GROWTH AND CHARACTERISATION OF GLYCINIUM MALEATE DIHYDRATE

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Abstract: ***Glycinium maleate dihydrate was grown by slow evaporation at room temperature. The grown crystals were characterized using FT-IR, UV-Vis, SEM and Powder X-ray diffraction. The presence of various functional groups was confirmed by FT-IR spectra. The UV-Vis spectra indicated that the crystal has good absorption in the entire visible region. The morphology of the crystal has been identified from SEM Analysis.***

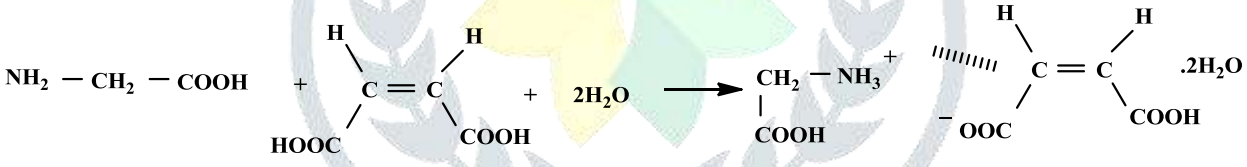
IndexTerms - Infrared spectrum, Powder XRD, SEM, UV-Vis spectra.

I. INTRODUCTION

Organic non-linear optical materials are of great interest due to its demand in photonic applications. A large number of organic nonlinear optical materials have been accounted because of its nonlinear optical applications. Amino acid mixed organic crystals have been great attention in the optical applications compared to other organic compound [1]. The amino acid glycine gains more when it reacted with maleic acid which is a dicarboxylic aid has great attention in the development of non-linear optical material. Due to its large pi conjugation [2]. In this study, Glycinium Maleate dehydrate is grown using water as a solvent and the grown crystals were characterized by FT-IR, UV-Vis, Powder X-ray Diffraction analysis and SEM analysis.

II. CRYSTAL GROWTH

Maleic acid was first dissolved in 40 ml of water. To this glycine was then added directly slowly by heating in a magnetic stirrer. The homogenous solution of glycinium maleate dihydrate was prepared at room temperature. The solution was then filtered to remove the suspended impurities and covered with filter paper and made holes on the filter paper and allowed to evaporate slowly. A good transparent crystal harvested in a few days which is in the following reaction.



III. CHARACTERIZATION TECHNIQUES

The grown crystals of glycinium maleate dehydrate were confirmed by powder X-ray diffraction analysis to confirm the crystallinity structure. The functional groups were identified by FT-IR using FT-IR Spectrometer in the range of 400-4000c m^{-1}. The optical properties of the crystals were examined by using lamda UV-Vis spectrometer.

