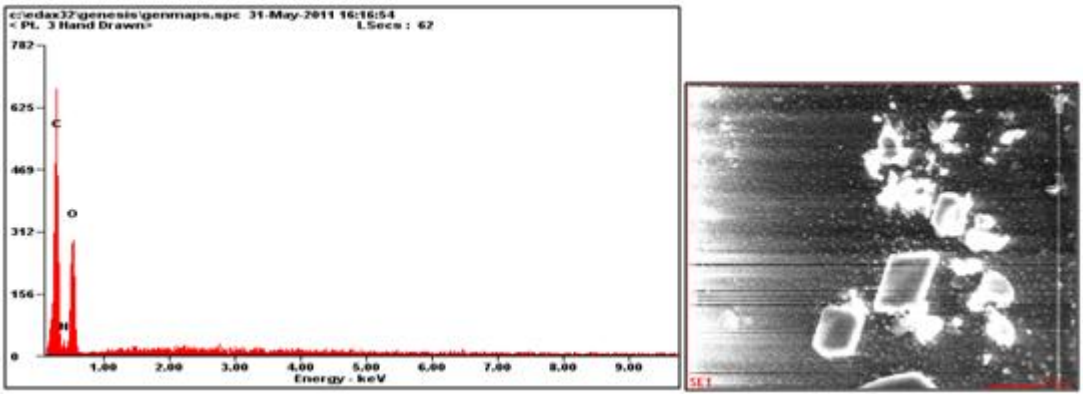



**Figure 2:** EDAX of GPAH showing peaks corresponding to expected elements C,N and O.

**Table 3:** Data from EDAX analysis of GPAH crystal

| *Element* | *Weight percentage (wt%)* | *Atomic percentage (At%)* |
|---|---|---|
| C | 56.24 | 62.60 |
| N | 07.00 | 06.68 |
| O | 36.77 | 30.73 |

### 2.3 Mechanical Studies

Micro hardness analysis is used to describe the hardness testing of materials with low applied loads.Well-polished single crystals of 1-Glutamic acid 1-Pyroglutamic acid Hydrate were subjected to Vickers Micro indentation techniques using Leica Optical Microscope Md 4000 and the hardness values are tabulated in Table 4. Figure 3 highlights the indentation mark in GPAH crystal. From the data a graph between $H_V$ and P is drawn (figure 4). It is found that the hardness value increases up to 30 gms and then decreases for higher loads. This is due to molecular strength developed in the crystal during crystallization process.

**Table 4:** Hardness values of 1-Glutamic acid 1-Pyroglutamic acid Hydrate

| *Force (gm)* | *d1 (µm)* | *d2 (µm)* | *(d1+d2)/2* | *Hardness ($H_V$)* |
|---|---|---|---|---|
| 5 | 42.56 | 41.34 | 41.95 | 5.4 |
| 10 | 43.48 | 40.41 | 41.95 | 10.5 |
| 15 | 44.85 | 39.49 | 42.17 | 15.8 |
| 20 | 46.68 | 40.72 | 43.70 | 19.5 |
| 25 | 44.85 | 41.65 | 43.25 | 24.9 |
| 30 | 43.49 | 41.65 | 42.56 | 30.7 |
| 35 | 54.00 | 49.05 | 51.53 | 24.5 |
| 40 | 56.29 | 51.52 | 53.91 | 22.7 |

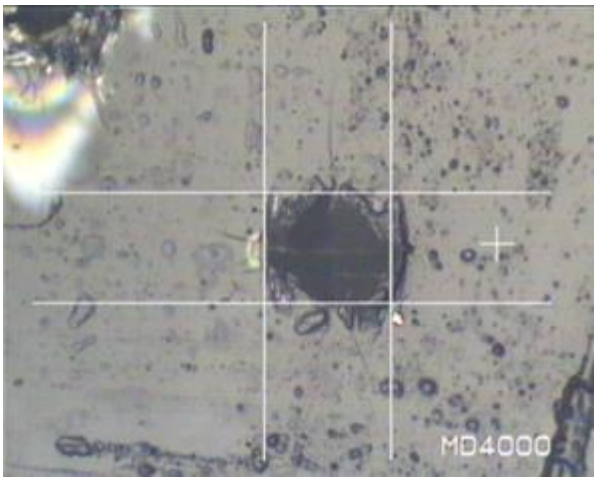



**Figure 3:** Micro Hardness Indentation Mark

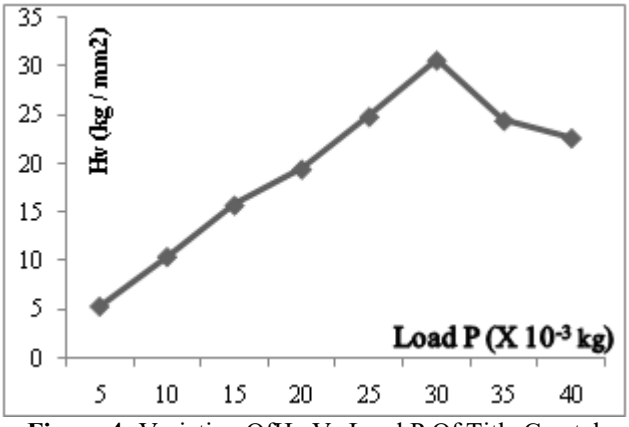



**Figure 4:** Variation Of$H_V$ Vs Load P Of Title Crystal

### 2.4 Etching Analysis

Nature and distribution of defects in crystals is to be known necessarily as they affect the performance when used as electronic devices. Etching studies have been carried out on GPAH organic single crystal to understand the growth mechanism and perfection of grown crystal. Water is used as etchant with the etching time of 5 Sec and 15 Sec, and the crystal images are shown in the photographs5 and 6

respectively. Randomly distributed etch patterns on the crystal surface is observed which increases with etching time. The nature of the etch pit pattern does not change indicating no change in the crystal morphology.

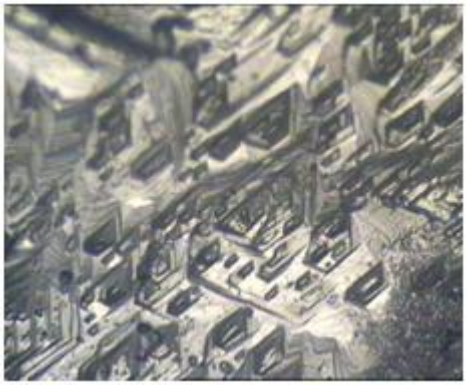


**Figure 5:** Etching time of 5 Sec

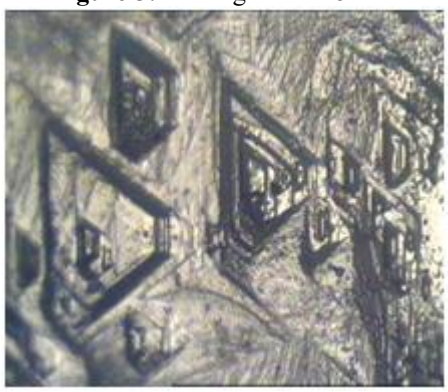


**Figure 6:** Etching time of 15 Sec

## 3. Conclusion

Good quality single crystals of GPAH grown successfully by the slow evaporation solution growth method were subjected to XRD, EDAX, microhardness and etching studies. From the XRD analysis, the lattice parameters are calculated and the monoclinic structure is confirmed. The chemical composition of the grown single crystal has been investigated from EDAX analysis which infers the absence of impurities in the nonlinear optical crystal.Microhardness and etching analysis indicate that the crystal is soft and has uniform growth morphology.

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