IV. RESULTS AND DISCUSSION

4.1 FT-IR spectral Analysis

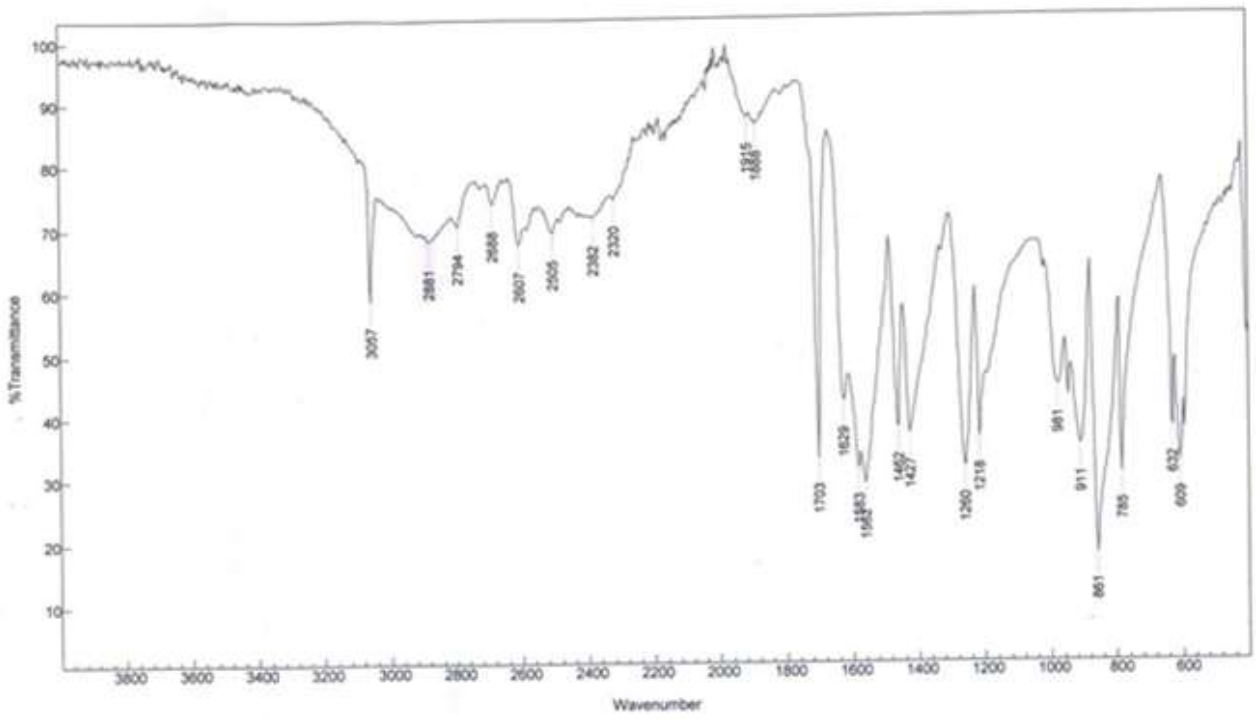
The FT-IR spectra for pure maleic acid as well as glycine doped maleic acid crystals were recorded using FT-IR instrument in the range 400-4200cm^{-1} is shown in the "**Figure 1**" and "**Figure 2**". The calculated frequencies obtained in FT-IR of pure maleic acid and formed crystals and their most probable assignments are presented in Table 1.

The peaks at 3057cm^{-1} in pure maleic acid are shifted to 3059cm^{-1} in formed crystal. All the peaks corresponding to medium C-H stretching frequencies at 2881cm^{-1}, 2794cm^{-1}, 2688cm^{-1} are shifted to 2871cm^{-1}, 2793cm^{-1} and 2685cm^{-1} which may be due to the incorporation of glycine. The weak C-H bending 1888cm^{-1} is shifted to 1887cm^{-1}. There was not much change in carbonyl stretching frequency. The NH^{2+} stretching frequency 1561cm-1 is found in doped crystal. The O-H bending frequencies 1462cm^{-1}, 1427cm^{-1} are shifted to 1456cm^{-1} and 1431cm^{-1}. The peaks correspond to C=C bending 961cm^{-1}, 911cm^{-1}, 861cm^{-1} are shifted to 983cm^{-1}, 910cm^{-1} and 850cm^{-1}.

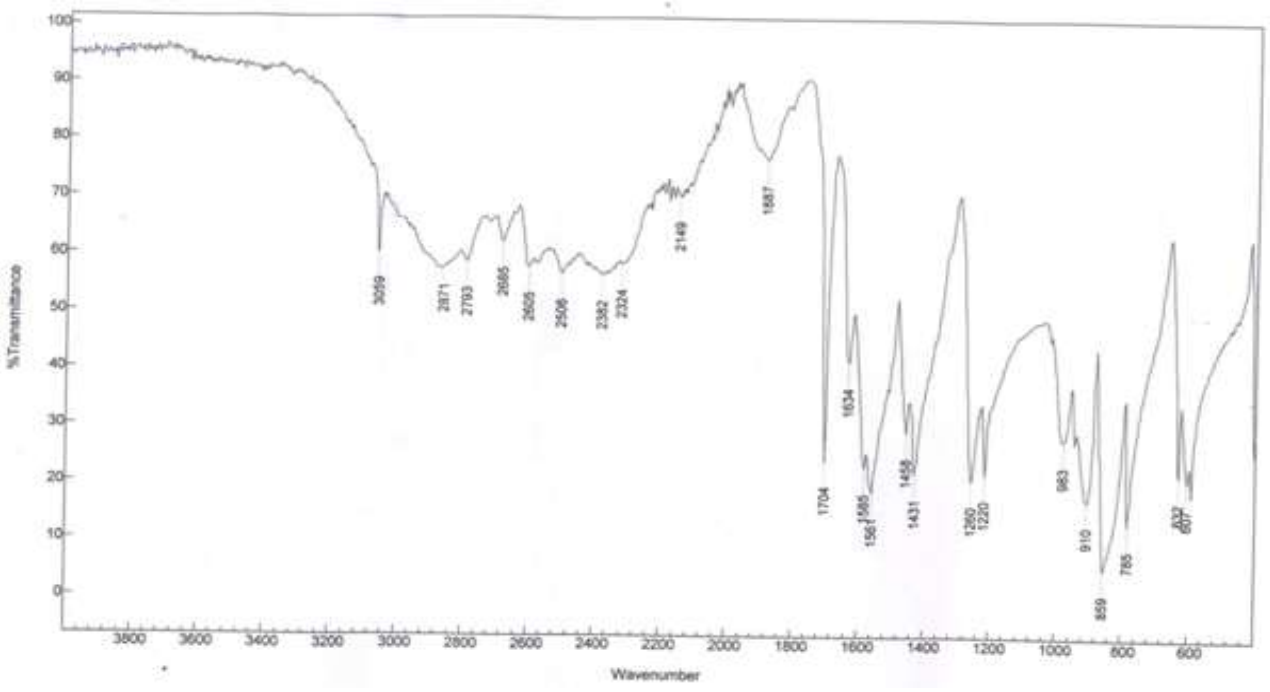
Table 1**:** IR absorption frequencies of pure Maleic acid and doped crystal

Wave number cm-1		Assignments
Maleic acid	Glycinium maleate dihydrate	
3057	3059	Strong broad N-H stretching weak O-H stretching
2881	2871	Medium C-H stretching

2794 2688	2793 2685	
1888	1887	Weak C-H bending
1704 1629	1704 1634	C=O stretching
-	1561	NH+ stretching
1462 1427	1456 1431	O-H bending
961 911 861 785	983 910 850 785	C=C bending



"Fig.1" FT-IR spectrum of pure Maleic acid

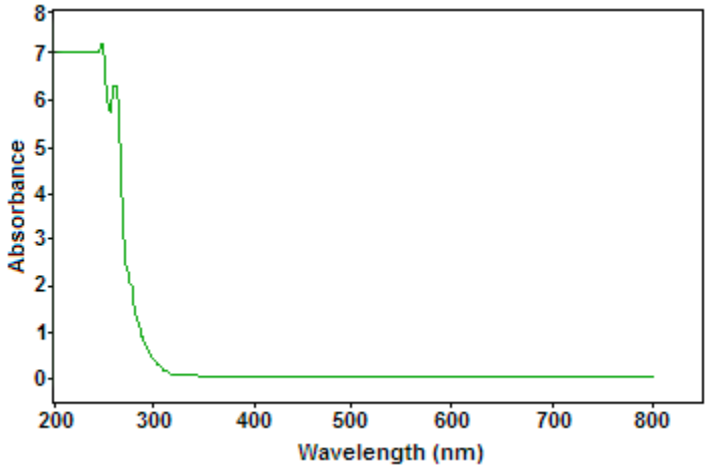


"Fig .2" FT-IR spectrum of Glycinium maleate dihydrate

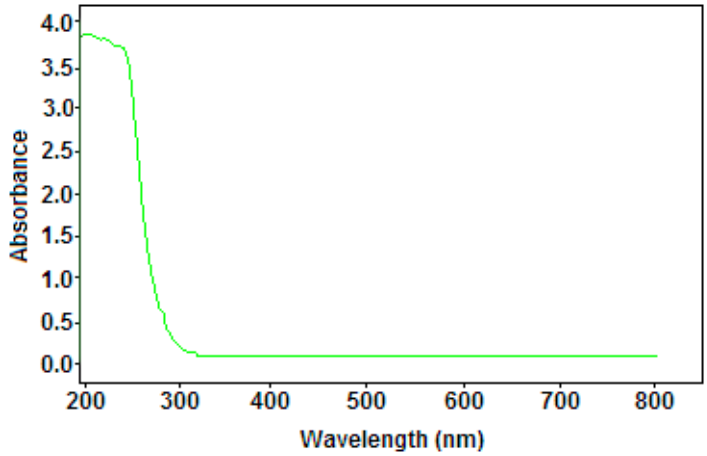
4.2 UV-Vis spectral Analysis

The UV-Visible spectrum of pure and doped crystal was recorded in the range of 200-800nm using lambda spectrometer. The UV-Visible spectrum of pure and doped crystals is shown in the "**Figure 3**" and "**Figure 4**".

Regarding the electronic absorption, there is n-π* transition of 260nm is observed in formed crystal. As there is no change in cut-off wavelength is observed in both the crystals and residual absorption is observed in both the crystal in the entire UV and Visible region.



"Fig 3" UV-Vis spectrum of pure Maleic acid

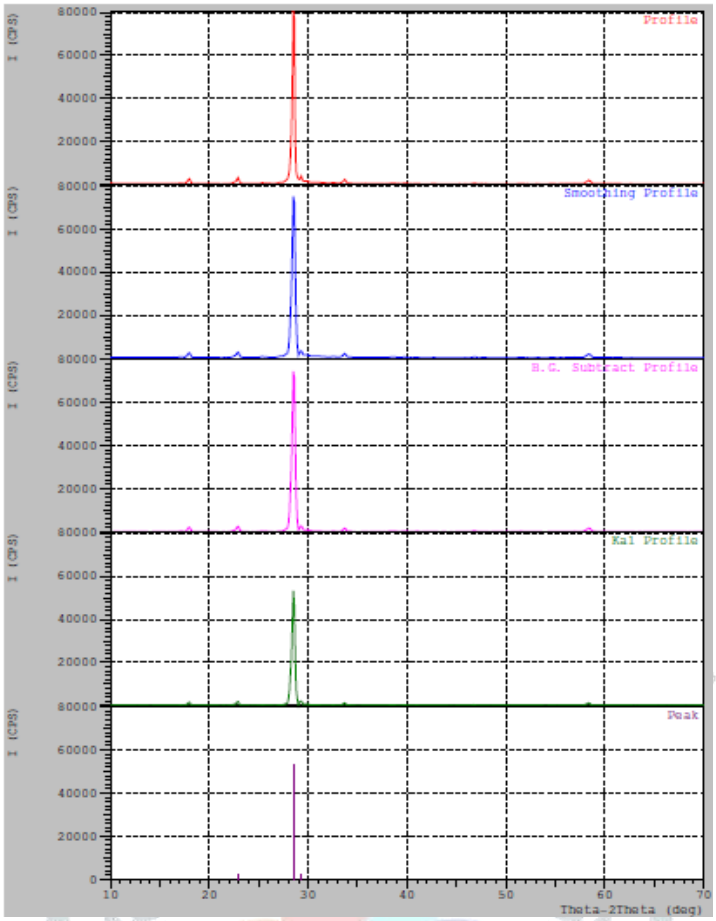


"Fig 4" UV-Vis spectrum of Glycinium maleate dihydrate

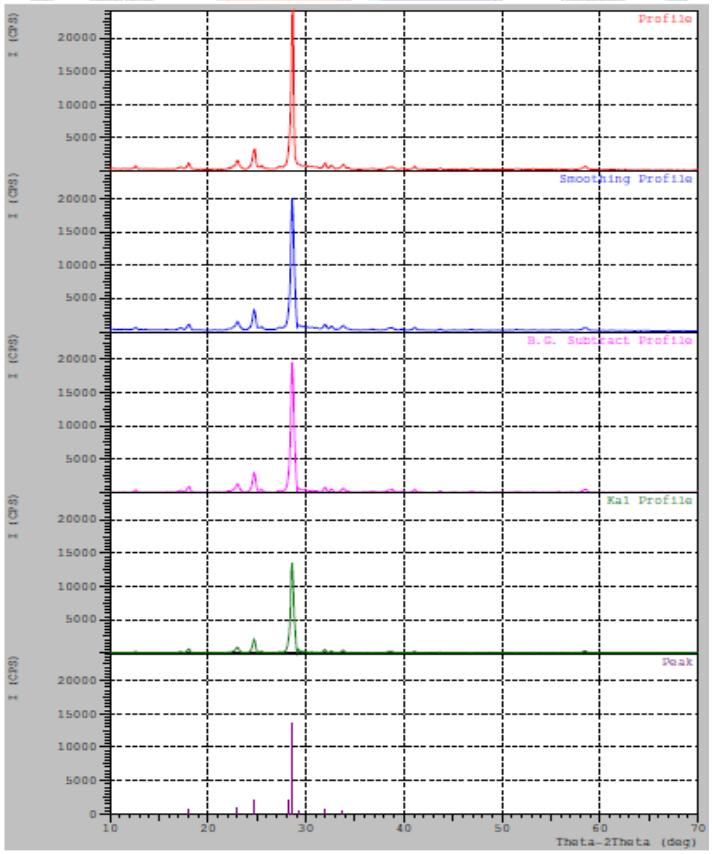
4.3 Powder X-ray diffraction Analysis

It is the most widely used X-ray diffraction technique for characterizing materials. This technique is used widely for studying particles in polycrystalline solids.

XRD peaks of glycinium maleate dihydrate are not similar to that of pure maleic acid. Additional peaks and phases are present in the formed crystals. All these facts suggested that the formed crystal have different structure as that of pure crystal. The XRD peaks are shown in the "**Figure 5**" and "**Figure 6**".



"Fig 5" XRD of pure Maleic acid

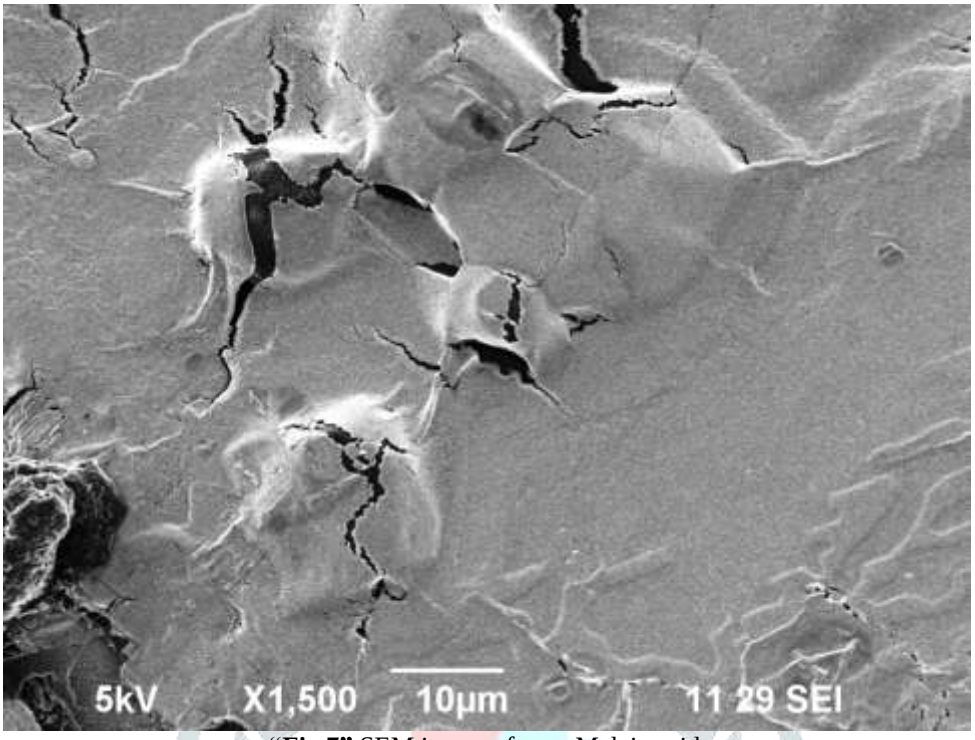


"Fig 6" XRD of Glycinium maleate dehydrate

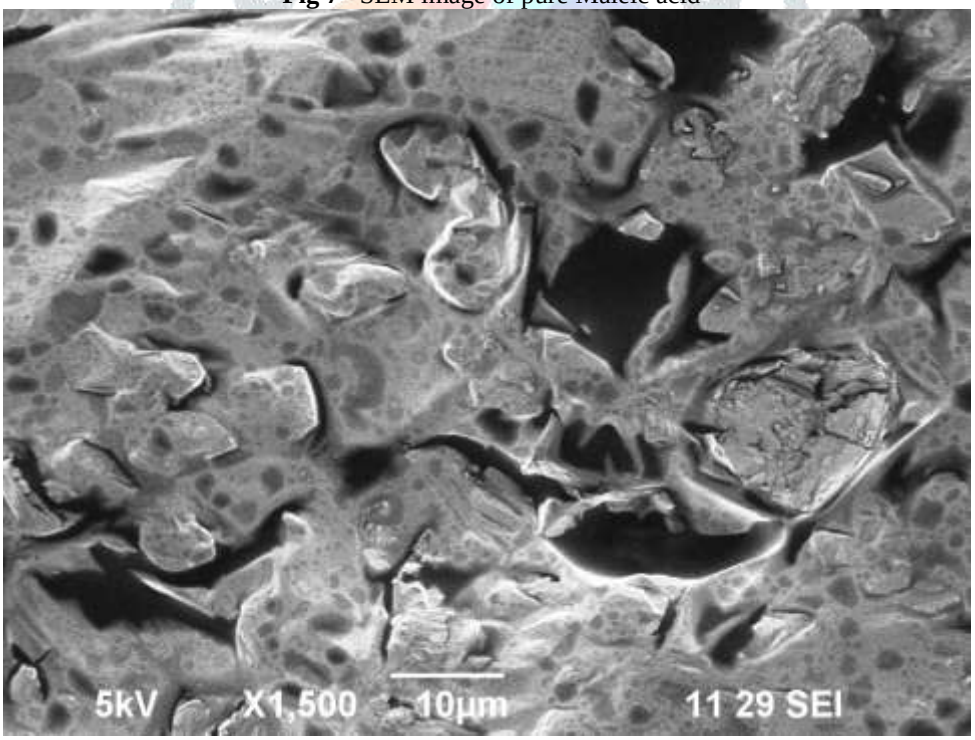
4.4 Scanning Electron Microscopic Analysis

The image is SEM is produced by scanning the sample with a focused electron beam and detecting the secondary and back scattered electrons from the conventional SEM image. The SEM image of maleic acid formed crystal is shown in the "**Figure 7**" and "**Figure 8**" respectively.

In the pure maleic acid, we observed scattered small ice pieces look like island. In the case of glycinium maleate dihydrate accumulated ice caps are observed. This confirms the crystallinity structure [9, 10].



"Fig 7" SEM image of pure Maleic acid



"Fig 8" SEM image of doped crystal

V. CONCLUSION

Good optical qualities crystals of maleic acid and glycinium maleate dihydrate are grown by slow evaporation at room temperature. Both the crystals were characterized by powder X-ray diffraction and confirmed the crystallinity nature of the crystal. The presence of the functional groups is assigned by FT-IR analysis. The UV absorption studies revealed the optical property of the crystal. SEM images revealed the morphology property.

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